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FINAL SAMPLING AND ANALYSIS PLAN FOR SOLID WASTE MANAGEMENT UNIT 11 OLD STORAGE BUILDING NSA CRANE IN

3/1/2011

TETRA TECH NUS

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001

SAMPLING AND ANALYSIS PLAN

FINAL

for

SWMU 11 – Old Storage Building 225

Naval Support Activity Crane
Crane, Indiana

Contract Task Order F27E

March 2011

NAVFAC Naval Facilities Engineering Command
Midwest
201 Decatur Avenue
Building 1A, Code EV
Great Lakes, Illinois 60088

TETRA TECH NUS, Inc.

SAP Worksheet No. 1 -- Title and Approval Page

(UFP-QAPP Manual Section 2.1)

FINAL
Revision 0
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
March 2011

RESOURCE CONSERVATION AND RECOVERY ACT
FACILITY INVESTIGATION

SWMU 11 – OLD STORAGE BUILDING 225
NAVAL SUPPORT ACTIVITY CRANE
CRANE, INDIANA

Prepared for:
Naval Facilities Engineering Command Midwest
201 Decatur Ave., Building 1A
Great Lakes, Illinois 60088

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
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Prepared under:
Contract No. N62470-08-D-1001
Contract Task Order F27E

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Approval Signatures:

Thomas J. Brent/ RPM/ Date
NSA Crane Environmental Restoration Site Manager

Doug Griffin/ RPM/ Date
Indiana Department of Environmental Management

Navy Chemist/ Date

SAP Worksheet No. 1 -- Title and Approval Page

(UFP-QAPP Manual Section 2.1)

DRAFT FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
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NSA Crane Environmental Restoration Site Manager

Doug Griffin/ RPM/ Date
Indiana Department of Environmental Management

Jonathan Tucker Digitally signed by TUCKER.JONATHAN.P.1239524180 Date: 2011.03.17 12:51:49 -04'00'
Navy Chemist/ Date

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at Solid Waste Management Unit (SWMU) 11 – Old Storage Building 225 at Naval Support Activity (NSA) Crane, Indiana under Contract Task Order (CTO) F27E, Contract N62470-08-D-1001, Comprehensive Long-term Environmental Action Navy (CLEAN).

SWMU 11 – Old Storage Building 225 (site) is located near the western boundary of NSA Crane, approximately midway between the northern and southern boundaries of the facility. The site is a rectangular shape and covers approximately 1 acre. Building 225 was used for storage of inert materials including pentachlorophenol (PCP), paints, sodium flourescein dye, solvents (acetone, toluene, and methyl ethyl ketone), and various other items including inks, staples, and wax paraffin. The building was destroyed by a fire on July 13, 1976.

Water used in fighting the fire in July 1976 may have become contaminated with the chemicals stored in the building. According to the "Green Water" Report (Department of the Navy, 1976), runoff from fire fighting activities was brilliant fluorescent green from the flourescein dye. The green colored water entered the drainage channel and the ditch east of the site and began flowing off-site. PCP, paints, sodium fluorescein, solvents, and other items stored in the building may have also entered the surface drainage and soils in the area of the site during firefighting efforts, and may continue to be present in these areas. Potentially contaminated water was contained by temporary dams that were constructed in the stream (Broom Branch) downstream of the site, but the water was eventually discharged from NSA Crane. The "Green Water" Report stated that several thousand gallons of water was pumped from the site and placed in a concrete reservoir to be properly disposed at a later date. The disposition of that water is unknown. Smoke from the fire may have also introduced contaminants that settled downwind from the site.

The primary purpose of the RFI described in this SAP is to conduct an initial site investigation of the potential contaminants at the site, and if present, to determine the nature and extent of those contaminants. This information will be used in the remedial decision process. The RFI will include the collection and analysis of surface and subsurface soil, sediment, and groundwater samples. These samples will be collected to provide representative coverage of the site for use in human health and ecological risk screening and potentially for risk assessment, if necessary.

Surface and subsurface soil, sediment, and groundwater samples will be analyzed for volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), including low level polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, dioxins/furans, metals, and cyanide.

Sediment samples will also be analyzed for total organic carbon (TOC) to support site-specific risk calculations.

The SAP contained herein was generated for, and complies with, applicable Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), and United States Environmental Protection Agency (USEPA) Region 5 requirements, regulations, guidance, and technical standards, as appropriate.

This SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the planned investigation at SWMU 11. Protocols for sample collection, handling and storage, chain-of-custody, laboratory and field analyses, data validation, and reporting are also addressed in this SAP. The investigation procedures utilized will comply with Tetra Tech Standard Operating Procedures (SOPs), which are included in Appendix A. The analytical procedures will follow the Laboratory SOPs which are included in Appendix B. The field work is scheduled to begin in April 2011. A complete schedule is presented in SAP Worksheet No. 16.

Field activities conducted under this SAP will be in accordance with the requirements a Site-Specific Health and Safety Plan than will be prepared by Tetra Tech for this investigation.

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Appendices

A - Tetra Tech Standard Operating Procedures and Field Forms
B - Laboratory Standard Operating Procedures
C - DQO Meeting Minutes
D - Background Reports
E - Project Screening Level Backup Documentation

ACRONYMS AND ABBREVIATIONS

AR	Administrative Record
BFB	Bromofluorobenzene
bgs	below ground surface
°C	degrees Celsius
CA	Corrective Action
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CFA	Cape Fear Analytical, LLC
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
CMS	Corrective Measures Study
COC	Contaminant of Concern
COPC	Constituent of Potential Concern
CPCS	Column Performance Check Solution
CSM	Conceptual Site Model
CTO	Contract Task Order
CWAP	Comprehensive Work Approval Process
%D	percent difference or percent drift
DAF	Dilution Attenuation Factor
DCL	Default Closure Level
DDT	dichlorodiphenyltrichloroethane
DFTPP	decafluorotriphenylphosphine
DO	dissolved oxygen
DON	Department of the Navy
DL	Detection Limit
DoD	Department of Defense
DPT	direct-push technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
Eco-SSL	Ecological Soil Screening Level
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
Empirical	Empirical Laboratories, LLC

ACRONYMS AND ABBREVIATIONS (CONTINUED)

EPC	Exposure Point Concentration
ERA	Ecological Risk Assessment
ERSM	Environmental Restoration Site Manager
ESL	Ecological Screening Level
EU	Exposure Unit
FD	Field Duplicate
FOL	Field Operations Leader
FTMR	Field Task Modification Request
g	gram
GC/ECD	Gas Chromatography/ Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GPS	global positioning system
HASP	Health and Safety Plan
HCl	hydrochloric acid
HHRA	Human Health Risk Assessment
HSM	Health and Safety Manager
IA	Investigative Area
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICS	Interference Check Standard
ICV	Initial Calibration Verification
IDEM	Indiana Department of Environmental Management
IDW	investigation-derived waste
IS	Internal Standard
IUPPS	Indiana Underground Plant Protection Services
L	liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit Of Quantitation
LUC	land use control
MCL	Maximum Contaminant Level
mg/L	milligram per liter
mg/kg	milligram per kilogram
mL	milliliter
MPC	Measurement Performance Criterion

ACRONYMS AND ABBREVIATIONS (CONTINUED)

msl	mean sea level
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NaOH	Sodium Hydroxide
NAVFAC	Naval Facilities Engineering Command
NC	no criteria
ND	non detect
NFA	No Further Action
NIRIS	Naval Installation Restoration information Solution
NOAA	National Oceanic and Atmospheric Administration
NSA	Naval Support Activity
NTU	Nephelometric Turbidity Unit
ORP	Oxidation-Reduction Potential
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCP	pentachlorophenol
PDF	Portable Document Format
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
ppb	part per billion
PPE	personal protective equipment
ppm	part per million
PQLG	Project Quantitation Limit Goal
PQO	project quality objective
PSL	Project Screening Level
PWD	Public Works Department
QA	quality assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	quality control
QSM	Quality Systems Manual
%R	percent recovery
r	Linear Regression Correlation Coefficient
r^2	Coefficient of Determination

ACRONYMS AND ABBREVIATIONS (CONTINUED)

R5 ESL-S	USEPA Region 5 Ecological Screening Level, Soil
R5 SED	USEPA Region 5 Ecological Screening Level, Sediment
RBSSL	Risk-Based Migration-to-Groundwater Soil Screening Level
RCRA	Resource Conservation and Recovery Act
R-DCLs	Risk-Default Closure Level
RF	Response Factor
RFI	Resource Conservation and Recovery Act Facility Investigation
RISC	Risk Integrated System of Closure
RPD	Relative Percent Difference
RPM	Remedial Project Manager
% RSD	Relative Standard Deviation
RSL	Regional Screening Level
R-RSL	Residential Regional Screening Level
RRT	Relative Retention Time
RT	Retention Time
SAP	Sampling and Analysis Plan
SDG	sample delivery group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language
SSO	Site Safety Officer
SVOC	semivolatile organic compound
SWMU	Solid Waste Management Unit
TAL	Target Analyte List
TBD	To Be Determined
TCL	Target Compound List
TOC	total organic carbon
T-RSL	Tapwater Regional Screening Level
Tetra Tech	Tetra Tech NUS, Inc.
UCL	Upper Confidence Limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
µg/L	microgram per liter
USEPA	United States Environmental Protection Agency
UST	underground storage tank
VOC	volatile organic compound
VSP	Visual Sample Plan

SAP Worksheet No. 2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Naval Support Activity (NSA) Crane, Solid Waste Management Unit (SWMU) 11 – Old Storage Building 225
Operable Units:	Not Applicable (NA)
Contractor Name:	Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number:	N62470-08-D-1001
Contract Title:	Comprehensive Long-term Environmental Action Navy (CLEAN)
Work Assignment Number:	Contract Task Order (CTO) F27E

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

2. Identify regulatory program: The Indiana Department of Environmental Management (IDEM) Hazardous Waste Closure and Corrective Action Programs, which implement and enforce the Resource Conservation and Recovery Act (RCRA), and related state laws and rules.

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Data Quality Objective (DQO) Meeting	November 4, 2010

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

Title	Date
"*Green Water" Report* (Department of the Navy [DON], 1976a)	September 20, 1976
Report on the Fire Investigation, Building 225, Crane Naval Weapons Support Center; Crane Indiana; 14-16 July 1976, NAVMAT Report 11320.2 (DON, 1976b)	1976

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

IDEM (lead regulatory agency), USEPA Region 5 (regulatory oversight), Naval Facilities Engineering Command (NAVFAC) Midwest, NSA Crane, (property owner), Tetra Tech (Navy contractor)

7. Lead organization: NAVFAC Midwest

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

NA, as there are no exclusions.

SAP Worksheet No. 3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Howard Hickey	NAVFAC Remedial Project Manager (RPM)/ Manages Project Activities for the Navy	NAVFAC Midwest	847-688-2600 X243	howard.hickey@navy.mil
Tom Brent	Environmental Restoration Site Manager (ERSM)/ Crane Point of Contact (POC)	NAVFAC Midwest Public Works Department (PWD) Crane	812-854-6160	thomas.brent@navy.mil
To Be Determined (TBD) (electronic upload)	NAVFAC Quality Assurance Officer (QAO)/ Government Chemist	NAVFAC Atlantic	TBD	TBD
Bonnie Capito (final cover letter only)	Librarian and Records Manager/ Navy Administrative Record (AR)	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil
Peter Ramanauskas	USEPA RPM/ Regulator Input	USEPA Region 5	312-866-7890	ramanauskas.peter@epamail.epa.gov
Doug Griffin	IDEM RPM/ Regulator Input	IDEM	317-233-2710	dgriffin@idem.in.gov
John Trepanowski (distribution letter only)	Program Manager/ Manages Navy Initiatives	Tetra Tech	610-382-1532	john.trepanowski@tetratech.com
Garth Glenn (distribution letter only)	Deputy Program Manager/ Manages Program Activities	Tetra Tech	757-461-3926	garth.glenn@tetratech.com
Anthony P. Klimek, P.E.	Project Manager (PM)/ Manages Project Activities	Tetra Tech	513-557-5057	tony.klimek@tetratech.com
Ralph Basinski	Crane Activity Coordinator/ Coordinates Tetra Tech Activities at NSA Crane	Tetra Tech	412-921-8308	ralph.basinski@tetratech.com
Tom Johnston, PhD (electronic copy only)	Quality Assurance Manager (QAM)/ Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech	412-921-8615	tom.johnston@tetratech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
Matt Soltis [Health and Safety Plan (HASP) only]	Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech	412-921-8912	matt.soltis@tetratech.com
Joe Samchuck (electronic copy only)	Data Validation Manager (DVM)/ Manages Data Validation	Tetra Tech	412-921-8510	joseph.samchuck@tetratech.com
George Ten Eyck	Field Operations Leader (FOL) and Site Safety Officer (SSO)/ Manages Field Operation and Site Safety Issues	Tetra Tech	513-557-5043	george.teneyck@tetratech.com
Mark Traxler (electronic copy only)	Project Chemist/ Provides Coordination with Laboratory	Tetra Tech	610-382-1171	mark.traxler@tetratech.com
Driller (TBD) (electronic copy only)	Drilling Subcontractor PM/ Provides Direct-Push Technology (DPT) Services	TBD	TBD	TBD
Kim Kostzer (electronic copy only)	Laboratory PM - Representative for Laboratory and Analytical Issues	Empirical Laboratories, LLC (Empirical)	615-345-1115	kkostzer@empirlabs.com
Chris Cornwell (electronic copy only)	Laboratory PM – Representative for Laboratory and Analytical Issues	Cape Fear Analytical, LLC (CFA)	910-795-0422	chris.cornwell@cfanalytical.com

SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

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

1. In the case of regulatory agency personnel with oversight authority, approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / e-mails will be retained in the project files and are listed in Worksheet No. 29 as project records.

2. E-mails will be sent to the Navy, Tetra Tech, and subcontractor project personnel who will be requested to verify by e-mail that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and is identified in Worksheet No. 29.

   A copy of the signed Worksheet No. 4 will be retained in the project files and is identified as a project document in Worksheet No. 29.

Name [1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Howard Hickey	NAVFAC/ Navy RPM/ Manages Project Activities for the Navy	847-688-2600 X243		All	
Tom Brent	NAVFAC/ ERSM/ Site POC	812-854-6160	See Worksheet No.1 for signature	All	
Doug Griffin	IDEM/ RPM/ Provides Regulator Input	317-233-2710	See Worksheet No.1 for signature	All	
Tetra Tech Project Team Personnel					
Tony Klimek, P.E.	Tetra Tech/ PM/ Manages Project Activities	513-557-5057	See Worksheet No.1 for signature	All	
Tom Johnston	Tetra Tech/ QAM/ Manages NAVFAC Contract QA Program and Implementation	412-21-8615	See Worksheet No.1 for signature	All	
George Ten Eyck	Tetra Tech/ FOL/SSO/ Manages Field Operation and Site Safety Issues	513-557-5043		All	
Matt Soltis	Tetra Tech/ HSM/ Manages Corporate Health and Safety Program	412-921-8912		HASP only	

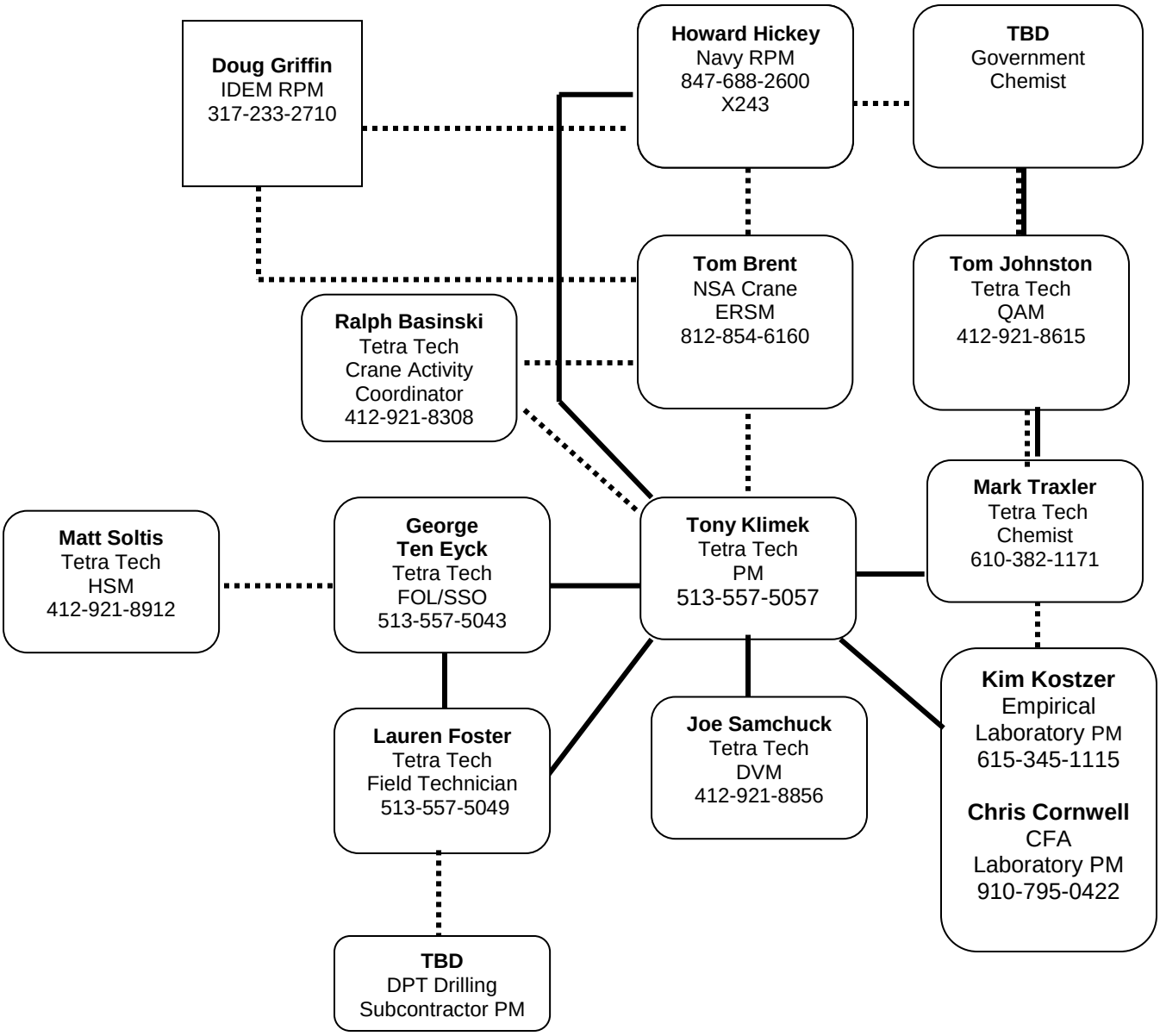
Name [1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Joe Samchuck	Tetra Tech/ DVM/ Manages Data Validation	412-921-8510		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, and 34-37	
Mark Traxler	Tetra Tech/ Project Chemist/ Provides Coordination with Laboratory	610-382-1171		All	
Subcontractor Personnel					
Kim Kostzer	Empirical/ Laboratory PM/ Representative for Laboratory and Analytical Issues	615-345-1115		Worksheet Nos. 6, 12, 14, 15, 19, 20, 23-28, 30, and 34-36	
Chris Cornwell	CFA/ Laboratory PM/ Representative for Laboratory and Analytical Issues	910-795-0422		Worksheet Nos. 6, 12, 14, 15, 19, 20, 23-28, 30, and 34-36	
TBD	TBD/ Subcontractor PM/ Driller for DPT Services	TBD		Worksheet Nos. 6, 14, 17, and Figures	

[1] Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.

SAP Worksheet No. 5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— Lines of Communication .....



SAP Worksheet No. 6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
SAP Amendments	Tetra Tech FOL/SSO Tetra Tech PM Navy RPM	George Ten Eyck Tony Klimek Howard Hickey	513-557-5043 513-557-5057 847-688-2600 x243	Tetra Tech FOL will verbally inform Tetra Tech PM within 24 hours of realizing a need for an amendment. Tetra Tech PM will document the proposed changes via a Field Task Modification Request (FTMR) form within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change. SAP amendments will be submitted by Tetra Tech PM to NAVFAC Midwest Program Management Office for review and approval. Tetra Tech PM will send scope changes to Project Team via e-mail within 1 business day.
Changes in field work schedule	Tetra Tech PM NSA Crane ERSM	Tony Klimek Tom Brent	513-557-5057 812-854-6160	Tetra Tech PM will verbally inform the NSA Crane ERSM on the day that a schedule change is known and will document via schedule impact letter within 1 business day of when the impact is realized.
Issues in the field that result in changes in scope of field work	Tetra Tech FOL/SSO Tetra Tech PM NSA Crane ERSM	George Ten Eyck Tony Klimek Tom Brent	513-557-5043 513-557-5057 812-854-6160	Tetra Tech FOL will inform Tetra Tech PM within 1 business day of when an issue is discovered; Tetra Tech PM will inform NSA Crane ERSM by close of the next working day; NSA Crane ERSM will issue scope change if warranted. The scope change is to be implemented before further work is executed. Tetra Tech PM will document the changes within 2 days of identifying the need for change on a FTMR form and obtain required approvals within 5 days of initiating the form.
Recommendations to stop work and initiate work upon corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Tetra Tech Project Chemist Tetra Tech HSM NSA Crane ERSM	George Ten Eyck Tony Klimek Tom Johnston Mark Traxler Matt Soltis Tom Brent	513-557-5043 513-557-5057 412-921-8615 610-382-1171 412-921-8912 812-854-6160	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), NSA Crane ERSM, and the identified Project Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.

Communication Driver	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech PM	George Ten Eyck Tony Klimek	513-557-5043 513-557-5057	Tetra Tech FOL will inform Tetra Tech PM verbally or by e-mail on the same day that a field data quality issue is discovered.
Analytical data quality issues	Empirical Laboratory PM CFA Laboratory PM Tetra Tech Project Chemist Tetra Tech DVM Tetra Tech PM NSA Crane ERSM	Kim Kostzer Chris Cornwell Mark Traxler Joe Samchuck Tony Klimek Tom Brent	615-345-1115 910-795-0422 610-382-1171 412-921-8510 513-557-5057 812-854-6160	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the Tetra Tech DVM/data validation staff and the Tetra Tech PM within one business day. The Tetra Tech DVM or Project Chemist notifies the Tetra Tech PM verbally or via e-mail within 48 hrs of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech PM verbally advises the NAVFAC RPM within 24 hours of notification from the Tetra Tech Project Chemist or DVM. The NAVFAC RPM takes corrective action that is appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the NAVFAC Navy Chemist.

SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role[1]	Organizational Affiliation	Responsibilities
Doug Griffin	RPM/ Provides regulator Input	IDEM	Participates in scoping, conducts data review and evaluation, and approves the SAP.
Tom Brent	ERSM/ Manages daily site activities related to this project	NSA Crane	Oversees site activities, participates in scoping, conducts data review and evaluation, and approves the SAP.
Howard Hickey	RPM/ Manages project	NAVFAC Midwest	Oversees project implementation including scoping and data review and evaluation.
Ralph Basinski	Crane Activity Coordinator/ Coordinates Tetra Tech activities at NSA Crane	Tetra Tech	Oversees project implementation including scoping and data review and evaluation.
Tony Klimek	PM/ Manages project on a daily basis	Tetra Tech	Oversees project, including financial, schedule, and technical day-to-day management of the project.
George Ten Eyck	FOL/SSO/ Manages field operations and oversees site activities to ensure safety requirements are met	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities. As SSO, responsible for on-site project specific health and safety training and monitoring site conditions. Details of these responsibilities are presented in the HASP.
Tom Johnston	QAM/ Oversees program and project QA activities	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Mark Traxler	Project Chemist/ Provides coordination with laboratory	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and QA of data validation deliverables.

Name	Title/Role[1]	Organizational Affiliation	Responsibilities
Joseph Samchuck	DVM/ Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including ensuring QA of data validation deliverables, providing technical advice on data usability, and coordinating and maintaining the data validation review schedule.
Matt Soltis	HSM/ Oversees health and safety activities	Tetra Tech	Oversees the CLEAN Program Health and Safety Program.
TBD	Driller	TBD	Performs DPT soil borings according to scope of work. Provide equipment to collect groundwater samples.
Kim Kostzer Chris Cornwell	Laboratory PM Laboratory PM	Empirical CFA	Coordinates analyses with lab chemists, ensures that scope of work is followed, provides QA of data packages, and communicates with Tetra Tech staff.

[1] In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may also be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

SAP Worksheet No. 8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

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

SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: NSA Crane SWMU 11 RCRA Facility Investigation (RFI) Projected Date(s) of Sampling: April 2011 Project Manager: Tony Klimek	Site Name: SWMU 11 – Old Storage Building 225 Site Location: Crane, Indiana

Date of Session: November 4, 2010
Scoping Session Purpose: Develop project quality objectives (PQOs) for RFI activities

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Tony Klimek	PM	Tetra Tech	513-557-5057	tony.klimek@tetratech.com	Management
Tom Brent	ERSM	NSA Crane	812-854-6160	thomas.brent@navy.mil	Management
Doug Griffin	RPM	IDEM	317-233-2710	dgriffin@idem.in.gov	State RPM
Howard Hickey	Navy RPM	NAVFAC Midwest	847-688-2600 X243	howard.hickey@navy.mil	NAVFAC RPM
Ralph Basinski	Crane Activity Coordinator	Tetra Tech	412-921-8308	ralph.basinski@tetratech.com	Management/ Oversight
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-6470	peggy.churchill@tetratech.com	DQO Facilitator

Comments/Decisions: Discussed SWMU 11 historical use and available data. Discussed the steps for the RFI in accordance with the SAP format.

Action Items: Tetra Tech assigned the task of preparing the Draft SAP.

Consensus Decisions: See meeting minutes in Appendix C. The meeting participants developed PQOs using USEPA's seven-step DQO process. Consensus decisions included the following:

1. Background documents such as the "Green Water" Report will be attached to the SAP.

2. Surface soil samples will be collected from the discharge area of the tank used to store green water after the fire.

3. Groundwater samples will be collected using a direct DPT (direct push technology) rig. Two downgradient and one upgradient samples will be collected. Permanent and/or temporary groundwater monitoring wells will not be installed during the initial screening sampling event.

4. Dioxins/furans samples from the surface soil can be composited by area. They should also be collected from the sediment and from areas downwind of the site. If dioxins/furans are determined to be a concern based on the initial screening sampling event, additional background samples will be collected in a subsequent sampling event.

5. Primarily, surface soil and sediment samples will be collected. Subsurface soil samples will be collected from the Underground Storage Tank (UST) area only. If present and available, two layers of sediment will be collected; 0 to 6 inches below ground surface (bgs) and 6 to 12 inches bgs from each sediment sampling location.

SAP Worksheet No. 10 -- Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

This worksheet presents general background information about SWMU 11 – Old Storage Building 225 and a conceptual site model (CSM) that describes potential contamination routes and possible exposure pathways. The CSM served as the basis for developing the sampling and analysis program.

10.1 PHYSICAL SITE DESCRIPTION

SWMU 11 – Old Storage Building 225 is located near the western boundary of NSA Crane, approximately midway between the northern and southern boundaries of the facility (see Figure 10-1). It is located on the northwest side of Highway 101 approximately 0.4 miles north of the intersection between H-101 and H-45. The site is a rectangular shape and covers approximately 1 acre, as shown on Figure 10-2.

The site is approximately 100 feet wide (southeast-northwest) and 350 feet long (southwest-northeast). Building 225 was destroyed in a fire on July 13, 1976. The site currently consists of the concrete pad from former Building 225, the adjacent concrete loading dock, a railroad siding spur that is adjacent to the loading dock, and grassed and paved areas surrounding the pad. The concrete pad is elevated approximately 2 feet above the adjacent grassed area on the southeastern side. SWMU 11 is bounded on the northwest by a set of railroad tracks and on the southeast by paved road H-101. There is a ditch on the northwestern side of the site that flows northeast; the ditch is between the railroad siding spur and railroad tracks. Building 224 and a gravel parking lot are southwest of the site. Building 2720, Railroad Repair Shop, is located approximately 100 feet west of the site. There are no physical barriers securing the site.

Historical drawings show an UST approximately 40 feet southwest of Building 225. The UST was designed to store fuel for a boiler room that was designed for Building 225 in 1950. According to an historical drawing, the UST was 24 feet long and 5.5 feet in diameter. It was installed by partially excavating the existing ground and then placing soil over the top of the UST to create a mound approximately 3 feet to 5 feet high above the adjoining ground. There was no visible evidence of the UST or the mound during a recent site visit. Presumably, it was removed shortly after the fire. No records were located regarding removal of this UST.

As shown on Figure 10-3, runoff from SWMU 11 drains to the ditch on the northwestern side of the site. The ditch drains to a culvert that conveys flow under multiple railroad tracks to a drainage channel in a wooded area west of the site. The drainage channel discharges to Broom Branch approximately 1,000 feet northwest of the site. Broom Branch then discharges into First Creek approximately 14,000 feet northwest of the site. First Creek then flows northwest and off of the NSA Crane facility approximately 1,000 feet northwest of where they join together.

NSA Crane is in the unglaciated Crawford upland physiographic province of southern Indiana, which is a rugged dissected plateau bordered on the west by the Wabash lowland and on the east by the Mitchell plain. Bedrock geology is mapped as Pennsylvanian and Mississippian sandstones, limestones, and shales overlain by Quaternary-age deposits. Groundwater flow in the area generally mimics topography and is assumed to flow northwest to the drainage channel toward Broom Branch. Depth to groundwater at SWMU 11 is unknown; based on other site investigations in the area, it is expected to be less than 20 feet bgs. The site is located at an elevation of 690 feet above mean sea level (msl). As shown on Figure 10-3, it is located at a topographic high point relative to the surrounding area.

The NSA Crane facility was a rural, forested, and farmed area when it was commissioned as a Navy facility in 1941; the site has been part of the Navy facility since that time. Most of NSA Crane is forested, including the area to the west of the site. There are no known historical or cultural concerns, such as Native American burial grounds or historic landmarks on or in the vicinity of the site. There is no land use controls (LUCs) associated with the site. The nearest residence is located greater than 1 mile to the west of the site, beyond the western boundary of the Facility in the town of Burns City.

10.2 SMWU 11 HISTORY

The exact construction date for Building 225 is unknown; however, it was existing in 1947. Based on historical drawings, a boiler room addition was designed to be added to the southwest side of the building in 1950. The design included the installation of a UST that was 24 feet long and 5.5 feet in diameter approximately 40 feet south of the building. The disposition of the UST is unknown.

Building 225 and its contents were destroyed by a fire on July 13, 1976. At the time Building 225 was destroyed by fire, it was used for inert storage of over 70,000 pounds of pentachlorophenol (PCP), approximately 50,000 gallons of paints, approximately 500 pounds of sodium flourescein dye, approximately 5,000 gallons of solvents (acetone, toluene, and methyl ethyl ketone), and various other items including inks, staples, and wax paraffin. The cause of the fire has been attributed to deterioration of containers which allowed contents to seep out onto cardboard containers and wooden pallets, causing spontaneous ignition. Stored inventory of cellulose nitrate binder and brown wrapping paper is believed to be the source of the fire.

The NSA Crane Fire Department pumped more than 600,000 gallons of water onto the blaze which burned out of control for several hours. The fire was contained to the Building 225 area, and no other buildings with exposure to the fire were threatened. However, the fire extended to grass in the area due to 55-gallon drums and aerosol cans which exploded from the building and landed in the surrounding grassy area. According to the “Fire Investigation” report, a slight northeast wind was reported during the fire (DON, 1976b). Building 225 and its contents were destroyed by the fire. Following the fire, the remaining debris and residue from the Building 225 site were removed and disposed off-site.

The concrete building slab, loading dock, and railroad siding spur have remained in place since Building 225 was destroyed by fire.

10.3 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

Environmental concern was generated by the observation that runoff water used in fighting the fire was colored a brilliant fluorescent green (due to the sodium fluorescein which was stored in the building) and was entering the drainage channel northwest of the site. The drainage channel discharged into Broom Branch, and then into First Creek and off the NSA Crane property. First Creek, which also receives discharge from Lake Greenwood, flows off of NSA Crane and into White River near Newberry, Indiana.

At the time of the fire, Broom Branch had little flow other than runoff from the water used in fighting the fire. PCP, paints, sodium fluorescein, solvents, and other items stored in the building may have entered the surface drainage, and soils in the area of the site, during firefighting efforts, and may continue to be present in these areas. According to the "Green Water" Report (DON, 1976a), a series of dams were constructed on Broom Branch to contain the impacted water shortly after the July 13, 1976 fire. A copy of the Green Water Report is presented in Appendix D. A schematic sketch in the Green Water report shows one dam (Dam #1) on the upstream side of the railroad tracks, and three additional dams (Dams #4, #2, and #3 - upstream to downstream) on Broom Branch downstream of the site and upstream of the discharge point into First Creek. The actual location of the dams is unknown. Dead fish were observed in the green water on July 14, 1976.

On July 14, 1976, water samples were collected from the areas of the dams along Broom Branch and analyzed for the presence of toxic materials. According to the Green Water Report, exhaustive analysis did not reveal any pollutants other than zinc, copper, lead, sodium fluorescein, and PCP. The report concluded that the metals were confined to the area of the site, and the dye was "reasonably non-toxic," leaving PCP as the primary contaminant of concern (COC). On July 14, 1976, PCP concentrations at the site were reported at greater than 30 milligrams per liter (mg/L), they were 30 mg/L in Broom Branch just below the railroad tracks, and were not reported in more downstream locations.

On July 14, 1976, water near the building was pumped out and transported to a concrete holding pond located near the Burns City Gate approximately 1 mile southwest of the site, as shown on Figure 10-1. The "Green Water Report" stated that several thousand gallons of water was pumped from the site and placed in a concrete reservoir to be properly disposed at a later date. The "Green Water" Report does not specifically identify the concrete reservoir in which the water was placed. The Building 2981 concrete tank was determined to be the most likely location of the concrete reservoir described in the "Green Water" Report. The Building 2981 concrete pond was the only concrete reservoir known to exist at the time of the fire and had a large capacity; therefore, it is assumed that this is the concrete reservoir referenced in the "Green Water" Report. The Building 2981 concrete tank was built as a sonobuoy test pond and has since been replaced with a smaller concrete test structure in the same location. The

approximate location of the former concrete tank is shown on Figure 10-4. On July 15, 1976, water from Broom Branch was pumped into fuel storage bladders for containment.

During the afternoon of July 15, 1976, heavy precipitation added an estimated 10 to 20 million gallons of water to the watershed. As a result, the water in Broom Branch flowed around Dam #2, and Dam # 3 was washed away. Visibly green water left the NSA Crane property. The visible limit of the dye is 0.2 mg/L; the highest value recorded off NSA Crane property was 0.4 mg/L. Flow in the channel receded and flow around Dam #2 stopped on July 17, 1976.

Contaminant concentrations decreased as the water moved downstream. As previously mentioned, the PCP levels near the site were reported at greater than 30 mg/L (or 30,000 parts per billion [ppb]), and at 30,000 ppb just downstream of the railroad tracks on July 14, 1976. Further downstream the PCP levels in the reservoir above Dam # 2 were typically 60 to 100 ppb. PCP levels in the water collected in the fuel bladders were 10 to 80 ppb.

Water quality at the three bridges across First Creek downstream of NSA Crane property and upstream of the White River was continuously monitored. The highest PCP level in First Creek at an offsite bridge (#2) downstream of NSA Crane was less than 10 ppb.

Beginning on July 19, 1976, the water contained in the reservoir behind Dam # 2 was released in a controlled manner into Broom Branch downstream of Dam #2. At the same time, the outflow from Lake Greenwood was manipulated to control the flow in First Creek so that no green water would be visible leaving the NSA Crane property. During this process, green colored water was visible leaving NSA Crane at only one time. The PCP level in the water leaving NSA Crane did not exceed 20 ppb. Water contained in the fuel bladders was also discharged in the same manner. The final disposition of the water collected and stored in the concrete reservoir is not known.

10.4 CONCEPTUAL SITE MODEL

Releases as a result of the fire in Building 225 are the likely source of potential contamination at SWMU 11. The historical information and analytical results from the “Green Water” Report (DON, 1976a) indicate that releases likely occurred directly onto the surface soil and potentially the subsurface soil, as well as migrating in the ditch and drainage channel sediment and floodplain soil following the hydrology for runoff. The site had been used as a storage facility of various metals, solvents, paints, and combustibles. Previous analytical results provide evidence that there was a release of zinc, copper, lead, sodium fluorescein, and PCP in the runoff, with all pollutants being confined, with the exception of PCP. An additional environmental concern is the possibility of residual contaminants from particulates resulting from the fire smoke that may have settled downwind of the site. The Report of Fire Investigation (DON, 1976b) reports that a northeast wind was blowing at 7 miles per hour during the fire, which may have dispersed airborne particulates in a southwesterly direction . The surface soil, subsurface soil, sediment,

and groundwater had not been tested. A CSM schematic is presented on Figure 10.5; the schematic is a generalized cross-section of the site facing southwest.

Based on site topography and the historical information of the fire, areas of interest, identified as Investigation Areas (IAs), include the following (see Figures 10-3 and 10-4):

- IA-1 - Building 225 Sub-slab
- IA-2 - Ditch (on northwest side of site)
- IA-3 - Adjacent Areas
- IA-4 – Downwind Areas
- IA-5 - UST Area
- IA-6 – Downgradient Area of Concrete Tank
- IA-7 - Drainage Channel

10.4.1 Potential Sources and Contaminants of Concern

Based on historical information, the fire, including firefighting measures, led to environmental exposure from the chemicals stored in Building 225. Because the list of chemicals may not be complete, the proposed sampling strategy for this site includes a wide range of potential contaminants. The target analytes for this investigation were selected from the list of priority pollutant chemicals and is based on the contaminants suspected to be present at the site during the fire and those known to be present at other NSA Crane sites. The analytical groups that will be investigated include: volatile organic compounds (VOCs); semivolatile organic compounds (SVOCs), including low level polycyclic aromatic hydrocarbons (PAHs); pesticides; polychlorinated biphenyls (PCBs); dioxins/furans; metals, and cyanide for surface soil, subsurface soil, sediment, and groundwater. Worksheet No. 15 identifies the target analytes that are included in each analytical group for each type of environmental media.

10.4.2 Potential Migration and Exposure Pathways

After release to the soil, contamination may (1) result in a complete exposure pathway to human and ecological receptors, and/or (2) serve as a source of contamination to groundwater and surface water and result in a complete exposure pathway through those routes. The impacts of contamination reaching First Creek may result in direct exposure to receptors in and along the stream. Floating and dissolved contaminants may also have soaked into ditch and channel areas to act as a contaminant reservoir for migration to groundwater and result in direct exposure to contaminated soil and sediment. Due to the firefighting measures and weather conditions in days following the fire, contamination affecting sediment in ditch and channel areas may have also soaked into the surrounding floodplain soil. The only evidence of previous contamination, however, was to surface water which was later washed out by a storm event. Residual contaminants in the form of particulates resulting from the fire smoke may have settled downwind of the site. Potential exposure pathways are illustrated on Figure 10-6 and Figure 10-7 for human and ecological receptors, respectively.

10.4.3 Potential Receptors

Human receptors at SWMU 11 include people who currently, or could in the future, interact with contaminated media. Current site users include workers in Buildings 2720 and 224 located within 100 feet of the site, and trespassers. The area is rural, and the nearest residence is more than 1 mile from the site. However, because future land use is unknown, it is common practice to evaluate the future use of a property as residential. Therefore, potential future receptors include residents and persons recreating at the site. Human receptors may be exposed to different media based on their specific activities. These media include surface and subsurface soil, sediment, and groundwater.

Ecological receptors include animal and plant species that could be affected by the contaminants that are present at the site. Typically, ecological receptors can be exposed only to surface media – surface soil, surface water, and upper layers of wetland or stream sediment. Exposure of ecological receptors to groundwater and subsurface soil is not anticipated; however, contamination in subsurface soil or groundwater may serve as sources of contamination to sediment or surface water through subsurface transport or diffuse flow to streams. The exposure media for ecological receptors are surface soil and sediment. Terrestrial plants, invertebrates, and vertebrates are exposed to surface soil by direct contact and ingestion of soil and other food items. Aquatic and semi-aquatic vegetation, benthic invertebrates, and aquatic organisms are exposed to sediment by direct contact and/or ingestion of sediment and other food items. Benthic invertebrates or other aquatic organisms that may have contacted or ingested contaminated media may be consumed by wildlife. Although terrestrial vertebrates may be exposed to chemicals found in the air via inhalation, this is not considered a significant pathway.

SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

This section describes the development of PQOs using USEPA's seven-step DQO/Systematic Planning Process.

11.1 PROBLEM STATEMENT

Based on site history and the CSM, it is unknown whether site-related contaminants are present in environmental media at SWMU 11 at concentrations that exceed applicable risk-based human health or ecological screening values. The data from the initial screening sampling event will be used with supplemental investigation data as deemed necessary by Project Team to locate contaminated areas, and to conduct a Human Health Risk Assessment (HHRA) and/or an Ecological Risk Assessment (ERA), If there is unacceptable risk to ecological or human receptors form exposure to site contaminants the project team will evaluate remedial alternatives in a Corrective Measures Study.

11.2 IDENTIFY THE INPUTS TO THE DECISIONS

The following chemical and physical data are needed to attain project objectives:

1. Chemical data: Surface and subsurface soil, sediment, and groundwater chemical data will be collected and analyzed to determine if target analytes are present in site media. The list of target analytes and associated Project Screening Levels (PSLs) for each matrix are presented in Worksheet No. 15. The sampling methods are presented in Worksheet No. 18, and the analytical methods are presented in Worksheet No. 19.

2. Subsurface soil screening: A photoionization detector (PID) will be used to measure VOCs and certain SVOCs (including PAH) levels in subsurface soil samples. Analyses will be conducted on the most contaminated subsurface soil interval at each boring location, based on the maximum PID reading. Visual observations will also be used to assist in the identification of subsurface soil with the greatest potential for contamination. The PID will be used in accordance with the manufacturer's guidance.

3. Physical: Well stabilization parameters will be measured during groundwater sampling to ensure that representative groundwater data is collected and to support risk calculations, if they become necessary.

4. Project Screening Levels (PSLs): The SWMU 11 RFI requires chemical data that can be compared to current USEPA and IDEM residential surface and subsurface soil, sediment, and groundwater risk-based screening criteria. The risk and regulatory criteria applicable to the SWMU 11 RFI include the IDEM Risk Integrated System of Closure (RISC) Default Closure Tables, Residential and Industrial

Closure Levels (R-DCLs and I-DCLs) (IDEM, 2009); USEPA Regions 3, 6 and 9 Residential Regional Screening Levels (R-RSLs) and risk-based migration-to-groundwater Soil Screening Levels (RBSSLs) for human health risk screening (USEPA, 2010a). ERA PSLs were selected by choosing the lowest value among USEPA Ecological Soil Screening Levels (Eco-SSLs) for plant, invertebrate, mammalian and avian values and this was selected as the ecological screening value (USEPA, 2005-2008). Eco-SSLs were used preferentially as soil screening values; however, Eco-SSLs are currently available for only a few analytes. USEPA Region 5 Ecological Screening Levels (ESLs) for soil (R5 ESL-S) and sediment (R5 SED) (USEPA, 2003) were used for screening values for analytes that do not have an Eco-SSL. The criterion for each analyte represents the PSL for each environmental matrix listed in Worksheet No. 15. A comprehensive list of the relevant environmental and medium-specific PSLs for the target analytes is provided in Appendix E.

To conduct comparisons of site data to screening values for surface soil, subsurface soil, sediment, and groundwater, the selected laboratory(s) should be able to achieve Limits of Quantitation (LOQs) that are low enough to measure constituent concentrations that are less than the PSLs.

Analytical data reported by the laboratory use the following reporting conventions: All results below the Detection Limit (DL) will be considered nondetects; positive results reported at concentrations between the DL and LOQ will be reported with a “J” qualifier; and analytes not found (not detected) in a sample will be reported at the Limit of Detection (LOD) with a "U" qualifier.

Several target analytes have PSLs that fall between the LOD and the LOQ. “J” flagged data will be accepted to achieve project goals; however, greater scrutiny will be applied in these cases. Additionally, the inability to quantify select analytes to PSL levels with confidence will be addressed in the risk screening uncertainty analysis. In cases where the laboratory LODs are greater than the PSLs, consistent with the USEPA Risk Assessment Guidance for Superfund, Part A (USEPA, 1989), if the analyte is not detected, the LOD will be reported and "U" qualified. An evaluation of these analytes will be also presented in the uncertainty section of the risk screening in the RFI Report.

5. Background: The background data set for various media at NSA Crane will also be used to determine whether metals present on-site are naturally occurring or are site-related. Background data for the various soil types identified at NSA Crane are described in the *Final Base-Wide Background Soil Investigation Report for NSWC Crane* (Tetra Tech, 2001). For risk assessment purposes, in the event that an analyte concentration exceeds a PSL, but is less than or equal to an established background concentration, the analyte will not be considered a COPC. For determining the extent of contamination, if a background concentration for a particular analyte is greater than the PSL for that analyte, the background concentration will replace the PSL prior to decision-making.

11.3 DEFINE THE STUDY BOUNDARIES

Based on the CSM, the study will be performed in a step-wise manner and will address the area of SWMU 11 as shown on Figure 10-3, and the area adjacent to the Building 2981 concrete tank as shown on Figure 10-4. Specific Investigative Areas

The following items address the horizontal, vertical, and temporal boundaries for the initial screening sampling event of the study:

1. Horizontal: The horizontal boundary of the study is defined as the outer perimeter of the area where the Building 225 fire took place and the outlet area of the Building 2981 Concrete Tank, based on information from aerial photographs and previous investigations. Horizontally, the area within SWMU 11 boundaries and drainage channel to Broom Branch will be investigated. Surface water is not of interest because the drainage channel is dry during part of the year. Lateral expansion of the horizontal study boundary may be necessary if concentrations in site media exceed PSLs (and background concentrations for metals) in the initial sampling round.

2. Vertical: The vertical boundary of the study is defined as soil from the surface to the top of the water table, which is estimated at less than 20 feet bgs at the site. Vertically, both surface and subsurface soil will be assessed. The interval of interest for surface soil is 0 to 2 feet bgs; the interval of interest for subsurface soil is a 2-foot interval selected based on field screening with a PID and/or visual observations. Surface soil data will be used for direct contact soil risk evaluations. Subsurface soil data will be collected at greater than 2 feet bgs to investigate for potential migration-to-groundwater.

3. Downwind Dioxin/Furan Investigation Area: A select area downwind of the fire smoke will be sampled for dioxins/furans. PSL exceedances at these locations would represent residual contaminants from particulates resulting from the fire smoke that may have settled downwind of the site. As such, EU boundaries and EPCs to represent the IA-4 Downwind Area will be addressed separately, if deemed necessary by the Project Team.

4. Groundwater: Groundwater downgradient of the known release may have been contaminated by infiltration of the runoff from firefighting measures. Upgradient groundwater samples will also be collected to provide a reference population. Groundwater will be assessed at the site by collecting groundwater samples during initial sampling. Because there are no known monitoring wells in the immediate vicinity, depth to groundwater is not known, but it is presumed to be less than 20 feet bgs based on other site investigations in the area.

5. Temporal: All target analyte concentrations are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future; therefore, no temporal constraints exist. SWMU 11 RFI initial sampling event field activities

are scheduled for early 2011. Subsequent field sampling activities, if deemed necessary based on the initial sampling event results, will be conducted in a timely manner.

The study area boundary may expand if COPCs are identified in relative site media.

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11.4 DEVELOP THE ANALYTIC APPROACH

The decision statements for each step of the study are as follows:

Initial Screening

Based on the results from the initial screening sampling event, determine whether chemical concentrations in site media (surface and subsurface soil, sediment, and groundwater) exceed the PSLs within and around areas that are the most likely areas to have been impacted by the fire at SWMU 11. If chemical concentrations in site media are less than the lowest risk-based screening values, then the Project Team will recommend No Further Action (NFA) for the site, and the Navy will submit a Closure Report to IDEM.

If any analyte is detected in site media at a maximum concentration that exceeds a risk-based screening value and is greater than the site-specific background concentration (for metals), the Project Team will meet to discuss a path forward. The Project Team will review the analytes that exceed the risk-based screening values based on specific factors that include the following:

- The environmental media that is identified with an exceedance,
- The particular compound(s) that is identified with an exceedance, and
- The magnitude of any exceedance, in frequency, distribution of samples, and concentrations as compared to the screening value.

The decision rules for the initial screening sampling event of this investigation are as follows:

1. If target analyte concentrations in all surface and subsurface soil and sediment samples in the initial round of sampling are less than PSLs, then recommend NFA.
2. For each target analyte, if the maximum measured concentration in any medium exceeds its human health or ecological screening value (and applicable background concentration for metals), then classify the chemical as a human health or ecological Chemical of Potential Concern (COPC) for that medium and risk type and collect another round of data in order to conduct a human health and ecological risk assessment; otherwise, exclude the chemical from further consideration in the risk screening.

3. For target analytes that are detected in groundwater, if the maximum detected concentration of any analyte exceeds the PSL, then installation of permanent groundwater monitoring wells will be considered by the Project Team to assess and monitor groundwater conditions at the site; otherwise, no additional groundwater samples will be required.

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Delineation of Exceedances of PSLs (and Background Concentrations for Metals)

If any COPCs are identified in soil, sediment or groundwater following the initial sampling event, then the Project Team will return to the site to collect one round of "step-out" samples (vertical or horizontal) to define the vertical and/or horizontal extent of COPC contamination. The second round of data will be used with the first round of data to evaluate risk to site users and to identify Contaminants of Concern (COCs) for presentation in the RFI Report. Delineation of COCs will be completed in the future if active remediation is required, which will be determined through an evaluation of corrective measures in the Corrective Measures Study (CMS).

Risk Assessment

Once COPCs have been identified and the second round of data is collected risk to site users will be evaluated. If risks are not unacceptable, as determined through the human health and ecological risk assessments, the Project Team will meet to discuss a path forward. Such a path would involve one or more of the following:

- Comparing chemical concentrations in site media to Industrial or Non-Default Closure (less conservative) criteria as potential options.
- Addressing exceedances as uncertainties in the risk assessment and a Corrective Measures Study (CMS) and implementing risk management options such as institutional or engineering controls.

To evaluate potential risk associated with exposure to soil and sediment, soil and sediment data collected across the defined EUs will be used to define EPCs, depending on the receptor for which the risk is being evaluated. For industrial and residential receptors, the soil data collected from subdivided EUs will provide the basis for determining the soil EPCs. These smaller subunits are more representative of the potential exposure associated with industrial and residential receptors. Trespasser, construction worker, and recreational user EUs will be defined as the entire area between Highway 101 and the railroad tracks, with a linear distance of approximately 430 feet (as shown on Figure 10-2). Therefore, data collected across the entire site will provide the basis for determining the EPCs for those receptors.

Data that are considered to be representative of current site conditions, which will include all initial screening samples and one round of supplemental "step-out" samples and random background samples that are needed to adequately define the levels of risk present on-site from COCs will be used in the risk assessment(s). Human health risks will be developed in accordance with USEPA Risk Assessment Guidance (USEPA, 1989), and ecological risks will be developed in accordance with USEPA Ecological Risk Assessment Guidance (USEPA, 1997).

For soil, average concentrations, as represented by the 95-percent upper confidence limit (UCL) of the arithmetic mean, will be determined for each EU, specific to the human receptor which it represents. The UCL will be determined using USEPA's ProUCL software (Version 4.00.05, or most current) and will be used to represent the EPC for soil. Surface soil, combined surface and subsurface soil, and sediment concentrations will be computed for the HHRA. For groundwater, maximum detected concentrations will be used to represent EPCs.

If COPC concentrations in the receptor exposure units defined for any medium represent an unacceptable human health risk, then proceed to a CMS; otherwise, recommend NFA from a human health perspective. Unacceptable human health risk is defined for this project as incremental lifetime cancer risk (ILCR) estimates exceeding the risk range of $1x10^{-6}$ to $1x10^{-4}$, or a non-cancer risk (i.e., hazard indices) exceeding 1 (on a target-organ specific basis). Risk management decisions will be made for risk from carcinogens that is within the acceptable range of $1x10^{-4}$ and $1x10^{-6}$.

For ecological risk screening, the maximum detected concentrations of each COPC in all surface soil, sediment, and groundwater samples will be compared to PSLs to determine if an analyte is a Chemical of Potential Ecological Concern (COPEC). Average concentrations (arithmetic means) of surface soil data

or sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), then the Project Team will determine the need to conduct a Baseline Ecological Risk Assessment (ERA) or consider the potential risks with respect to remedial actions.

For COCs detected in groundwater, if the maximum measured concentration of any COC exceeds the PSL, then permanent groundwater monitoring wells will be considered by the Project Team for installation to assess and monitor groundwater conditions at the Site.

11.5 SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA

Because the initial screening sampling event sample locations depend on biased sampling, probability limits for false positive and false negative decision errors were not established for soil, sediment, or groundwater samples. Simple comparisons of measured concentrations to PSLs will be used. Sample locations were selected to determine the nature of surface and subsurface soil, sediment, and groundwater contamination from areas most likely to be contaminated based on the CSM.

This biased selection of sample locations does not support the use of quantitative statistics to estimate decision performance, as specified in the USEPA QA/G-4, QA/G-5, and QA/G-5S DQO guidance documents (USEPA, 2006, 2002a, and 2002b, respectively). However, the quantity of samples to be collected in the initial sampling round is deemed sufficient by the Project Team to determine whether unacceptable environmental conditions are present.

The Project Team will use the data from the initial screening sampling event, and additional data collected to conduct risk assessment, as well as additional random background data to determine whether the amount and type of data collected are sufficient to support the attainment of project objectives, which include the identification of COPCs to support the planned risk assessment calculations for human health and ecological receptors. This process may involve an evaluation of contaminant concentrations and uncertainty for contaminants that have PSLs which are below the LODs to ensure that contaminants are likely to have been detected, if present. If all data have been collected as planned and no data points are missing or rejected for quality reasons, the investigation will be considered satisfactory. If any data gaps are identified, including missing or rejected data, the Project Team will assess whether the project objectives have been obtained. This assessment will depend on the number and type of identified data gaps; therefore, a more detailed strategy cannot be presented at this time. All stakeholders, including the IDEM RPM, the Navy RPM and ERSM, and the Tetra Tech PM, will be involved in rendering the final conclusion by consensus regarding adequacy of the data. All analytical data collected per the sampling design should meet the QA criteria established in Worksheet Nos. 19 through 37 and the prescribed detection limit requirements for each COPC.

11.6 DEVELOP THE PLAN FOR OBTAINING DATA

Based on the information presented above, a detailed plan was developed to obtain the necessary data to answer the problem for the RFI. The sampling design and rationale for all initial screening sampling event samples, “step-out” samples, and random background HHRA/ERA support samples that will be collected are provided in Worksheet No. 17.

SAP Worksheet No. 12 -- Measurement Performance Criteria Table – Field Quality Control Samples

(UFP-QAPP Manual Section 2.6.2)

Quality Control (QC) Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Trip Blank	VOCs	One per cooler of VOC samples shipped to laboratory.	Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Equipment Rinsate Blank	All analytical groups	One per 20 samples per matrix per sampling equipment[1].	Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Filtered Rinsate Blank	Dissolved Metals (if necessary due to high turbidity)	One per filter brand[2].	Bias/ Contamination	No analytes ≥ ½ LOQ, except common laboratory contaminants, which must be < LOQ.	S & A
Field Duplicate (FD)	All analytical groups	One per 10 field samples.	Precision	Values > 5X LOQ: Relative Percent Difference (RPD) must be ≤30 (aqueous)[3,4] ; ≤50 (solids)[3,4].	S & A
Cooler Temperature Indicator	All analytical groups	One per cooler.	Representativeness	Temperature must be less than 6 degrees Celsius (<6 °C).	S

1 – Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
2 – A filtered rinsate blank will be collected if filtered samples (e.g., Dissolved Metals) are collected.
3 – If duplicate values for non-metals are < 5x LOQ, absolute difference should be < 2x LOQ.
4 – If duplicate values for metals are < 5x LOQ, absolute difference should be < 4x LOQ.

SAP Worksheet No. 13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Aerial Photographs	Aerial Photographic Site Analysis, NSA Crane, Crane, Indiana	USEPA, Characterization Research Division, 2005	Data will be used to generate approximate storage building location on topographic or geographical information system maps and to select proposed boring locations at SMWU 11.	None.
"Green Water" Report	Green Water Report for SWMU 11, Naval Weapons Support Center, Crane, Indiana	Department of the Navy, September 20, 1976	Data was used to identify IAs.	Due to the age of data, data will not be used in risk calculations. The data would not represent current site conditions.
Initial Assessment Study	Initial Assessment Study of Naval Weapons Support Center, Crane, Indiana	Naval Energy and Environmental Support Activity, May 1983	Data was used to identify IAs.	Due to the age of data, data will not be used in risk calculations. The data would not represent current site conditions.
Background Metals Study	Basewide background Soil investigation report, naval Surface warfare Center crane, Crane, Indiana	Naval facilities Engineering Command, January 2001	Data may be used to recalculate environmental risks.	None, the data were fully validated.

SAP Worksheet No. 14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASK PLAN

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

- Mobilization/Demobilization
- Site-Specific Health and Safety Training
- Utility Clearance
- Monitoring Equipment Calibration
- Sample Collection Tasks
- Surface and Subsurface Soil Boring Sampling
- Sediment Sampling
- Groundwater Well Installation and Development
- Groundwater Sampling
- Investigation-Derived Waste (IDW) Management
- Global Positioning System (GPS) Locating
- Field Decontamination Procedures
- Field Documentation Procedures
- Sample Custody and Shipment Tasks
- Quality Control Tasks

Mobilization/Demobilization

Mobilization will consist of the delivery of all equipment, materials, and supplies to the site, complete assembly in satisfactory working order of all such equipment at the site, and satisfactory storage at the site of all such materials and supplies. Tetra Tech will coordinate with the Navy to identify appropriate locations for the storage of equipment and supplies. Site-specific health and safety training for all Tetra Tech subcontractors will be provided as part of site mobilization.

Demobilization will consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the conduct of the investigation.

Site-Specific Health and Safety Training

There are no specialized/non-routine project specific training requirements or certifications needed by personnel to successfully complete the project or tasks. All field personnel will have appropriate training to conduct the field activities to which they are assigned. Each site worker will be required to have

completed the OSHA 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training. Safety requirements are addressed in greater detail in the site-specific HASP.

Utility Clearance

Prior to commencing any work at NSA Crane, the Comprehensive Work Approval Process (CWAP) will be followed. The CWAP will identify constraints in the work area, such as the locations of eagle's nests, archaeological sites, wetlands, etc., that may affect work at the site and other requirements that must be met prior to commencing work. One week prior to the commencement of any subsurface intrusive activities, the Tetra Tech FOL or designee will contact Indiana Underground Plant Protection Services (IUPPS) to complete a utility clearance ticket for the areas under investigation. Work permits, if required by the facility, will be obtained prior to conducting field activities. The Tetra Tech FOL will be responsible for coordinating these activities.

Monitoring Equipment Calibration

These procedures are described in Worksheet No. 22.

Sample Collection Tasks

Site-specific Standard Operating Procedures (SOPs) have been developed for field activities at this NSA Crane site, including sample collection tasks. These SOPs are presented in Appendix A. Sample labeling will be in accordance with SOP-02 (Sample Labeling, Appendix A), and the sample numbering scheme will be in accordance with SOP-03 (Sample Identification and Nomenclature, Appendix A). Methods for recording data will be in accordance with SOP-04 (Sample Custody and Documentation of Field Activities, Appendix A), and the selection of sample containers, sample preservation, packaging, and shipping will be in accordance with SOP-05 (Sample Preservation, Packaging, and Shipping, Appendix A).

The sampling and analysis program is outlined in Worksheet No. 18, and the sampling requirements for each type of analyses (i.e., bottleware, preservation, holding time) are listed in Worksheet No. 19. Field and laboratory QC samples will also be collected as outlined in Worksheet No. 20.

Surface and Subsurface Soil Sampling

Surface soils at NSA Crane are identified as the top 2 feet of soil (from 0 to 2 feet bgs). Surface soil does not include surface pavement or gravel. The ground surface will begin at the bottom of a pavement or gravel layer. The demarcation line between gravel and surface soil will be where more than fifty percent of the material is soil based on a visual assessment by the sampler. At locations where samples are

collected through a pavement layer, the pavement will be replaced in kind prior to demobilization of field activities.

Soil samples will be collected in accordance with SOP-07 (Soil Coring and Sampling Using Hand Auger Techniques, Appendix A) and SOP-11 (Subsurface Soil and groundwater Sampling Using DPT, Appendix A). Surface soil samples (from 0 to 2 feet bgs) will be collected with a hand auger, backhoe, or DPT, depending on site conditions. Sample jars will be filled using either a decontaminated stainless steel trowel or dedicated disposable plastic trowel. Subsurface soil samples will be collected using a DPT rig, DPT rig with auger, or backhoe, and stainless steel or disposable trowel. The subsurface soil borings will be described by the Site Geologist in accordance with SOP-08 (Soil Sample Logging, Appendix A) and will be screened for evidence of contamination with a PID. Use of the PID will be in accordance with the manufacturer's instructions. Any qualitative visual signs of potential contamination (such as soil staining) will be noted on the soil boring log.

Sediment Sampling

Sediment samples will be collected from two depths – 0 to 6 inches bgs, and 6 inches bgs to 12 inches bgs. The sediment sampling procedures discussed in SOP-09 (Sediment Sampling, Appendix A) will be followed.

Groundwater Sampling

Initial screening groundwater samples will be collected using a DPT drill rig as described in SOP-11 (Subsurface Soil and Groundwater Sampling Using Direct-Push Technology, Appendix A).

Groundwater Well Installation and Development

If determined to be necessary by the Project Team based on the initial screening sampling event results, additional groundwater wells will be installed in accordance with SOP-12 (Monitoring Well Installation, Appendix A). Development and sampling of the wells will then be performed in accordance with SOP-13 (Monitoring Well Development, Appendix A), SOP-14 (Measurement of Water Levels, Appendix A), SOP-15 (Low Flow Well Purging and Stabilization, Appendix A), SOP-16 (Monitoring Well Sampling, Appendix A), and SOP-17 (Calibration and Care of Water Quality Meters, Appendix A).

Investigation-Derived Waste Management

It is not anticipated that significant volumes of solid or semi-solid IDW in the form of soil or sediment will be generated during field activities, including collection of surface and subsurface soil samples using DPT or backhoe excavations. Soil will be replaced into the excavation from which it was excavated. If gross contamination is encountered (e.g., any non-soil contaminated material such as free product or soil with PID readings greater than 100 parts per million [ppm]), then excavation will cease. Any grossly

contaminated material that is brought to the surface will not be returned to the excavation but will be segregated from other excavated soil and placed on a plastic liner. The grossly contaminated material will be securely staged until arrangements are made for proper off-site disposal.

IDW that is generated, including personal protective equipment (PPE) and decontamination fluids, will be handled in accordance with SOP-10 (Management of Investigation-Derived Waste, Appendix A).

Global Positioning System Locating

A GPS unit will be used to locate all sampling points in accordance with SOP-01 (Global Positioning System, Appendix A). The GPS equipment will be checked on control monuments before and after day's use, and these checks will be documented in the field notebook. To ensure sub-meter accuracy, the GPS SOP requires a minimum of six satellites to capture a position.

Field Decontamination Procedures

Sample containers will be provided certified clean (I-Chem 300 or equivalent) from Empirical and CFA. Decontamination of sampling equipment will not be necessary for this project if only dedicated and disposable hand trowels will be used. However, if decontamination is necessary, the requirements outlined in this section will apply. Decontamination of reusable sampling equipment (e.g., non-disposable hand trowels, hand augers, or DPT or backhoe equipment) will be conducted prior to sampling and between samples at each location. Decontamination of equipment will be conducted according to the sequence established in SOP-06 (Decontamination of Field Sampling Equipment, Appendix A).

Field Documentation Procedures

Field documentation will be performed in accordance with SOP-04 (Sample Custody and Documentation of Field Activity, Appendix A).

A summary of all field activities will be properly recorded in a bound logbook with consecutively number pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use. Field documentation will be performed in accordance with SOP-04 (Sample Custody and Documentation of Field Activity, Appendix A).

Sample Custody and Shipment Tasks

Sample custody and shipment tasks are defined in SOP-05 (Sample Preservation, Packaging, and Shipping, Appendix A) and are discussed in Worksheet No. 27.

Quality Control Tasks

QA/QC samples will be collected at frequencies listed in Worksheet No. 12.

14.2 ADDITIONAL PROJECT-RELATED TASKS

Additional project-related tasks include:

- Analytical tasks
- Data generation procedures
- Data handling and management
- Data tracking and control
- Assessment and oversight
- Data review
- Project reports

Analytical Tasks

Chemical analyses for VOCs, SVOCs (including low level PAHs), PCBs, pesticides, metals, cyanide, and total organic carbon (TOC) will be performed by Empirical, which is a current Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accredited laboratory. Chemical analyses for dioxins/furans will be performed by CFA, which is also a current DoD ELAP accredited laboratory. Copies of the DoD ELAP accreditation for Empirical and CFA are included in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 30. Empirical and CFA will meet the PSLs specified in Worksheet No. 15 and will perform the chemical analyses following laboratory-specific SOPs (see Worksheet Nos. 19 and 23) that were developed based on the methods listed in Worksheet Nos. 19 and 30. Copies of laboratory SOPs are included in Appendix B.

All soil results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and associated electronic data deliverables (EDDs). This information will also be captured in the project database, which will eventually be uploaded to Naval Installation Restoration information Solution (NIRIS). Percent moisture information will also be captured in the RFI Report.

The analytical data packages provided by Empirical and CFA will be in a contract laboratory program-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.).

Data Generation Procedures

- Project documentation and records include the following:
  - Field sample collection and field measurement records as described in Worksheet Nos. 27 and 29.
  - Laboratory data package deliverables as described in the analytical specifications.
  - Data assessment documents and records as listed in Worksheet No. 29.
- Data recording formats are described in Worksheet No. 27.

Data Handling and Management

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

Data Tracking and Control

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

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

- **Data Storage, Archiving, and Retrieval**. The data packages received from Empirical and CFA are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech Navy CLEAN file system and archived in secure files. The field records including field log books, sample logs, chain-of-custody records, and field calibration logs will be submitted by the Tetra Tech FOL to be entered into the Navy CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.

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

Assessment and Oversight

Refer to Worksheet No. 32 for assessment findings and corrective actions and to Worksheet No. 33 for QA Management Reports.

Data Review

Data verification is described in Worksheet No. 34, data validation is described in Worksheet Nos. 35 and 36, and usability assessment is described in Worksheet No. 37.

Project Reports – Draft and final versions of project reports will be prepared and submitted to the Navy and IDEM for review. The reports will include the following sections:

- Executive Summary – will include a brief description of the work conducted and the findings.

- Introduction and Background – will include a description of the history of operations and activities at the site and a summary of any previous investigations and removal actions.

- Description of Field Investigations – will include a summary of the work performed in accordance with the approved SAP and any field modifications as documented by the Tetra Tech FOL. This section will include maps showing the sampling locations and tables summarizing the data collected.

- Data Quality – will include a summary of quantitative analytical performance indicators such as completeness, precision, accuracy, bias, and sensitivity and qualitative indicators such as representativeness and comparability. This section includes a reconciliation of project data with the DQOs and an identification of deviations from this SAP.

  A data usability assessment will be used to identify significant deviations in analytical performance that could affect the ability to meet project objectives. The elements of this review are presented in Worksheet No. 37.

- Nature and Extent of Contamination – will include a discussion of the contamination detected in each medium sampled in relation to the CSM of the site. This section will note the removals previously conducted (if applicable), contamination addressed, and any additional contaminants found during

this field effort. Detected contaminant concentrations will be tabulated for each medium and depicted on maps.

- Contaminant Fate and Transport – will include a description of the contaminants detected and their behavior in soil, bedrock, groundwater, surface water, and sediment, particularly with emphasis on the future migration of these contaminants to any possible exposure areas.

- Summary and Conclusions – includes a summary of the findings, conclusions as to whether delineation of contamination is adequate, and recommendations for further investigations, if needed.

Tetra Tech will submit the draft report and respond to comments received on the draft report before any additional sampling begins. The final version of the report will submitted in hardcopy and electronic format to the project stakeholders.

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SAP Worksheet No. 15 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil
Analytical Group: VOCs

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1,1-Trichloroethane	71-55-6	1.9	29.8	R-DCL	R5 ESL-S	0.63	0.005	0.0025	0.00125
1,1,2,2-Tetrachloroethane	**79-34-5**	**0.00052**	0.127	**RBSSL**	R5 ESL-S	**1.7E-04**	**0.005**	**0.0025**	**0.00125**
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	3,000	NC	RBSSL	None	1,000	0.005	0.0025	0.00125
1,1,2-Trichloroethane	**79-00-5**	**0.0016**	28.6	**RBSSL**	R5 ESL-S	**5.3E-04**	**0.005**	**0.0025**	**0.00125**
1,1-Dichloroethane	75-34-3	0.014	20.1	RBSSL	R5 ESL-S	0.0047	0.005	0.0025	0.00125
1,1-Dichloroethene	75-35-4	0.058	8.28	R-DCL	R5 ESL-S	0.019	0.005	0.0025	0.00125
1,2,3-Trichlorobenzene	87-61-6	1.7	NC	RBSSL	None	0.57	0.005	0.0025	0.00125
1,2,4-Trichlorobenzene	120-82-1	0.14	11.1	RBSSL	R5 ESL-S	0.047	0.005	0.0025	0.00125
1,2-Dibromo-3-chloropropane (DBCP)	**96-12-8**	**0.0000028**	0.0352	**RBSSL**	R5 ESL-S	**9.3E-07**	**0.005**	**0.0025**	**0.00125**
1,2-Dibromoethane (EDB)	**106-93-4**	**0.000036**	1.23	**RBSSL**	R5 ESL-S	**1.2E-05**	**0.005**	**0.0025**	**0.00125**
1,2-Dichlorobenzene	95-50-1	7.2	2.96	RBSSL	R5 ESL-S	0.99	0.005	0.0025	0.00125
1,2-Dichloroethane	**107-06-2**	**0.00084**	21.2	**RBSSL**	R5 ESL-S	**2.8E-04**	**0.005**	**0.0025**	**0.00125**
1,2-Dichloropropane	**78-87-5**	**0.0026**	32.7	**RBSSL**	R5 ESL-S	**8.7E-04**	**0.005**	**0.0025**	**0.00125**
1,3-Dichlorobenzene	541-73-1	2.3	37.7	R-DCL	R5 ESL-S	0.77	0.005	0.0025	0.00125
1,4-Dichlorobenzene	106-46-7	0.0082	0.546	RBSSL	R5 ESL-S	0.0027	0.005	0.0025	0.00125
2-Butanone (MEK)	78-93-3	30	89.6	RBSSL	R5 ESL-S	10	0.010	0.005	0.0025
2-Hexanone	591-78-6	0.22	12.6	RBSSL	R5 ESL-S	0.073	0.010	0.005	0.0025

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4-Methyl-2-pentanone (MIBK)	108-10-1	9.0	443	RBSSL	R5 ESL-S	3.0	0.010	0.005	0.0025
Acetone	67-64-1	28	2.5	R-DCL	R5 ESL-S	0.83	0.010	0.005	0.0025
Benzene	**71-43-2**	**0.0042**	0.255	**RBSSL**	R5 ESL-S	**0.0014**	**0.005**	**0.0025**	**0.00125**
Bromochloromethane	74-97-5	NC	NC	None	None	NC	0.005	0.0025	0.00125
Bromodichloromethane	**75-27-4**	**0.00064**	0.54	**RBSSL**	R5 ESL-S	**2.1E-04**	**0.005**	**0.0025**	**0.00125**
Bromoform	75-25-2	0.046	15.9	RBSSL	R5 ESL-S	0.015	0.005	0.0025	0.00125
Bromomethane	74-83-9	0.044	0.235	RBSSL	R5 ESL-S	0.015	0.010	0.005	0.0025
Carbon disulfide	75-15-0	6.2	0.0941	RBSSL	R5 ESL-S	0.031	0.005	0.0025	0.00125
Carbon tetrachloride	**56-23-5**	**0.0034**	2.98	**RBSSL**	R5 ESL-S	**0.0011**	**0.005**	**0.0025**	**0.00125**
Chlorobenzene	108-90-7	1.2	13.1	RBSSL	R5 ESL-S	0.40	0.005	0.0025	0.00125
Chloroethane	75-00-3	0.65	0.65	R-DCL	R5 ESL-S	0.22	0.010	0.005	0.0025
Chloroform	**67-66-3**	**0.0011**	1.19	**RBSSL**	R5 ESL-S	**3.7E-04**	**0.005**	**0.0025**	**0.00125**
Chloromethane	74-87-3	0.98	10.4	RBSSL	R5 ESL-S	0.33	0.010	0.005	0.0025
cis-1,2-Dichloroethene	156-59-2	0.4	0.7837	R-DCL	R5 ESL-S	0.13	0.005	0.0025	0.00125
cis-1,3-Dichloropropene	**10061-01-5**	**0.0030**	0.3979	**RBSSL**	R5 ESL-S	**0.0010**	**0.005**	**0.0025**	**0.00125**
Cyclohexane	110-82-7	69	NC	R-DCL	None	23	0.005	0.0025	0.00125
Dibromochloromethane	**124-48-1**	**0.00078**	2.05	**RBSSL**	R5 ESL-S	**2.6E-04**	**0.005**	**0.0025**	**0.00125**
Dichlorodifluoromethane	75-71-8	12.2	39.5	RBSSL	R5 ESL-S	4.1	0.010	0.005	0.0025
Ethylbenzene	100-41-4	0.034	5.16	RBSSL	R5 ESL-S	0.011	0.005	0.0025	0.00125
Isopropylbenzene	98-82-8	11	NC	R-DCL	None	3.7	0.005	0.0025	0.00125
Methyl acetate	79-20-9	150	NC	RBSSL	None	50	0.010	0.005	0.0025
Methylcyclohexane	108-87-2	NC	NC	None	None	NC	0.005	0.0025	0.00125
Methylene chloride	75-09-2	0.023	4.05	R-DCL	R5 ESL-S	0.0077	0.010	0.005	0.0025

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Methyl-tert-butyl ether	1634-04-4	0.056	NC	RBSSL	None	0.019	0.005	0.0025	0.00125
Styrene	100-42-5	3.5	4.69	R-DCL	R5 ESL-S	1.2	0.005	0.0025	0.00125
Tetrachloroethene	**127-18-4**	**0.00098**	9.92	**RBSSL**	**R5 ESL-S**	**3.3E-04**	**0.005**	**0.0025**	**0.00125**
Toluene	108-88-3	12	5.45	R-DCL	R5 ESL-S	1.8	0.005	0.0025	0.00125
trans-1,2-Dichloroethene	156-60-5	0.62	0.784	RBSSL	R5 ESL-S	0.21	0.005	0.0025	0.00125
trans-1,3-Dichloropropene	**10061-02-6**	**0.0030**	0.3979	**RBSSL**	R5 ESL-S	**0.0010**	**0.005**	**0.0025**	**0.00125**
Trichloroethene	79-01-6	0.014	12.4	RBSSL	R5 ESL-S	0.0047	0.005	0.0025	0.00125
Trichlorofluoromethane	75-69-4	17	16.4	RBSSL	R5 ESL-S	5.5	0.010	0.005	0.0025
Vinyl chloride	**75-01-4**	**0.00011**	0.646	**RBSSL**	R5 ESL-S	**3.7E-05**	**0.010**	**0.005**	**0.0025**
Xylenes (total)	1330-20-7	4.0	10	RBSSL	R5 ESL-S	1.3	0.015	0.0075	0.00375

CAS – Chemical Abstracts Service
mg/kg – milligrams per kilogram
PQLG – Project Quantitation Limit Goal
NC – No Criteria

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, Dilution Attenuation Factor (DAF) = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Soil
Analytical Group: SVOCs (including Low Level PAHs)

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1'-Biphenyl	92-52-4	380	NC	RBSSL	None	130	0.333	0.167	0.083
1,2,4,5-Tetrachlorobenzene	95-94-3	1.0	2.02	RBSSL	R5 ESL-S	0.33	0.333	0.167	0.083
1,4-Dioxane	**123-91-1**	**0.0028**	**2.05**	**RBSSL**	**R5 ESL-S**	**0.00093**	**0.333**	**0.167**	**0.083**
2,2'-Oxybis(1-chloropropane)	**108-60-1**	**0.0024**	19.9	**RBSSL**	R5 ESL-S	**8.0E-04**	**0.333**	**0.167**	**0.083**
2,3,4,6-Tetrachlorophenol	**58-90-2**	130	**0.199**	RBSSL	**R5 ESL-S**	**0.066**	**0.333**	**0.167**	**0.083**
2,4,5-Trichlorophenol	95-95-4	250	14.1	R-DCL	R5 ESL-S	4.7	0.333	0.167	0.083
2,4,6-Trichlorophenol	**88-06-2**	**0.07**	9.94	**R-DCL**	R5 ESL-S	**0.023**	**0.333**	**0.167**	**0.083**
2,4-Dichlorophenol	120-83-2	1.1	87.5	R-DCL	R5 ESL-S	0.37	0.333	0.167	0.083
2,4-Dimethylphenol	**105-67-9**	9	**0.01**	R-DCL	**R5 ESL-S**	**0.0033**	**1.33**	**0.667**	**0.333**
2,4-Dinitrophenol	**51-28-5**	0.29	**0.0609**	R-DCL	**R5 ESL-S**	**0.020**	**0.333**	**0.167**	**0.083**
2,4-Dinitrotoluene	**121-14-2**	**0.0058**	1.28	**RBSSL**	R5 ESL-S	**0.0019**	**0.333**	**0.167**	**0.083**
2,6-Dinitrotoluene	**606-20-2**	1.0	**0.0328**	RBSSL	**R5 ESL-S**	**0.011**	**0.333**	**0.167**	**0.083**
2-Chloronaphthalene	**91-58-7**	42	**0.0122**	R-DCL	**R5 ESL-S**	**0.0041**	**0.333**	**0.167**	**0.083**
2-Chlorophenol	**95-57-8**	0.75	**0.243**	R-DCL	**R5 ESL-S**	**0.081**	**0.333**	**0.167**	**0.083**
2-Methylphenol (o-Cresol)	95-48-7	14	40.4	R-DCL	R5 ESL-S	4.7	0.333	0.167	0.083
2-Nitroaniline	**88-74-4**	**0.67**	74.1	**R-DCL**	R5 ESL-S	**0.22**	**1.33**	**0.667**	**0.333**
2-Nitrophenol	88-75-5	1.6	1.6	RBSSL	R5 ESL-S	0.53	1.33	0.667	0.333
3,3'-Dichlorobenzidine	**91-94-1**	**0.020**	0.646	**RBSSL**	R5 ESL-S	**0.0067**	**0.333**	**0.167**	**0.083**
3-Methylphenol (m-Cresol)	108-39-4	9.8	3.49	R-DCL	R5 ESL-S	1.2	0.333	0.167	0.083
3-Nitroaniline	99-09-2	NC	3.16	None	R5 ESL-S	1.05	1.33	0.667	0.333
4,6-Dinitro-2-methylphenol	**534-52-1**	**0.10**	0.144	**RBSSL**	R5 ESL-S	**0.033**	**3.3**	**1.67**	**0.83**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4-Bromophenyl phenyl ether	101-55-3	NC	NC	None	None	NC	0.333	0.167	0.083
4-Chloro-3-methyl phenol	59-50-7	86	7.95	RBSSL	R5 ESL-S	2.6	0.333	0.167	0.083
4-Chloroanaline	**106-47-8**	**0.0028**	1.1	**RBSSL**	R5 ESL-S	**9.3E-04**	**0.333**	**0.167**	**0.083**
4-Chlorophenyl phenyl ether	7005-72-3	NC	NC	None	None	NC	0.333	0.167	0.083
4-Methylphenol (p-Cresol)	106-44-5	1.1	163	R-DCL	R5 ESL-S	0.37	0.333	0.167	0.083
4-Nitroaniline	**100-01-6**	**0.028**	21.9	**RBSSL**	R5 ESL-S	**0.0093**	**1.33**	**0.667**	**0.333**
4-Nitrophenol	100-02-7	NC	5.12	None	R5 ESL-S	1.7	1.33	0.667	0.333
Acetophenone	98-86-2	22	300	RBSSL	R5 ESL-S	7.3	0.333	0.167	0.083
Atrazine	**1912-24-9**	**0.0038**	NC	**RBSSL**	None	**0.0013**	**0.333**	**0.167**	**0.083**
Benzaldehyde	100-52-7	16.2	NC	RBSSL	None	5.4	0.333	0.167	0.083
Bis(2-chloroethoxy)methane	**111-91-1**	0.50	**0.302**	RBSSL	**R5 ESL-S**	**0.10**	**0.333**	**0.167**	**0.083**
Bis(2-chloroethyl)ether	**111-44-4**	**0.000062**	23.7	**RBSSL**	R5 ESL-S	**2.1E-05**	**0.333**	**0.167**	**0.083**
Bis(2-ethylhexyl)phthalate	117-81-7	22	0.925	RBSSL	R5 ESL-S	0.31	0.333	0.167	0.083
Butyl benzyl phthalate	**85-68-7**	10	**0.239**	RBSSL	**R5 ESL-S**	**0.080**	**0.333**	**0.167**	**0.083**
Caprolactam	105-60-2	90	NC	RBSSL	None	30	0.333	0.167	0.083
Carbazole	86-74-8	5.9	NC	R-DCL	None	2.0	0.333	0.167	0.083
Dibenzofuran	132-64-9	4.9	NC	R-DCL	None	1.6	0.333	0.167	0.083
Diethyl phthalate	84-66-2	240	24.8	RBSSL	R5 ESL-S	8.3	0.333	0.167	0.083
Dimethyl phthalate	131-11-3	240	734	RBSSL	R5 ESL-S	80	0.333	0.167	0.083
Di-n-butyl phthalate	**84-74-2**	184	**0.15**	RBSSL	**R5 ESL-S**	**0.050**	**0.333**	**0.167**	**0.083**
Di-n-octyl phthalate	117-84-0	2,000	709	R-DCL	R5 ESL-S	240	0.333	0.167	0.083
Hexachlorobenzene	**118-74-1**	**0.011**	0.199	**RBSSL**	R5 ESL-S	**0.0037**	**0.333**	**0.167**	**0.083**
Hexachlorobutadiene	**87-68-3**	**0.034**	0.0398	**RBSSL**	R5 ESL-S	**0.011**	**0.333**	**0.167**	**0.083**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Hexachlorocyclopentadiene	77-47-4	14	0.755	RBSSL	R5 ESL-S	0.25	0.333	0.167	0.083
Hexachloroethane	**67-72-1**	**0.058**	**0.596**	**RBSSL**	**R5 ESL-S**	**0.019**	**0.333**	**0.167**	**0.083**
Isophorone	78-59-1	0.46	139	RBSSL	R5 ESL-S	0.15	0.333	0.167	0.083
Nitrobenzene	**98-95-3**	**0.0016**	**1.31**	**RBSSL**	**R5 ESL-S**	**5.3E-04**	**0.333**	**0.167**	**0.083**
N-Nitrosodi-n-propylamine	**621-64-7**	**0.00014**	**0.544**	**RBSSL**	**R5 ESL-S**	**4.7E-05**	**0.333**	**0.167**	**0.083**
N-Nitrosodiphenylamine	86-30-6	1.5	0.545	RBSSL	R5 ESL-S	0.18	0.333	0.167	0.083
Pentachlorophenol	**87-86-5**	**0.028**	**2.1**	**R-DCL**	**Eco-SSL**	**0.0093**	**1.33**	**0.667**	**0.333**
Phenol	108-95-2	56	120	R-DCL	R5 ESL-S	19	0.333	0.167	0.083
PAHs[2]									
2-Methylnaphthalene	91-57-6	3.1	29	R-DCL	Eco-SSL	1.0	0.010	0.005	0.0025
Acenaphthene	83-32-9	130	29	R-DCL	Eco-SSL	9.7	0.010	0.005	0.0025
Acenaphthylene	208-96-8	18	29	R-DCL	Eco-SSL	6.0	0.010	0.005	0.0025
Anthracene	120-12-7	1,700	29	R-RSL	Eco-SSL	9.7	0.010	0.005	0.0025
Benzo(a)anthracene	56-55-3	0.15	1.1	R-RSL	Eco-SSL	0.050	0.010	0.005	0.0025
Benzo(a)pyrene	50-32-8	0.015	1.1	R-RSL	Eco-SSL	0.0050	0.010	0.005	0.0025
Benzo(b)fluoranthene	205-99-2	0.15	1.1	R-RSL	Eco-SSL	0.050	0.010	0.005	0.0025
Benzo(g,h,i)perylene	191-24-2	170	1.1	R-RSL	Eco-SSL	0.37	0.010	0.005	0.0025
Benzo(k)fluoranthene	207-08-9	1.5	1.1	R-RSL	Eco-SSL	0.37	0.010	0.005	0.0025
Chrysene	218-01-9	15	1.1	R-RSL	Eco-SSL	0.37	0.010	0.005	0.0025
Dibenzo(a,h)anthracene	53-70-3	0.015	1.1	R-RSL	Eco-SSL	0.0050	0.010	0.005	0.0025
Fluoranthene	206-44-0	230	29	R-RSL	Eco-SSL	9.7	0.010	0.005	0.0025
Fluorene	86-73-7	170	29	R-DCL	Eco-SSL	9.7	0.010	0.005	0.0025
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	1.1	R-RSL	Eco-SSL	0.050	0.010	0.005	0.0025

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Naphthalene	**91-20-3**	**0.0094**	29	**RBSSL**	Eco-SSL	**0.0031**	**0.010**	**0.005**	**0.0025**
Phenanthrene	85-01-8	13	29	R-DCL	Eco-SSL	4.3	0.010	0.005	0.0025
Pyrene	129-00-0	170	1.1	R-RSL	Eco-SSL	0.37	0.010	0.005	0.0025

Notes:

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, DAF = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

[2] The low level PAHs will be analyzed by SW-846 Method 8270C Full Scan to obtain lower detection limits.

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Matrix: Soil
Analytical Group: Pesticides

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
alpha-BHC	319-84-6	0.0012	0.0994	RBSSL	R5 ESL-S	4.0E-04	0.0007	0.00035	0.00017
beta-BHC	319-85-7	0.044	0.00398	RBSSL	R5 ESL-S	0.0013	0.0007	0.00035	0.00017
delta-BHC	319-86-8	0.0012	9.94	RBSSL	R5 ESL-S	4.0E-04	0.0007	0.00035	0.00017
gamma-BHC (Lindane)	58-89-9	0.072	0.005	RBSSL	R5 ESL-S	0.0017	0.0007	0.00035	0.00017
Heptachlor	76-44-8	0.024	0.00598	RBSSL	R5 ESL-S	0.0020	0.0007	0.00035	0.00017
Aldrin	309-00-2	0.013	0.00332	RBSSL	R5 ESL-S	0.0011	0.0007	0.00035	0.00017
Heptachlor epoxide	1024-57-3	0.003	0.152	RBSSL	R5 ESL-S	0.0010	0.0007	0.00035	0.00017
Endosulfan I	959-98-8	37	0.119	R-RSL	R5 ESL-S	0.040	0.0007	0.00035	0.00017
Dieldrin	60-57-1	0.0034	0.0049	RBSSL	Eco-SSL	0.0011	0.0007	0.00035	0.00017
4,4'-DDE	72-55-9	0.94	0.021	RBSSL	Eco-SSL	0.0070	0.0007	0.00035	0.00017
Endrin	72-20-8	8.8	0.0101	RBSSL	R5 ESL-S	0.0034	0.0007	0.00035	0.00017
Endosulfan II	33213-65-9	37	0.119	R-RSL	R5 ESL-S	0.040	0.0007	0.00035	0.00017
4,4'-DDD	72-54-8	1.3	0.021	RBSSL	Eco-SSL	0.0070	0.0007	0.00035	0.00017
Endosulfan sulfate	1031-07-8	37	0.0358	R-RSL	R5 ESL-S	0.012	0.0007	0.00035	0.00017
4,4'-DDT	50-29-3	1.3	0.021	RBSSL	Eco-SSL	0.0070	0.0007	0.00035	0.00017
Methoxychlor	72-43-5	198	0.0199	RBSSL	R5 ESL-S	0.0066	0.0007	0.00035	0.00017
Endrin ketone	53494-70-5	1.8	0.0105	R-RSL	R5 ESL-S	0.0035	0.0007	0.00035	0.00017
Endrin aldehyde	7421-93-4	1.8	0.0105	R-RSL	R5 ESL-S	0.0035	0.0007	0.00035	0.00017
alpha-Chlordane	5103-71-9	0.26	0.224	RBSSL	R5 ESL-S	0.075	0.0007	0.00035	0.00017
gamma-Chlordane	5103-74-2	0.26	0.224	RBSSL	R5 ESL-S	0.075	0.0007	0.00035	0.00017
Toxaphene	8001-35-2	0.19	0.119	RBSSL	R5 ESL-S	0.040	0.033	0.022	0.011

Notes:

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, DAF = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Soil
Analytical Group: PCBs

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor-1016	**12674-11-2**	0.39	**0.000332**	R-RSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1221	**11104-28-2**	0.0024	**0.000332**	RBSSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1232	**11141-16-5**	0.0024	**0.000332**	RBSSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1242	**53469-21-9**	0.11	**0.000332**	RBSSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1248	**12672-29-6**	0.10	**0.000332**	RBSSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1254	**11097-69-1**	0.11	**0.000332**	R-RSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Aroclor-1260	**11096-82-5**	0.22	**0.000332**	R-RSL	**R5 ESL-S**	**1.1E-04**	**0.017**	**0.008**	**0.004**
Total PCBs	1336-36-3	1.8	NC	R-DCL	None	0.60	-	-	-

Notes:

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, DAF = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Soil
Analytical Group: Metals and Cyanide

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aluminum	7429-90-5	7,700	NC	R-RSL	None	2,600	10	5.0	2.5
Antimony	**7440-36-0**	3.1	**0.27**	R-RSL	**Eco-SSL**	**0.090**	**0.75**	**0.40**	**0.25**
Arsenic	**7440-38-2**	**0.026**	18	**RBSSL**	Eco-SSL	**0.0087**	**0.30**	**0.30**	**0.15**
Barium	7440-39-3	1,500	330	R-RSL	Eco-SSL	110	2.0	0.50	0.25
Beryllium	7440-41-7	16	21	R-RSL	Eco-SSL	5.3	0.25	0.10	0.05
Cadmium	7440-43-9	7.0	0.36	R-RSL	Eco-SSL	0.12	0.25	0.10	0.05
Calcium	7440-70-2	NC	NC	None	None	NC	250	100	50
Chromium	**7440-47-3**	**0.017**	26	**RBSSL**	Eco-SSL	**0.0057**	**0.25**	**0.20**	**0.10**
Cobalt	7440-48-4	2.3	13	R-RSL	Eco-SSL	0.77	0.63	0.50	0.25
Copper	7440-50-8	310	28	R-RSL	Eco-SSL	9.3	0.5	0.4	0.25
Iron	7439-89-6	5,500	NC	R-RSL	None	1800	5.0	3.0	1.5
Lead	7439-92-1	81	11	R-DCL	Eco-SSL	3.7	0.15	0.15	0.075
Magnesium	7439-95-4	NC	NC	None	None	NC	250	150	50
Manganese	7439-96-5	180	220	R-RSL	Eco-SSL	60	0.75	0.30	0.15
Mercury	7439-97-6	2.3	0.10	R-RSL	R5 ESL-S	0.033	0.03	0.026	0.013
Nickel	7440-02-0	150	38	R-RSL	Eco-SSL	13	0.5	0.3	0.25
Potassium	7440-09-7	NC	NC	None	None	NC	250	150	50
Selenium	7782-49-2	5.2	0.52	R-DCL	Eco-SSL	0.17	0.30	0.25	0.15
Silver	7440-22-4	31	4.2	R-DCL	Eco-SSL	1.4	0.25	0.10	0.05
Sodium	7440-23-5	NC	NC	None	None	NC	250	150	50
Thallium	**7440-28-0**	2.8	**0.0569**	R-DCL	**R5 ESL-S**	**0.019**	**0.40**	**0.20**	**0.15**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Vanadium	7440-62-2	39	7.8	R-RSL	Eco-SSL	2.6	0.63	0.50	0.25
Zinc	7440-66-6	2,300	46	R-RSL	Eco-SSL	15	1.0	0.5	0.25
Cyanide	57-12-5	0.94	1.3	R-DCL	R5 ESL-S	0.31	0.5	0.25	0.125

Notes:

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, DAF = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Soil
Analytical Group: Dioxins/Furans

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	CFA		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,2,3,4,6,7,8,9-OCDD	**3268-87-9**	1.5E-02	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**1.0E-05**	**6.67E-06**	**2.86E-07**
1,2,3,4,6,7,8,9-OCDF	**39001-02-0**	1.5E-02	**3.86E-05**	R-RSL	**R5 ESL-S**	**1.3E-05**	**1.0E-05**	**6.67E-06**	**3.14E-07**
1,2,3,4,6,7,8-HpCDD	**35822-46-9**	4.5E-04	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**5.0E-06**	**3.33E-06**	**1.18E-07**
1,2,3,4,6,7,8-HpCDF	67562-39-4	4.5E-04	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	1.45E-07
1,2,3,4,7,8,9-HpCDF	55673-89-7	4.5E-04	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	2.05E-07
1,2,3,4,7,8-HxCDD	**39227-28-6**	4.5E-05	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**5.0E-06**	**3.33E-06**	**1.93E-07**
1,2,3,4,7,8-HxCDF	70648-26-9	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	2.02E-07
1,2,3,6,7,8-HxCDD	**57653-85-7**	4.5E-05	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**5.0E-06**	**3.33E-06**	**1.75E-07**
1,2,3,6,7,8-HxCDF	57117-44-9	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	2.02E-07
1,2,3,7,8,9-HxCDD	**19408-74-3**	4.5E-05	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**5.0E-06**	**3.33E-06**	**1.41E-07**
1,2,3,7,8,9-HxCDF	72918-21-9	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	1.10E-07
1,2,3,7,8-PeCDD	**40321-76-4**	4.5E-06	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**5.0E-06**	**3.33E-06**	**1.03E-07**
1,2,3,7,8-PeCDF	57117-41-6	1.5E-04	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	2.00E-07
2,3,4,6,7,8-HxCDF	60851-34-5	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	5.0E-06	3.33E-06	1.23E-07
2,3,4,7,8-PeCDF	57117-31-4	1.5E-05	3.86E-05	R-RSL	R5 ESL-S	5.0E-06	5.0E-06	3.33E-06	2.61E-07
2,3,7,8-TCDD	**1746-01-6**	4.5E-06	**1.99E-07**	R-RSL	**R5 ESL-S**	**6.6E-08**	**1.0E-06**	**6.67E-07**	**5.2E-08**
2,3,7,8-TCDF	51207-31-9	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	1.0E-06	6.67E-07	1.04E-07
Total HpCDD	37871-00-4	NC	NC	None	None	NC	NA	NA	NA
Total HpCDF	38998-75-3	NC	NC	None	None	NC	NA	NA	NA
Total HxCDD	34465-46-8	NC	NC	None	None	NC	NA	NA	NA
Total HxCDF	55684-94-1	NC	NC	None	None	NC	NA	NA	NA

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	CFA		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Total PeCDD	36088-22-9	NC	NC	None	None	NC	NA	NA	NA
Total PeCDF	30402-15-4	NC	NC	None	None	NC	NA	NA	NA
Total TCDD	41903-57-5	NC	NC	None	None	NC	NA	NA	NA
Total TCDF	55722-27-5	NC	NC	None	None	NC	NA	NA	NA

Notes:

[1] The PSL references for surface and subsurface soil are: RBSSL - USEPA Regions 3, 6, and 9 Risk-Based Soil Screening Level, Migration to Groundwater, DAF = 20 (November, 2010); R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens and derived by multiplying criteria for 2,3,7,8-TCDD by World Health Organization Toxicity Equivalent Factor (May, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Sediment
Analytical Group: SVOCs (including Low Level PAHs)

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,1'-Biphenyl	92-52-4	390	NC	R-RSL	None	130	0.333	0.167	0.083
1,2,4,5-Tetrachlorobenzene	95-94-3	1.8	1.252	R-RSL	R5 SED	0.42	0.333	0.167	0.083
1,4-Dioxane	123-91-1	4.9	2.05	R-RSL	R5 ESL-S	0.68	0.333	0.167	0.083
2,2'-Oxybis(1-chloropropane)	**108-60-1**	**0.027**	19.9	**R-DCL**	R5 ESL-S	**0.0090**	**0.333**	**0.167**	**0.083**
2,3,4,6-Tetrachlorophenol	58-90-2	180	0.129	R-RSL	R5 SED	0.043	0.333	0.167	0.083
2,4,5-Trichlorophenol	95-95-4	250	14.1	R-DCL	R5 ESL-S	4.7	0.333	0.167	0.083
2,4,6-Trichlorophenol	**88-06-2**	**0.07**	0.208	**R-DCL**	R5 SED	**0.023**	**0.333**	**0.167**	**0.083**
2,4-Dichlorophenol	**120-83-2**	1.1	**0.0817**	R-DCL	**R5 SED**	**0.027**	**0.333**	**0.167**	**0.083**
2,4-Dimethylphenol	**105-67-9**	9.0	**0.304**	R-DCL	**R5 SED**	**0.10**	**1.33**	**0.667**	**0.333**
2,4-Dinitrophenol	**51-28-5**	0.29	**0.00621**	R-DCL	**R5 SED**	**0.0021**	**0.333**	**0.167**	**0.083**
2,4-Dinitrotoluene	**121-14-2**	1.6	**0.0144**	R-RSL	**R5 SED**	**0.0048**	**0.333**	**0.167**	**0.083**
2,6-Dinitrotoluene	**606-20-2**	6.1	**0.0398**	R-RSL	**R5 SED**	**0.013**	**0.333**	**0.167**	**0.083**
2-Chloronaphthalene	91-58-7	42	0.417	R-DCL	R5 SED	0.14	0.333	0.167	0.083
2-Chlorophenol	**95-57-8**	0.75	**0.0319**	R-DCL	**R5 SED**	**0.011**	**0.333**	**0.167**	**0.083**
2-Methylphenol (o-Cresol)	**95-48-7**	14	**0.0554**	R-DCL	**R5 SED**	**0.018**	**0.333**	**0.167**	**0.083**
2-Nitroaniline	**88-74-4**	**0.67**	74.1	**R-DCL**	R5 ESL	**0.22**	**1.33**	**0.667**	**0.333**
2-Nitrophenol	88-75-5	12	1.6	R-RSL	R5 ESL	0.53	1.33	0.667	0.333
3,3'-Dichlorobenzidine	**91-94-1**	**0.062**	0.127	**R-DCL**	R5 SED	**0.021**	**0.333**	**0.167**	**0.083**
3-Methylphenol (m-Cresol)	108-39-4	9.8	0.0524	R-DCL	R5 SED	0.017	0.333	0.167	0.083
3-Nitroaniline	99-09-2	NC	3.16	None	R5 ESL-S	1.0	1.33	0.667	0.333
4,6-Dinitro-2-methylphenol	**534-52-1**	0.49	**0.104**	R-RSL	**R5 SED**	**0.035**	**3.3**	**1.67**	**0.83**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
4-Bromophenyl phenyl ether	101-55-3	NC	1.55	None	R5 SED	0.52	0.333	0.167	0.083
4-Chloro-3-methyl phenol	59-50-7	610	0.388	R-RSL	R5 SED	0.13	0.333	0.167	0.083
4-Chloroanaline	**106-47-8**	0.97	**0.146**	R-DCL	**R5 SED**	**0.049**	**0.333**	**0.167**	**0.083**
4-Chlorophenyl phenyl ether	7005-72-3	NC	NC	None	None	NC	0.333	0.167	0.083
4-Methylphenol (p-Cresol)	**106-44-5**	1.1	**0.0202**	R-DCL	**R5 SED**	**0.0067**	**0.333**	**0.167**	**0.083**
4-Nitroaniline	100-01-6	24	21.9	R-RSL	R5 ESL-S	7.3	1.33	0.667	0.333
4-Nitrophenol	**100-02-7**	NC	**0.0133**	None	**R5 SED**	**0.0044**	**1.33**	**0.667**	**0.333**
Acetophenone	98-86-2	780	300	R-RSL	R5 ESL-S	100	0.333	0.167	0.083
Atrazine	**1912-24-9**	**0.048**	NC	**R-DCL**	None	**0.016**	**0.333**	**0.167**	**0.083**
Benzaldehyde	100-52-7	780	NC	R-RSL	None	260	0.333	0.167	0.083
Bis(2-chloroethoxy)methane	**111-91-1**	18	**0.302**	R-RSL	**R5 ESL-S**	**0.10**	**0.333**	**0.167**	**0.083**
Bis(2-chloroethyl)ether	**111-44-4**	**0.0007**	3.52	**R-DCL**	R5 SED	**2.3E-04**	**0.333**	**0.167**	**0.083**
Bis(2-ethylhexyl)phthalate	**117-81-7**	35	**0.182**	R-RSL	**R5 SED**	**0.061**	**0.333**	**0.167**	**0.083**
Butyl benzyl phthalate	85-68-7	260	1.97	R-RSL	R5 SED	0.66	0.333	0.167	0.083
Caprolactam	105-60-2	3,100	NC	R-RSL	None	1000	0.333	0.167	0.083
Carbazole	86-74-8	5.9	NC	R-DCL	None	2.0	0.333	0.167	0.083
Dibenzofuran	132-64-9	4.9	0.449	R-DCL	R5 SED	0.15	0.333	0.167	0.083
Diethyl phthalate	**84-66-2**	450	**0.295**	R-DCL	**R5 SED**	**0.098**	**0.333**	**0.167**	**0.083**
Dimethyl phthalate	131-11-3	1,100	734	R-DCL	R5 ESL-S	240	0.333	0.167	0.083
Di-n-butyl phthalate	84-74-2	610	1.114	R-RSL	R5 SED	0.37	0.333	0.167	0.083
Di-n-octyl phthalate	117-84-0	2,000	40.6	R-DCL	R5 SED	14	0.333	0.167	0.083
Hexachlorobenzene	**118-74-1**	0.30	**0.02**	R-RSL	**R5 SED**	**0.0067**	**0.333**	**0.167**	**0.083**
Hexachlorobutadiene	**87-68-3**	6.1	**0.0265**	R-RSL	**R5 SED**	**0.0088**	**0.333**	**0.167**	**0.083**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Hexachlorocyclopentadiene	77-47-4	37	0.901	R-RSL	R5 SED	0.30	0.333	0.167	0.083
Hexachloroethane	67-72-1	2.8	0.584	R-DCL	R5 SED	0.19	0.333	0.167	0.083
Isophorone	78-59-1	5.3	0.432	RBSSL	R5 SED	0.14	0.333	0.167	0.083
Nitrobenzene	**98-95-3**	**0.028**	0.145	**R-DCL**	R5 SED	**0.0093**	**0.333**	**0.167**	**0.083**
N-Nitrosodi-n-propylamine	**621-64-7**	**0.0006**	0.544	**R-DCL**	R5 ESL-S	**2.0E-04**	**0.333**	**0.167**	**0.083**
N-Nitrosodiphenylamine	86-30-6	9.7	0.545	R-DCL	R5 ESL-S	0.18	0.333	0.167	0.083
Pentachlorophenol	**87-86-5**	**0.028**	23	**R-DCL**	R5 SED	**0.0093**	**1.33**	**0.667**	**0.333**
Phenol	108-95-2	56	120	R-DCL	R5 ESL-S	19	0.333	0.167	0.083
PAHs[2]									
2-Methylnaphthalene	91-57-6	3.1	0.0202	R-DCL	R5 SED	0.0067	0.010	0.005	0.0025
Acenaphthene	**83-32-9**	130	**0.00671**	R-DCL	**R5 SED**	**0.0022**	**0.010**	**0.005**	**0.0025**
Acenaphthylene	**208-96-8**	18	**0.00587**	R-DCL	**R5 SED**	**0.0020**	**0.010**	**0.005**	**0.0025**
Anthracene	120-12-7	1,700	0.0572	R-RSL	R5 SED	0.019	0.010	0.005	0.0025
Benzo(a)anthracene	56-55-3	0.15	0.108	R-RSL	R5 SED	0.036	0.010	0.005	0.0025
Benzo(a)pyrene	50-32-8	0.015	0.15	R-RSL	R5 SED	0.0050	0.010	0.005	0.0025
Benzo(b)fluoranthene	205-99-2	0.15	10.4	R-RSL	R5 SED	0.050	0.010	0.005	0.0025
Benzo(g,h,i)perylene	191-24-2	170	0.17	R-RSL	R5 SED	0.057	0.010	0.005	0.0025
Benzo(k)fluoranthene	207-08-9	1.5	0.24	R-RSL	R5 SED	0.080	0.010	0.005	0.0025
Chrysene	218-01-9	15	0.166	R-RSL	R5 SED	0.055	0.010	0.005	0.0025
Dibenzo(a,h)anthracene	53-70-3	0.015	0.033	R-RSL	R5 SED	0.0050	0.010	0.005	0.0025
Fluoranthene	206-44-0	230	0.423	R-RSL	R5 SED	0.14	0.010	0.005	0.0025
Fluorene	86-73-7	170	0.0774	R-DCL	R5 SED	0.026	0.010	0.005	0.0025
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	0.2	R-RSL	R5 SED	0.050	0.010	0.005	0.0025

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Naphthalene	91-20-3	0.7	0.176	R-RSL	R5 SED	0.059	0.010	0.005	0.0025
Phenanthrene	85-01-8	13	0.204	R-DCL	R5 SED	0.068	0.010	0.005	0.0025
Pyrene	129-00-0	170	0.195	R-RSL	R5 SED	0.065	0.010	0.005	0.0025

Notes:

[1] The PSL references for sediment are: R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

[2] The low level PAHs will be analyzed by SW-846 Method 8270C Full Scan to obtain lower detection limits.

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Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Sediment
Analytical Group: Pesticides

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
alpha-BHC	319-84-6	0.0072	0.006	R-DCL	R5 SED	0.0020	0.0007	0.00035	0.00017
beta-BHC	319-85-7	0.026	0.005	R-DCL	R5 SED	0.0017	0.0007	0.00035	0.00017
delta-BHC	319-86-8	0.0072	71.5	R-DCL	R5 SED	0.0024	0.0007	0.00035	0.00017
gamma-BHC (Lindane)	58-89-9	0.0094	0.00237	R-DCL	R5 SED	7.9E-04	0.0007	0.00035	0.00017
Heptachlor	**76-44-8**	0.11	**0.0006**	R-RSL	**R5 SED**	**2.0E-04**	**0.0007**	**0.00035**	**0.00017**
Aldrin	309-00-2	0.029	0.002	R-RSL	R5 SED	6.7E-04	0.0007	0.00035	0.00017
Heptachlor epoxide	1024-57-3	0.053	0.00247	R-RSL	R5 SED	8.2E-04	0.0007	0.00035	0.00017
Endosulfan I	959-98-8	20	0.00326	R-DCL	R5 SED	0.0011	0.0007	0.00035	0.00017
Dieldrin	60-57-1	0.0072	0.0019	R-DCL	R5 SED	6.3E-04	0.0007	0.00035	0.00017
4,4'-DDE	72-55-9	1.4	0.00316	R-RSL	R5 SED	0.0010	0.0007	0.00035	0.00017
Endrin	72-20-8	0.99	0.00222	R-DCL	R5 SED	7.4E-04	0.0007	0.00035	0.00017
Endosulfan II	33213-65-9	20	0.00194	R-DCL	R5 SED	6.5E-04	0.0007	0.00035	0.00017
4,4'-DDD	72-54-8	2.0	0.00488	R-RSL	R5 SED	0.0016	0.0007	0.00035	0.00017
Endosulfan sulfate	1031-07-8	20	0.0346	R-DCL	R5 SED	0.012	0.0007	0.00035	0.00017
4,4'-DDT	50-29-3	1.7	0.00416	R-RSL	R5 SED	0.0014	0.0007	0.00035	0.00017
Methoxychlor	72-43-5	31	0.0136	R-RSL	R5 SED	0.0045	0.0007	0.00035	0.00017
Endrin ketone	53494-70-5	0.99	0.48	R-DCL	R5 SED	0.16	0.0007	0.00035	0.00017
Endrin aldehyde	7421-93-4	0.99	0.48	R-DCL	R5 SED	0.16	0.0007	0.00035	0.00017
alpha-Chlordane	5103-71-9	1.6	0.0045	R-RSL	R5 SED	0.0015	0.0007	0.00035	0.00017
gamma-Chlordane	5103-74-2	1.6	0.0045	R-RSL	R5 SED	0.0015	0.0007	0.00035	0.00017
Toxaphene	**8001-35-2**	0.44	**7.70E-06**	R-RSL	**R5 SED**	**2.6E-06**	**0.033**	**0.022**	**0.011**

Notes:

[1] The PSL references for sediment are: R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Sediment
Analytical Group: PCBs

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor-1016	12674-11-2	0.39	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1221	11104-28-2	0.14	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1232	11141-16-5	0.14	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1242	53469-21-9	0.22	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1248	12672-29-6	0.22	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1254	11097-69-1	0.11	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Aroclor-1260	11096-82-5	0.22	0.0598	R-RSL	R5 SED	0.020	0.017	0.008	0.004
Total PCBs	1336-36-3	NC	NC	None	None	NC	-	-	-

Notes:

[1] The PSL references for sediment are: R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Sediment
Analytical Group: Metals and Cyanide

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aluminum	7429-90-5	7,700	NC	R-RSL	None	2,600	10	5.0	2.5
Antimony	**7440-36-0**	3.1	**0.27**	R-RSL	**Eco-SSL**	**0.090**	**0.75**	**0.40**	**0.25**
Arsenic	7440-38-2	0.39	9.79	R-RSL	R5 SED	0.13	0.30	0.30	0.15
Barium	7440-39-3	1,500	330	R-RSL	Eco-SSL	110	2.0	0.50	0.25
Beryllium	7440-41-7	16	21	R-RSL	Eco-SSL	5.3	0.25	0.10	0.05
Cadmium	7440-43-9	7.0	0.99	R-RSL	R5 SED	0.33	0.25	0.10	0.05
Calcium	7440-70-2	NC	NC	None	None	NC	250	100	50
Chromium	7440-47-3	0.29	43.4	R-RSL	R5 SED	0.097	0.25	0.20	0.10
Cobalt	7440-48-4	2.3	50	R-RSL	R5 SED	0.77	0.63	0.50	0.25
Copper	7440-50-8	310	31.6	R-RSL	R5 SED	10	0.5	0.4	0.25
Iron	7439-89-6	5,500	35.8	R-RSL	R5 SED	12	5.0	3.0	1.5
Lead	7439-92-1	81	11	R-DCL	Eco-SSL	3.7	0.15	0.15	0.075
Magnesium	7439-95-4	NC	NC	None	None	NC	250	150	50
Manganese	7439-96-5	180	220	R-RSL	Eco-SSL	60	0.75	0.30	0.15
Mercury	7439-97-6	2.3	0.10	R-RSL	R5 ESL-S	0.033	0.03	0.026	0.013
Nickel	7440-02-0	150	22.7	R-RSL	R5 SED	7.6	0.5	0.3	0.25
Potassium	7440-09-7	NC	NC	None	None	NC	250	150	50
Selenium	7782-49-2	5.2	0.52	R-DCL	Eco-SSL	0.17	0.30	0.25	0.15
Silver	7440-22-4	31	0.5	R-DCL	R5 SED	0.17	0.25	0.10	0.05
Sodium	7440-23-5	NC	NC	None	None	NC	250	150	50
Thallium	**7440-28-0**	2.8	**0.0569**	R-DCL	**R5 ESL-S**	**0.019**	**0.40**	**0.20**	**0.15**

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Vanadium	7440-62-2	39	7.8	R-RSL	Eco-SSL	2.6	0.63	0.50	0.25
Zinc	7440-66-6	2,300	121	R-RSL	R5 SED	40	1.0	0.5	0.25
Cyanide	**57-12-5**	**0.94**	**0.0001**	**R-DCL**	**R5 SED**	**3.3E-05**	**0.5**	**0.25**	**0.125**

Notes:

[1] The PSL references for sediment are: R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Sediment
Analytical Group: Dioxins/Furans

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	CFA		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
1,2,3,4,6,7,8,9-OCDD	**3268-87-9**	1.5E-02	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**1.0E-05**	**6.67E-06**	**2.86E-07**
1,2,3,4,6,7,8,9-OCDF	**39001-02-0**	1.5E-02	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**1.0E-05**	**6.67E-06**	**3.14E-07**
1,2,3,4,6,7,8-HpCDD	**35822-46-9**	4.5E-04	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**5.0E-06**	**3.33E-06**	**1.18E-07**
1,2,3,4,6,7,8-HpCDF	**67562-39-4**	4.5E-04	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**1.45E-07**
1,2,3,4,7,8,9-HpCDF	**55673-89-7**	4.5E-04	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**2.05E-07**
1,2,3,4,7,8-HxCDD	**39227-28-6**	4.5E-05	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**5.0E-06**	**3.33E-06**	**1.93E-07**
1,2,3,4,7,8-HxCDF	**70648-26-9**	4.5E-05	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**2.02E-07**
1,2,3,6,7,8-HxCDD	**57653-85-7**	4.5E-05	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**5.0E-06**	**3.33E-06**	**1.75E-07**
1,2,3,6,7,8-HxCDF	**57117-44-9**	4.5E-05	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**2.02E-07**
1,2,3,7,8,9-HxCDD	**19408-74-3**	4.5E-05	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**5.0E-06**	**3.33E-06**	**1.41E-07**
1,2,3,7,8,9-HxCDF	**72918-21-9**	4.5E-05	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**1.10E-07**
1,2,3,7,8-PeCDD	**40321-76-4**	4.5E-06	**3.3E-06**	R-RSL	**R5 SED**	**1.1E-06**	**5.0E-06**	**3.33E-06**	**1.03E-07**
1,2,3,7,8-PeCDF	**57117-41-6**	1.5E-04	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**2.00E-07**
2,3,4,6,7,8-HxCDF	**60851-34-5**	4.5E-05	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**1.23E-07**
2,3,4,7,8-PeCDF	**57117-31-4**	1.5E-05	**1.3E-08**	R-RSL	**R5 SED**	**4.3E-09**	**5.0E-06**	**3.33E-06**	**2.61E-07**
2,3,7,8-TCDD	**1746-01-6**	4.5E-06	**1.2E-07**	R-RSL	**R5 SED**	**4.0E-08**	**1.0E-06**	**6.67E-07**	**5.2E-08**
2,3,7,8-TCDF	51207-31-9	4.5E-05	3.86E-05	R-RSL	R5 ESL-S	1.3E-05	1.0E-06	6.67E-07	1.04E-07
Total HpCDD	37871-00-4	NC	NC	None	None	NC	NA	NA	NA
Total HpCDF	38998-75-3	NC	NC	None	None	NC	NA	NA	NA
Total HxCDD	34465-46-8	NC	NC	None	None	NC	NA	NA	NA
Total HxCDF	55684-94-1	NC	NC	None	None	NC	NA	NA	NA

Analyte	CAS Number	PSL (mg/kg)		PSL Reference[1]		PQLG (mg/kg)	CFA		
		HHRA	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Total PeCDD	36088-22-9	NC	NC	None	None	NC	NA	NA	NA
Total PeCDF	30402-15-4	NC	NC	None	None	NC	NA	NA	NA
Total TCDD	41903-57-5	NC	NC	None	None	NC	NA	NA	NA
Total TCDF	55722-27-5	NC	NC	None	None	NC	NA	NA	NA

Notes:

[1] The PSL references for sediment are: R-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 SED – USEPA Region 5 Ecological Screening Level, Sediment (August, 2003); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2003). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Groundwater
Analytical Group: VOCs

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,1,1-Trichloroethane	71-55-6	200	G-DCL	67	1.0	0.5	0.25
1,1,2,2-Tetrachloroethane	**79-34-5**	**0.067**	**T-RSL**	**0.022**	**1.0**	**0.5**	**0.25**
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	140	VAPOR	47	1.0	0.5	0.25
1,1,2-Trichloroethane	**79-00-5**	**0.24**	**T-RSL**	**0.080**	**1.0**	**0.5**	**0.25**
1,1-Dichloroethane	75-34-3	2.4	T-RSL	0.80	1.0	0.5	0.25
1,1-Dichloroethene	75-35-4	7.0	G-DCL	2.3	1.0	0.5	0.25
1,2,3- Trichlorobenzene	87-61-6	2.9	T-RSL	0.97	1.0	0.5	0.25
1,2,4-Trichlorobenzene	**120-82-1**	**0.41**	**T-RSL**	**0.14**	**1.0**	**0.5**	**0.25**
1,2-Dibromo-3-chloropropane (DBCP)	**96-12-8**	**0.00032**	**T-RSL**	**1.1E-04**	**2.0**	**1.0**	**0.5**
1,2-Dibromoethane (EDB)	**106-93-4**	**0.0065**	**T-RSL**	**0.0022**	**1.0**	**0.5**	**0.25**
1,2-Dichlorobenzene	95-50-1	37	T-RSL	12	1.0	0.5	0.25
1,2-Dichloroethane	**107-06-2**	**0.15**	**T-RSL**	**0.050**	**1.0**	**0.5**	**0.25**
1,2-Dichloropropane	**78-87-5**	**0.39**	**T-RSL**	**0.13**	**1.0**	**0.5**	**0.25**
1,3-Dichlorobenzene	541-73-1	80	G-DCL	27	1.0	0.5	0.25
1,4-Dichlorobenzene	**106-46-7**	**0.43**	**T-RSL**	**0.14**	**1.0**	**0.5**	**0.25**
2-Butanone (MEK)	78-93-3	710	T-RSL	240	10	5.0	2.5
2-Hexanone	**591-78-6**	**4.7**	**T-RSL**	**1.6**	**5.0**	**2.5**	**1.5**
4-Methyl-2-pentanone (MIBK)	108-10-1	200	T-RSL	67	10	5.0	2.5
Acetone	67-64-1	2,200	T-RSL	730	10	5.0	2.5
Benzene	**71-43-2**	**0.41**	**T-RSL**	**0.14**	**1.0**	**0.5**	**0.25**
Bromochloromethane	74-97-5	NC	None	NC	1.0	0.5	0.25

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Bromodichloromethane	**75-27-4**	**0.12**	**T-RSL**	**0.040**	**1.0**	**0.5**	**0.25**
Bromoform	75-25-2	8.5	T-RSL	2.8	2.0	1.0	0.5
Bromomethane	**74-83-9**	**0.87**	**T-RSL**	**0.29**	**1.0**	**0.5**	**0.25**
Carbon disulfide	75-15-0	100	T-RSL	33	1.0	0.5	0.25
Carbon tetrachloride	**56-23-5**	**0.37**	**VAPOR**	**0.12**	**1.0**	**0.5**	**0.25**
Chlorobenzene	108-90-7	9.1	T-RSL	3.0	1.0	0.5	0.25
Chloroethane	75-00-3	62	G-DCL	21	1.0	0.5	0.25
Chloroform	**67-66-3**	**0.19**	**T-RSL**	**0.063**	**1.0**	**0.5**	**0.25**
Chloromethane	74-87-3	19	T-RSL	6.3	1.0	0.5	0.25
cis-1,2-Dichloroethene	156-59-2	7.3	T-RSL	2.4	1.0	0.5	0.25
cis-1,3-Dichloropropene	10061-01-5	4.0	T-RSL	1.3	1.0	0.5	0.25
Cyclohexane	110-82-7	100	VAPOR	33	1.0	0.5	0.25
Dibromochloromethane	**124-48-1**	**0.15**	**T-RSL**	**0.050**	**1.0**	**0.5**	**0.25**
Dichlorodifluoromethane	75-71-8	1.5	VAPOR	0.50	1.0	0.5	0.25
Ethylbenzene	100-41-4	1.5	T-RSL	0.50	1.0	0.5	0.25
Isopropylbenzene	98-82-8	68	T-RSL	23	1.0	0.5	0.25
Methyl acetate	79-20-9	3,700	T-RSL	1,200	2.0	1.0	0.5
Methylcyclohexane	108-87-2	NC	None	NC	1.0	0.5	0.25
Methylene chloride	75-09-2	4.8	T-RSL	1.6	4.0	2.0	1.0
Methyl-tert-butyl ether	1634-04-4	12	T-RSL	4.0	1.0	0.5	0.25
Styrene	100-42-5	100	G-DCL	33	1.0	0.5	0.25
Tetrachloroethene	**127-18-4**	**0.11**	**T-RSL**	**0.037**	**1.0**	**0.5**	**0.25**
Toluene	108-88-3	230	T-RSL	77	1.0	0.5	0.25

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
trans-1,2-Dichloroethene	156-60-5	11	T-RSL	3.7	1.0	0.5	0.25
trans-1,3-Dichloropropene	10061-02-6	4.0	T-RSL	1.3	1.0	0.5	0.25
Trichloroethene	79-01-6	2.0	T-RSL	0.67	1.0	0.5	0.25
Trichlorofluoromethane	75-69-4	18	VAPOR	6.0	1.0	0.5	0.25
Vinyl chloride	**75-01-4**	**0.016**	**T-RSL**	**0.0053**	**1.0**	**0.5**	**0.25**
Xylenes (total)	1330-20-7	20	T-RSL	6.7	4.0	2.0	1.0

µg/L – micrograms per liter

Notes:

[1] The PSL references for groundwater are: T-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Level (December, 2009); VAPOR – USEPA vapor screening values calculated from 2002 Vapor guidance and 2010 toxicity factors (November, 2002; May, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009). Refer to Appendix E for further explanation and justification of PSLs.

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Matrix: Groundwater
Analytical Group: SVOCs (including Low Level PAHs)

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,1'-Biphenyl	92-52-4	180	T-RSL	60	5.0	2.5	1.25
1,2,4,5-Tetrachlorobenzene	95-94-3	1.1	T-RSL	0.37	5.0	2.5	1.25
1,4-Dioxane	**123-91-1**	**0.67**	**T-RSL**	**0.22**	**5.0**	**2.5**	**1.25**
2,2'-Oxybis(1-chloropropane)	**108-60-1**	**0.32**	**T-RSL**	**0.11**	**5.0**	**2.5**	**1.25**
2,3,4,6-Tetrachlorophenol	58-90-2	110	T-RSL	37	5.0	2.5	1.25
2,4,5-Trichlorophenol	95-95-4	370	T-RSL	120	5.0	2.5	1.25
2,4,6-Trichlorophenol	**88-06-2**	**3.6**	**G-DCL**	**1.2**	**5.0**	**2.5**	**1.25**
2,4-Dichlorophenol	120-83-2	11	T-RSL	3.7	5.0	2.5	1.25
2,4-Dimethylphenol	105-67-9	73	T-RSL	24	20	10	5.0
2,4-Dinitrophenol	**51-28-5**	**7.3**	**T-RSL**	**2.4**	**50**	**25**	**10**
2,4-Dinitrotoluene	**121-14-2**	**0.22**	**T-RSL**	**0.073**	**5.0**	**2.5**	**1.25**
2,6-Dinitrotoluene	**606-20-2**	**3.7**	**T-RSL**	**1.2**	**5.0**	**2.5**	**1.25**
2-Chloronaphthalene	91-58-7	290	T-RSL	97	5.0	2.5	1.25
2-Chlorophenol	95-57-8	18	T-RSL	6.0	5.0	2.5	1.25
2-Methylphenol (o-Cresol)	95-48-7	180	T-RSL	60	5.0	2.5	1.25
2-Nitroaniline	88-74-4	37	T-RSL	12	20	10	5.0
2-Nitrophenol	88-75-5	7.3	T-RSL	2.4	5.0	2.5	1.25
3,3'-Dichlorobenzidine	**91-94-1**	**0.15**	**T-RSL**	**0.050**	**5.0**	**2.5**	**1.25**
3-Methylphenol (m-Cresol)	108-39-4	180	T-RSL	60	5.0	2.5	1.25
3-Nitroaniline	99-09-2	NC	None	NC	20	10	5.0
4,6-Dinitro-2-methylphenol	**534-52-1**	**0.29**	**T-RSL**	**0.097**	**20**	**10**	**5.0**

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
4-Bromophenyl phenyl ether	101-55-3	NC	None	NC	5.0	2.5	1.25
4-Chloro-3-methyl phenol	59-50-7	370	T-RSL	120	5.0	2.5	1.25
4-Chloroanaline	**106-47-8**	**0.34**	**T-RSL**	**0.11**	**5.0**	**2.5**	**1.25**
4-Chlorophenyl phenyl ether	7005-72-3	NC	None	NC	5.0	2.5	1.25
4-Methylphenol (p-Cresol)	106-44-5	18	T-RSL	6.0	5.0	2.5	1.25
4-Nitroaniline	**100-01-6**	**3.4**	**T-RSL**	**1.1**	**20**	**10**	**5.0**
4-Nitrophenol	100-02-7	NC	None	NC	20	10	5.0
Acetophenone	98-86-2	370	T-RSL	120	5.0	2.5	1.25
Atrazine	**1912-24-9**	**0.29**	**T-RSL**	**0.097**	**5.0**	**2.5**	**1.25**
Benzaldehyde	100-52-7	370	T-RSL	120	5.0	2.5	1.25
Bis(2-chloroethoxy)methane	111-91-1	11	T-RSL	3.7	5.0	2.5	1.25
Bis(2-chloroethyl)ether	**111-44-4**	**0.012**	**T-RSL**	**0.0040**	**5.0**	**2.5**	**1.25**
Bis(2-ethylhexyl)phthalate	**117-81-7**	**4.8**	**T-RSL**	**1.6**	**5.0**	**2.5**	**1.25**
Butyl benzyl phthalate	85-68-7	35	T-RSL	12	5.0	2.5	1.25
Caprolactam	105-60-2	1,800	T-RSL	600	5.0	2.5	1.25
Carbazole	86-74-8	43	G-DCL	14	5.0	2.5	1.25
Dibenzofuran	**132-64-9**	**3.7**	**T-RSL**	**1.2**	**5.0**	**2.5**	**1.25**
Diethyl phthalate	84-66-2	2,900	T-RSL	970	5.0	2.5	1.25
Dimethyl phthalate	131-11-3	2,900	T-RSL	970	5.0	2.5	1.25
Di-n-butyl phthalate	84-74-2	370	T-RSL	120	5.0	2.5	1.25
Di-n-octyl phthalate	117-84-0	20	G-DCL	6.7	5.0	2.5	1.25
Hexachlorobenzene	**118-74-1**	**0.042**	**T-RSL**	**0.014**	**5.0**	**2.5**	**1.25**
Hexachlorobutadiene	**87-68-3**	**0.86**	**T-RSL**	**0.29**	**5.0**	**2.5**	**1.25**

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Hexachlorocyclopentadiene	**77-47-4**	**0.019**	**VAPOR**	**0.0063**	**5.0**	**2.5**	**1.25**
Hexachloroethane	**67-72-1**	**3.7**	**T-RSL**	**1.2**	**5.0**	**2.5**	**1.25**
Isophorone	78-59-1	71	T-RSL	24	5.0	2.5	1.25
Nitrobenzene	**98-95-3**	**0.12**	**T-RSL**	**0.040**	**5.0**	**2.5**	**1.25**
N-Nitrosodi-n-propylamine	**621-64-7**	**0.0096**	**T-RSL**	**0.0032**	**5.0**	**2.5**	**1.25**
N-Nitrosodiphenylamine	86-30-6	14	T-RSL	4.7	5.0	2.5	1.25
Pentachlorophenol	**87-86-5**	**0.17**	**T-RSL**	**0.057**	**20**	**10**	**5.0**
Phenol	108-95-2	1,100	T-RSL	370	5.0	2.5	1.25
PAHs[2]							
2-Methylnaphthalene	91-57-6	15	T-RSL	5.0	0.2	0.1	0.05
Acenaphthene	83-32-9	220	T-RSL	73	0.2	0.1	0.05
Acenaphthylene	208-96-8	71	G-DCL	24	0.2	0.1	0.05
Anthracene	120-12-7	1,100	T-RSL	370	0.2	0.1	0.05
Benzo(a)anthracene	**56-55-3**	**0.029**	**T-RSL**	**0.0097**	**0.2**	**0.1**	**0.05**
Benzo(a)pyrene	**50-32-8**	**0.0029**	**T-RSL**	**9.7E-04**	**0.2**	**0.1**	**0.05**
Benzo(b)fluoranthene	**205-99-2**	**0.029**	**T-RSL**	**0.0097**	**0.2**	**0.1**	**0.05**
Benzo(g,h,i)perylene	191-24-2	110	T-RSL	37	0.2	0.1	0.05
Benzo(k)fluoranthene	207-08-9	0.29	T-RSL	0.097	0.2	0.1	0.05
Chrysene	218-01-9	2.9	T-RSL	0.97	0.2	0.1	0.05
Dibenzo(a,h)anthracene	**53-70-3**	**0.0029**	**T-RSL**	**9.7E-04**	**0.2**	**0.1**	**0.05**
Fluoranthene	206-44-0	150	T-RSL	50	0.2	0.1	0.05
Fluorene	86-73-7	150	T-RSL	50	0.2	0.1	0.05
Indeno(1,2,3-c,d)pyrene	**193-39-5**	**0.029**	**T-RSL**	**0.0097**	**0.2**	**0.1**	**0.05**

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Naphthalene	**91-20-3**	**0.14**	**T-RSL**	**0.047**	**0.2**	**0.1**	**0.05**
Phenanthrene	85-01-8	23	G-DCL	7.7	0.2	0.1	0.05
Pyrene	129-00-0	110	T-RSL	37	0.2	0.1	0.05

Notes:

[1] The PSL references for groundwater are: T-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Level (December, 2009); VAPOR – USEPA vapor screening values calculated from 2002 Vapor guidance and 2010 toxicity factors (November, 2002; May, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009). Refer to Appendix E for further explanation and justification of PSLs.

[2] The low level PAHs will be analyzed by SW-846 Method 8270C Full Scan to obtain lower detection limits.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaped rows indicated the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Groundwater
Analytical Group: Pesticides

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
alpha-BHC	**319-84-6**	**0.011**	**T-RSL**	**0.0037**	**0.02**	**0.01**	**0.005**
beta-BHC	319-85-7	0.037	T-RSL	0.012	0.02	0.01	0.005
delta-BHC	**319-86-8**	**0.011**	**T-RSL**	**0.0037**	**0.02**	**0.01**	**0.005**
gamma-BHC (Lindane)	58-89-9	0.061	T-RSL	0.020	0.02	0.01	0.005
Heptachlor	**76-44-8**	**0.015**	**T-RSL**	**0.0050**	**0.02**	**0.01**	**0.005**
Aldrin	**309-00-2**	**0.0040**	**T-RSL**	**0.0013**	**0.02**	**0.01**	**0.005**
Heptachlor epoxide	**1024-57-3**	**0.0074**	**T-RSL**	**0.0025**	**0.02**	**0.01**	**0.005**
Endosulfan I	959-98-8	22	T-RSL	7.3	0.02	0.01	0.005
Dieldrin	**60-57-1**	**0.0042**	**T-RSL**	**0.0014**	**0.02**	**0.01**	**0.005**
4,4'-DDE	72-55-9	0.20	T-RSL	0.067	0.02	0.01	0.005
Endrin	72-20-8	1.1	T-RSL	0.37	0.02	0.01	0.005
Endosulfan II	33213-65-9	22	T-RSL	7.3	0.02	0.01	0.005
4,4'-DDD	72-54-8	0.28	T-RSL	0.093	0.02	0.01	0.005
Endosulfan sulfate	1031-07-8	22	T-RSL	7.3	0.02	0.01	0.005
4,4'-DDT	50-29-3	0.20	T-RSL	0.067	0.02	0.01	0.005
Methoxychlor	72-43-5	18	T-RSL	6.0	0.02	0.01	0.005
Endrin ketone	53494-70-5	1.1	T-RSL	0.37	0.02	0.01	0.005
Endrin aldehyde	7421-93-4	1.1	T-RSL	0.37	0.02	0.01	0.005
alpha-Chlordane	5103-71-9	0.19	T-RSL	0.063	0.02	0.01	0.005
gamma-Chlordane	5103-74-2	0.19	T-RSL	0.063	0.02	0.01	0.005
Toxaphene	**8001-35-2**	**0.061**	**T-RSL**	**0.020**	**1.0**	**0.667**	**0.333**

Notes:

[1] The PSL references for groundwater are: T-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Level (December, 2009); VAPOR – USEPA vapor screening values calculated from 2002 Vapor guidance and 2010 toxicity factors (November, 2002; May, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009). Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Groundwater
Analytical Group: PCBs

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aroclor-1016	12674-11-2	0.96	T-RSL	0.32	0.5	0.25	0.125
Aroclor-1221	**11104-28-2**	**0.0068**	**T-RSL**	**0.0023**	**0.5**	**0.25**	**0.125**
Aroclor-1232	**11141-16-5**	**0.0068**	**T-RSL**	**0.0023**	**0.5**	**0.25**	**0.125**
Aroclor-1242	**53469-21-9**	**0.034**	**T-RSL**	**0.011**	**0.5**	**0.25**	**0.125**
Aroclor-1248	**12672-29-6**	**0.034**	**T-RSL**	**0.011**	**0.5**	**0.25**	**0.125**
Aroclor-1254	**11097-69-1**	**0.034**	**T-RSL**	**0.011**	**0.5**	**0.25**	**0.125**
Aroclor-1260	**11096-82-5**	**0.034**	**T-RSL**	**0.011**	**0.5**	**0.25**	**0.125**
Total PCBs	-	NC	None	NC	0.5	0.25	0.125

Notes:

[1] The PSL references for groundwater are: T-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Level (December, 2009); VAPOR – USEPA vapor screening values calculated from 2002 Vapor guidance and 2010 toxicity factors (November, 2002; May, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009). Refer to Appendix E for further explanation and justification of PSLs.

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Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

Matrix: Groundwater
Analytical Group: Metals (Total and Dissolved) and Cyanide

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Aluminum	7429-90-5	3,700	T-RSL	1200	200	100	50
Antimony	**7440-36-0**	**1.5**	**T-RSL**	**0.50**	**3.0**	**2.0**	**1.0**
Arsenic	**7440-38-2**	**0.045**	**T-RSL**	**0.015**	**1.5**	**1.5**	**0.75**
Barium	7440-39-3	730	T-RSL	240	40	20	10
Beryllium	**7440-41-7**	**4.0**	**G-DCL**	**1.3**	**5.0**	**2**	**1.0**
Cadmium	7440-43-9	1.8	T-RSL	0.60	1.5	0.50	0.25
Calcium	7440-70-2	NC	None	NC	5000	2000	1000
Chromium	**7440-47-3**	**0.043**	**T-RSL**	**0.014**	**10**	**4.0**	**2.0**
Cobalt	**7440-48-4**	**1.1**	**T-RSL**	**0.37**	**3.1**	**2.5**	**1.3**
Copper	7440-50-8	150	T-RSL	50	10	8.0	4.0
Iron	7439-89-6	2,600	T-RSL	870	100	60	30
Lead	7439-92-1	15	T-RSL	5.0	3.0	3.0	1.5
Magnesium	7439-95-4	NC	None	NC	5000	3000	1000
Manganese	7439-96-5	88	T-RSL	29	15	10	5.0
Mercury	7439-97-6	1.1	T-RSL	0.37	0.2	0.16	0.08
Nickel	7440-02-0	73	T-RSL	24	10	6.0	3.0
Potassium	7440-09-7	NC	None	NC	5000	3000	1000
Selenium	7782-49-2	18	T-RSL	6.0	6.0	5.0	3.0
Silver	7440-22-4	18	T-RSL	6.0	10	2.0	1.0
Sodium	7440-23-5	NC	None	NC	5000	3000	1000
Thallium	7440-28-0	2.0	G-DCL	0.67	2.0	1.0	0.75

Analyte	CAS Number	PSL (µg/L)	PSL Reference[1]	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Vanadium	7440-62-2	18	T-RSL	6.0	13	10	5.0
Zinc	7440-66-6	1,100	T-RSL	370	20	10	5.0
Cyanide	57-12-5	73	T-RSL	24	0.02	0.01	0.005

Notes:

[1] The PSL references for groundwater are: T-RSL - USEPA Regions 3, 6, and 9 Regional Screening Level for Soil, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Level (December, 2009); VAPOR – USEPA vapor screening values calculated from 2002 Vapor guidance and 2010 toxicity factors (November, 2002; May, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009). Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicate that the PSL is between the laboratory LOQ and LOD. The Project Team has agreed to accept this data for decision making if results below the LOQ are "J" qualified and the results are discussed in the uncertainties section of the Risk Assessment.

Bolded and Shaded rows indicate the PSL is less than the LOD; therefore, the Project Team has agreed to report non-detected results at the LOD and any limitations on data use that result from having detection limits that are greater than PSLs will be described in the RFI Report.

SAP Worksheet No. 16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Initial screening soil, sediment and groundwater sampling	Tetra Tech	04/01/2011	04/15/2011	RFI Report (if no additional sampling)	12/26/2011 (draft) 10/05/2012 (final)
Additional “step-out” and other sampling if necessary	Tetra Tech	07/01/2011	07/15/2011	RFI Report (if additional sampling is performed)	03/26/2012 (draft) 01/05/2013 (final)

SAP Worksheet No. 17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The RFI field data collection program will be within the boundaries of SWMU 11 including the drainage channel to Broom Branch and area downgradient of the Building 2981 concrete tank. Initial sampling will be performed to determine if there are target analytes in environmental media as a result of operations, and the fire and firefighting measures including the drainage channel that discharges to Broom Branch. If the maximum concentration of any target analyte exceeds an applicable human health or ecological PSL, additional sampling may be required to define the nature and extent of COPC contamination and to support human health and ecological risk assessments. The planned initial screening sample locations are presented on Figures 17-1, 17-2, 17-3, and 17-4.

Chemicals that may have been present in former Building 225 during the 1976 fire and have the potential to impact environmental media include the following:

- Solvents, paints, and other chemical compounds: Various chemicals were stored onsite, and may have leaked onto the ground and leached into the subsurface during operations, and the fire and subsequent firefighting efforts. Analyses will include the Target Compound List (TCL) for VOCs and SVOCs (including low level PAHs).

- Pesticides and PCBs: Although there is an inventory list of what was being stored in Building 225 at the time of the fire, its accuracy and reliability is unknown. Pesticides and PCBs have been detected at other NSA Crane sites and may have been present at this site. Therefore, analyses will include the 21 TCL pesticides and 7 TCL PCBs.

- Dioxins/Furans: Dioxins and furans occur as byproducts from the incineration of chlorinated substances as well as a naturally occurring byproduct of combustion. Analyses will include a standard screen of dioxin and furan congeners. Surface soil and sediment samples from the investigative areas (except the concrete tank), in the vicinity of the former building, and at the downwind areas will be analyzed for dioxins/furans.

- Metals: Various metals may have been present on site in the form of paints and other chemical compounds. Analyses will include the full list of 23 Target Analyte List (TAL) Metals and cyanide

- Cyanide. Cyanide is on the full TAL and may have been present at the site

Soil Sampling

The initial screening soil sampling program consists of collecting surface soil and subsurface soil from biased locations across the site. Generally, surface and subsurface soil samples will be horizontally

aligned. Subsurface soil samples will be collected from the sampling interval with the greatest potential for contamination based on field screening techniques. This approach, coupled with the ability to make field decisions to determine the extent of contamination, supports both the delineation of contamination and the risk assessment objectives.

Initial screening surface and subsurface samples will be collected from locations that have a greater potential to have subsurface contamination (based on site history). The proposed initial screening soil sampling locations are shown on Figures 17-1, 17-2, and 17-3. If site conditions require a location to be moved (i.e., boulder, tree, etc.), the field sampler will move to an alternate location to collect the necessary sample. If this occurs, the field sampler will document the reason in the field logbook. Generally, surface and subsurface soil samples will be analyzed for SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide. In addition, surface soil samples collected under the floor slab and subsurface soil samples will also be analyzed for VOCs. Also, select soil samples collected from the following areas will be analyzed for dioxins/furans: below the floor slab, in the ditch, the three adjacent areas, and the four downwind areas.

Soil sampling will be performed in the investigative areas (IA) as described below and as shown on Figures 17-1, 17-2, and 17-3.

- **IA-1 – Building 225 Sub-slab.** Contaminants may have infiltrated through joints in the concrete floor slab. Four surface soil samples and four subsurface soil samples will be collected from below the floor slab and aggregate material. The four locations will be selected in the field along joints in the floor slab and will be horizontally aligned. One of the four surface soil samples will be analyzed for dioxins/furans. Based on the CSM, eight soil samples will be collected from IA-1. Four surface soil samples from discrete locations will be analyzed for VOCs, SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide; one surface soil sample collected from the sub-slab will be analyzed for dioxins/furans. Four subsurface soil samples from discrete locations will be analyzed for VOCs, SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide.

- **IA-2 – Ditch.** The ditch along the west side of the site conveyed runoff from the site and contaminants may have infiltrated into the soil in the area. Surface soil samples will be collected from the ditch. The first sampling location will be where the ditch drains into the culvert at the north end of the site. There will be five additional sampling locations south of that point approximately every 50' along the ditch. Because it is an open area, surface soil samples from this area will not be analyzed also be analyzed for VOCs. One soil sample collected in the ditch will be analyzed for dioxins/furans. Based on the CSM, six surface soil samples will be collected from IA-2. Six samples from discrete locations will be analyzed for SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide; one sample will be analyzed for dioxins/furans.

- **IA-3 – Adjacent Areas.** The adjacent areas consist of three distinct areas near Building 225. The areas are the grassed area east and adjacent to the Building 225 floor slab; the adjacent area north of the floor slab containing both paved and grassed areas; and the adjacent area south of the floor slab containing both paved and grassed areas. These areas may have been contaminated during the fire. Surface samples will be collected from four locations from the grassed area east and adjacent to the Building 225 floor slab and from two locations in the each of the two adjacent areas south and north of the Building 225 floor slab. Because it is an open area, the surface soil samples from these areas will not be analyzed for VOCs. In each of the three adjacent areas, one sample will be analyzed for dioxins/furans.. Based on the CSM, eight surface soil samples will be collected from the three adjacent areas that comprise IA-3. Eight surface soil samples from discrete locations at three of the adjacent areas (immediately north, south, and east of the building) will be analyzed for SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide; three soil samples collected at the adjacent areas (one sample representing each of these three areas) will be analyzed for dioxins/furans.

- **IA-4 – Downwind Areas.** The downwind areas consist of four distinct areas and consist of 1) two grassed areas northeast of Building 225 and across Highway 101; and 2) two unpaved areas southwest of Building 225. Based on the CSM, the two areas northeast of Building 225 are believed to be upwind of the fire. The areas southwest of Building 225 are believed to be downwind of the fire. These areas may have been contaminated during the fire by airborne particulates containing residual dioxins/furans. Two discrete surface soil samples will be collected from the upwind northeast areas, while four discrete surface soil samples will be collected from the downwind southwest areas. Samples will be collected from open, grassy areas to be determined in the field, identified using GPS, and recorded in the field log book. Based on the CSM, six surface soil samples will be collected from the downwind areas that comprise IA-4; the six samples will be analyzed for dioxins/furans.

- **IA-5 – UST Area.** Historical drawings indicate that a UST was located approximately 40 feet south of Building 225; the disposition of this UST is not known. Subsurface soil samples from this area will be analyzed for fuel contaminants. Subsurface soil samples will be collected from near the approximate bottom elevation of the UST as estimated by field observations and field measurements. Based on the CSM, four subsurface soil samples will be collected from IA-5. These soil samples will be analyzed for VOCs and SVOCs (including low level PAHs).

- **IA-6 - Building 2981 Concrete Tank.** Green water used to fight the fire was contaminated with the chemicals released during the fire. Some of the contaminated water was transported to the Building 2981 concrete tank for holding. The disposition of this water is unknown and may have been released in to the nearby drainage. Therefore, four surface soil samples will be collected downstream from the concrete tank outlet; the locations of these samples will be determined in the field based on the location of the tank and the hydrology of the surrounding area. Because it is an open area, samples from this area will not be analyzed for VOCs. Four surface soil samples will be collected

from IA-6. Two samples from discrete locations will be analyzed for SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide.

Sample Depth – A DPT rig will be used to collect surface and subsurface soil samples. Samples for all applicable analytical groups will be collected from the 0- to 2-foot interval for surface soil. Subsurface soil samples will be collected from IA-1 – Building 225 Sub-slab and IA-5 – UST Area. The samples will be collected from the specific 2-foot boring that has the greatest potential for contamination based on visual and field instrument screening as determined in the field. At IA-1, the soil borings will be advanced to refusal or 15 feet, whichever is shallower. At IA-5, the subsurface soil samples may be collected to 10 feet bgs or to the estimated bottom of fill or UST elevation, whichever is shallower. If there are no obvious signs of contamination, the subsurface soil samples will be collected from the 4- to 6-foot interval.

Soil Sample Quantities – As described above, 28 surface soil samples (plus 4 duplicate samples for QC purposes) and 8 subsurface soil samples (plus 1 duplicate samples for QC purposes) will be collected and analyzed for certain target analytes, which require analysis to low levels to support future risk assessments, if necessary. Due to the volatile nature of VOCs, surface soil samples will not be analyzed for VOCs except those from below the floor slab. The planned soil sample locations are presented on Figures 17-1, 17-2, and 17-3. Additionally, at the discretion of the Tetra Tech FOL, the sampler may collect up to 12 “step-out” samples when areas of obvious or likely contamination are encountered during the initial screening sampling event. The flexibility to collect these additional samples extends both horizontally and vertically. Areas requiring additional sampling will be discerned by visual signs and the experience of the Tetra Tech FOL. Emphasis will be placed on collecting samples required to delineate contaminated areas.

One round of “step-out” samples will be collected at the boundaries of the area(s) to be sampled if PSL exceedances are detected in initial screening surface and subsurface soil samples to delineate areas of COPCs. Up to 30 additional “step-out” samples will be collected from 25 feet away in each general direction (north, east, south, or west) where PSL exceedances from the initial screening are unbounded, and “step-out” samples can be collected. For instance, if there are buildings, roads, or other impediments that preclude extending the boundary by 25 feet in a general direction, no sample will be collected and the impediment will be documented in the field sampler’s notes. These “step-out” samples will be collected from the same depth as the corresponding initial screening sample and analyzed for only those target analytes that exceeded PSLs (and exceeded background levels for metals). If the Project Team deems them necessary, additional “step-out” samples will be collected from a depth of two feet below the initial screening sample that exceeds one or more PSLs to vertically delineate COPCs. Samples are expected to be collected by a DPT; however, a hand auger or backhoe may be used, if necessary.

Based on a review of the analytical data and risk screening calculations from the initial screening sampling event a HHRA and an ERA will be conducted in accordance with Sections 11.4 and 11.5.

Sediment Sampling

The initial screening sediment sampling program consists of collecting surface sediment samples from the drainage channel which conveyed runoff from the fire to Broom Branch. Surface sediment samples will be collected from two depth intervals at horizontally aligned locations. This approach, coupled with the ability to make field decisions to delineate the extent of contamination as described as follows, supports both the delineation of contamination and the calculation of risk estimates.

The proposed sediment sample location grid was adjusted to follow the drainage path, and further adjustments may be necessary in the field if sediment is not available at the proposed locations. An attempt will be made to minimize relocation of sampling points because it affects the representativeness of individual samples; however, collecting the total number of proposed sediment samples is deemed more important to achieving project goals than strict adherence to the sampling grid. The planned sediment sample locations are presented on Figure 17-4.

- **IA-7 – Drainage Channel.** Surface sediment samples will be collected from four locations in the drainage channel at two depth intervals. The first sampling location will be where the culvert from the site discharges to the channel. Three additional sampling locations will be selected west (downgradient) of that point approximately every 100 feet along the channel. The first set of surface sediment samples will be collected from 0 to 6 inches bgs. The second set of surface sediment samples will be collected from the same locations between 6 to 12 inches bgs. Sediment samples will be analyzed for SVOCs (including low level PAHs), pesticides, PCBs, metals, and cyanide. Sediment samples will also be analyzed for TOC to support site-specific risk calculations. Because it is an open area, surface sediment samples from this area will not be analyzed for VOCs. One sediment sample from each depth set of the four surface sediment samples identified above will be analyzed for dioxins/furans. Based on the CSM, eight sediment samples will be collected from IA-7. Four discrete shallow surface samples and four discrete deeper surface soil samples will be analyzed for SVOCs (including low level PAHs), PCBs, pesticides, metals, cyanide, and TOC; two samples from one of the sediment locations (one sample representing each depth set) will be analyzed for dioxins/furans.

Sediment Sample Quantities

Eight sediment samples (plus one duplicate sample for QC purposes) will be collected from the drainage channel to Broom Branch during the initial screening sampling event. Based on the initial screening sample results, the Project Team will determine if one round of “step-out” sample collection is required. Up to six additional “step-out” sediment samples (three locations at two depths) will be collected 50 feet or 100 feet from where PSL exceedances from the initial screening are unbounded, and “step-out” samples can be collected. Based on initial screening sediment sample results, additional soil samples

from the floodplain may also be collected. Sections 11.4 and 11.5 will be used to guide the Project Team in this decision.

Groundwater Sampling

Groundwater samples will be collected from three locations during the initial screening sampling event using the DPT rig. Two samples will be collected from locations located downgradient of potentially contaminated areas of the site, and one sample will be collected from a location upgradient of the site as shown on Figure 17-4. The downgradient locations will be downgradient of areas with the greatest potential for contamination based on historical data and field screening observations. The need for installation of permanent monitoring wells will be determined by Project Team consensus based on the results from the initial screening sampling event groundwater samples.

Three groundwater samples (plus one duplicate sample for QC purposes) will be collected and analyzed for VOCs, SVOCs (including low level PAHs), pesticides, PCBs, and total (and dissolved, if groundwater is highly turbid) metals and cyanide. Groundwater samples will also be analyzed for field parameters, including water levels, pH, specific conductivity, turbidity, temperature, Oxidation-Reduction Potential (ORP), and dissolved oxygen (DO) to support field sampling decisions and site-specific risk calculations.

Based on the results from the initial screening sampling event, permanent groundwater monitoring wells may be deemed necessary by the Project Team as a selected remedy from a CMS and would be placed in areas where subsurface soil contamination concentrations and groundwater sample analysis data indicate the greatest potential for groundwater contamination. If permanent groundwater monitoring wells are required, monitoring well locations will be identified in a separate SAP.

Additional Sampling for Delineation of COPCs and Risk Assessments

One additional round of “step out” sampling will be performed if initial screening sampling (as described above) results indicate that contaminant levels exceed PSLs and COPCs are identified. Additional samples may include surface soil, subsurface soil, surface water, sediment, and/or groundwater.

Field Quality Control Samples

Field QC samples will be collected as part of the investigation, including FDs, trip blanks, equipment rinsate blanks, and field blanks. Worksheet No. 20 presents the field QC sample summary. Also, additional sample volume will be collected as necessary for the laboratory QC of Matrix Spike (MS)/ Matrix Spike Duplicate (MSD) analyses (for VOCs, SVOCs, including low level PAHs, pesticides, and PCBs) and MS/laboratory duplicate analyses (for metals and cyanide).

SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SB01	11SS010002 and 11SSFDMMDDYYNN[2]	Soil	0 - 2	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1 + 1 FD	
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
	11SB01NNNN[3] and 11SBFDMMDDYYNN[2]	Soil	>2[4]	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1 + 1 FD	
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
11SB02	11SS020002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				Dioxins/Furans	1	
	11SB02NNNN[3]	Soil	>2[4]	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB03	11SS030002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
	11SB03NNNN[3]	Soil	>2[4]	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SB04	11SS040002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
	11SB04NNNN[3]	Soil	>2[4]	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB05	11SS050002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB06	11SS060002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB07	11SS070002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB08	11SS080002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB09	11SS090002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				Dioxins/Furans	1	
11SB10	11SS100002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB11	11SS110002 and 11SSFDMMDDYYNN[2]	Soil	0 – 2	SVOCs/Low Level PAHs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
				Dioxins/Furans	1 + 1 FD	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SB12	11SS120002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB13	11SS130002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB14	11SS140002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				Dioxins/Furans	1	
11SB15	11SS150002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB16	11SS160002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB17	11SS170002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB18	11SS180002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				Dioxins/Furans	1	
11SB19	11SS190002	Soil	0 – 2	Dioxins/Furans	1	SOP-07, SOP-08, SOP-11
11SB20	11SS200002	Soil	0 – 2	Dioxins/Furans	1	SOP-07, SOP-08, SOP-11
11SB21	11SS210002 and 11SSFDMMDDYYNN[2]	Soil	0 – 2	Dioxins/Furans	1 + 1 FD	SOP-07, SOP-08, SOP-11
11SB22	11SS220002	Soil	0 – 2	Dioxins/Furans	1	SOP-07, SOP-08, SOP-11
11SB23	11SS230002	Soil	0 – 2	Dioxins/Furans	1	SOP-07, SOP-08, SOP-11
11SB24	11SS240002	Soil	0 – 2	Dioxins/Furans	1	SOP-07, SOP-08, SOP-11

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SB25	11SB25NNNN[3]	Soil	>2[4]	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
11SB26	11SB26NNNN[3]	Soil	>2[4]	VOCs	1	SOP-07, SOP-08, SOP-11
				SVOCs/Low Level PAHs	1	
11SB27	11SB27NNNN[3]	Soil	>2[4]	VOCs	1	
				SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
11SB28	11SB28NNNN[3]	Soil	>2[4]	VOCs	1	
				SVOCs/Low Level PAHs	1	
11SB29	11SS290002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB30	11SS300002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB31	11SS310002 and 11SSFDMMDDYYNN[2]	Soil	0 – 2	SVOCs/Low Level PAHs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
11SB32	11SS3			SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB33 to 11SBXX[5]	11SS(25-XX)0002	Soil	0 – 2	SVOCs/Low Level PAHs	1	SOP-07, SOP-08, SOP-11
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
	11SB(25-XX)NNNN[3]	Soil	>2[4]	VOCs	1	
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11SB(XX+1) to 11SBYY[6]	TBD	Soil	TBD	COPCs (TBD)	1 (each location)	SOP-07, SOP-08, SOP-11

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SB(YY+1) to 11SBZZ[7]	TBD	Soil	TBD	COPCs (TBD)	1 (each location)	SOP-07, SOP-08, SOP-11
11SD01	11SD010006	Sediment	0-0.5	SVOCs/Low Level PAHs	1	SOP-09
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	
	11SD010612	Sediment	0.5-1.0	SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	
11SD02	11SD020006 and 11SDF	Sediment	0-0.5	SVOCs/Low Level PAHs	1 + 1 FD	SOP-09
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
				Dioxins/Furans	1 + 1 FD	
				TOC	1 + 1 FD	
	11SD020612	Sediment	0.5-1.0	SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				Dioxins/Furans	1	
				TOC	1	
11SD03	11SD030006	Sediment	0-0.5	SVOCs/Low Level PAHs	1	SOP-09
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	
	11SD030612	Sediment	0.5-1.0	SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
11SD04	11SD040006	Sediment	0-0.5	SVOCs/Low Level PAHs	1	SOP-09
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	
	11SD040612	Sediment	0.5-1.0	SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
				TOC	1	
11TW01	11GW0101 and 11GWFDMMDDYYNN[2]	Groundwater	Shallow (<20)	VOCs	1 + 1 FD	SOP-11, SOP-14, SOP-15, SOP-16, SOP-17
				SVOCs/Low Level PAHs	1 + 1 FD	
				Pesticides/PCBs	1 + 1 FD	
				Metals/Cyanide	1 + 1 FD	
11TW02	11GW0201	Groundwater	Shallow (<20)	VOCs	1	SOP-11, SOP-14, SOP-15, SOP-16, SOP-17
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11TW03	11GW0301	Groundwater	Shallow (<20)	VOCs	1	SOP-11, SOP-14, SOP-15, SOP-16, SOP-17
				SVOCs/Low Level PAHs	1	
				Pesticides/PCBs	1	
				Metals/Cyanide	1	
11MWT01 to MWTXX[8]	11MWT0101 to 11MWTXX01	Groundwater	TBD	COPCs (TBD)	1 (each location)	SOP-11, SOP-12, SOP-13, SOP-14, SOP-15, SOP-16, SOP-17

1 SOP or worksheet that describes the sample collection procedures (Worksheet No. 21).
2 FD locations may change in the field based on visual and olfactory observations and PID readings; "MMDDYY" represents date collected; NN represents the sequential number of this type of QC sample collected on that date
3 Depth of samples will be determined in the field; "NNNN" represents top and bottom sample depth. For example, if sample is collected from 4- to 6-feet bgs, the depth will be recorded as 0406.
4 If there are no PID readings or visual observations that cause a subsurface depth to be selected in a biased manner, then the 4- to 6-foot bgs depth interval will be selected.
5 Up to 12 additional "step-out" samples may be collected at the discretion of the Tetra Tech FOL during the initial screening sampling event.
6 Up to 30 additional "step-out" samples may be collected to delineate COPCs.
7 Additional random background samples may be collected to support risk assessments. The sample quantities and sample locations will be determined using VSP.
8 The number of permanent monitoring wells and their locations will be determined by the Project Team based on the initial screening sample event data.

SAP Worksheet No. 19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

MATRIX	ANALYTICAL GROUP	ANALYTICAL AND PREPARATION METHOD/ SOP REFERENCE (1)	CONTAINERS (number, size, and type)	SAMPLE VOLUME (units)	PRESERVATION REQUIREMENTS (chemical, temperature, light protected)	MAXIMUM HOLDING TIME (preparation/ analysis)
Groundwater and aqueous QC samples	VOCs	SW-846 5030/8260B, Empirical SOP-202	Three 40-milliliter (mL) glass vials	5 mL	Hydrochloric acid (HCl) to pH<2; cool to ≤6 °C; no headspace	14 days to analysis
Soil and sediment	VOCs	SW-846 5035/8260B, Empirical SOP-202/225	Three 5-gram (g) Encore samplers or terracores	5 g	Sodium bisulfate in water, Cool to ≤6 °C; methanol, freeze to < -10 °C	48 hours from sampling to preparation, 14 days to analysis
Groundwater and aqueous QC samples	SVOCs (Including Low Level PAHs)	SW-846 3510C/3520/ 8270C/8270C-Low, Empirical SOP-201/300	Two 1-liter (L) glass amber bottles	1,000 mL	Cool to ≤6 °C	7 days until extraction, 40 days to analysis
Soil and sediment	SVOCs (Including Low Level PAHs)	SW-846 3546/8270C/ 8270C-Low, Empirical SOP-201/343	One 4-ounce (oz) glass jar	30 g	Cool to ≤6 °C	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	Pesticides/ PCBs	SW-846 3510C/3520/ 8081B/8082A, Empirical SOP-211/302	Two 1-L glass amber bottles	1,000 mL	Cool to ≤6 °C	7 days until extraction, 40 days to analysis
Soil and sediment	Pesticides/ PCBs	SW-846 3546/ 3550/8081B/8082A, Empirical SOP-211/343	One 4-oz glass jar	30 g	Cool to ≤6 °C	14 days until extraction, 40 days to analysis
Groundwater and aqueous QC samples	Metals, Including Mercury (and Dissolved Metals)	SW-846 3010A/ 6010C/7470A, Empirical SOP-100/103/105	One 500-mL plastic bottle	50 mL / 30 mL for mercury	Nitric acid to pH <2; Cool to ≤6 °C	180 days to analysis except mercury, 28 days for mercury
Soil and sediment	Metals, Including Mercury	SW-846 3050B/ 6010C/7471A, Empirical SOP-100/104/105	One 4-oz glass jar	1 to 2 grams / 0.3 g for mercury	Cool to ≤6 °C	180 days to analysis except mercury, 28 days for mercury

MATRIX	ANALYTICAL GROUP	ANALYTICAL AND PREPARATION METHOD/ SOP REFERENCE (1)	CONTAINERS (number, size, and type)	SAMPLE VOLUME (units)	PRESERVATION REQUIREMENTS (chemical, temperature, light protected)	MAXIMUM HOLDING TIME (preparation/ analysis)
Groundwater and aqueous QC samples	Cyanide	SW-846 9012A, Empirical SOP-164/175	One 250-mL plastic bottle	50 mL	Sodium hydroxide (NaOH) to a pH > 12; Cool to ≤6 °C	14 days to analysis
Soil and sediment	Cyanide	SW-846 9012A, Empirical SOP-164	One 4-oz glass jar	5 g	Cool to ≤6 °C	14 days to analysis
Aqueous QC samples	Dioxins/ Furans	SW-846 8290A, CFA CF-OA-E-001, CF-OA-E-002	Two 1-L glass amber bottles	1,000 mL	Cool to ≤6 °C	30 days for extraction, 45 days for analysis
Soil and sediment	Dioxins/ Furans	SW-846 8290A, CFA CF-OA-E-001, CF-OA-E-002	One 8-oz glass jar with Teflon®-lined lid	30 g	Cool to ≤6 °C	30 days for extraction, 45 days for analysis
Sediment	TOC	Lloyd Kahn, Empirical SOP-221	One 4-oz glass jar	30 g	Cool to ≤6 °C	14 days to analysis

Notes:

1 Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet No.23).

SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs[1]	No. of Equip. Blanks	No. of Trip Blanks	Total No. of Samples to Lab
Soil	VOCs	12	2	2/2	1	1	16
	SVOCs/Low Level PAHs	30	4	3/3	2	NA	36
	PCBs	26	4	3/3	2	NA	32
	Pesticides	26	4	3/3	2	NA	32
	Metals/Cyanide	26	4	3/3	2	NA	32
	Dioxins/Furans	11	2	1/1	1	NA	14
Sediment	SVOCs/Low Level PAHs	8	1	1/1	0	NA	9
	Pesticides	8	1	1/1	0	NA	9
	PCBs	8	1	1/1	0	NA	9
	Metals/Cyanide	8	1	1/1	0	NA	9
	TOC	8	1	1/1	0	NA	9
	Dioxins/Furans	2	1	1/1	0	NA	3
Groundwater	VOCs	3	1	1/1	1	1	6
	SVOCs/Low Level PAHs	3	1	1/1	1	NA	5
	PCBs	3	1	1/1	1	NA	5
	Pesticides	3	1	1/1	1	NA	5
	Total Metals/Cyanide	3	1	1/1	1	NA	5
	Dissolved Metals	0 Minimum, 3 Maximum	1	1/1	1[2]	NA	5

[1] Although the MS/MSD is not typically considered a field QC, it is included here because location determination is often established in the field. The MS/MSD are not included in the Total No. of Samples sent to the Lab. For Total and Dissolved Metals, an MD will be collected in place of an MSD.

[2] The equipment blank for the Dissolved Metals, if collected, will be obtained by passing rinse water through a 0.45-micron filter.

The quantities identified above are for the initial screening sampling event. Additional samples may be required, but cannot be quantified at this time.

SAP Worksheet No. 21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP-01	Global Positioning System, 12/10 Rev. 0	Tetra Tech	GPS unit	Y (project-specific)	Contained in Appendix A
SOP-02	Sample Labeling, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-03	Sample Identification Nomenclature, 02/11 , Rev. 1	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-04	Sample Custody and Documentation of Field Activities, ,02/11 Rev. 1	Tetra Tech	Field logbook, sample log sheets, boring logs	Y (project-specific)	Contained in Appendix A
SOP-05	Sample Preservation, Packaging, and Shipping, , Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-06	Decontamination of Field Sampling Equipment, 12/10, Rev. 0	Tetra Tech	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate free detergent, deionized water	Y (project-specific)	Contained in Appendix A
SOP-07	Soil Coring and Sampling Using Hand Auger Techniques, 02/11, Rev. 1	Tetra Tech	Stainless steel auger bucket, extension rods, and T-handle	Y (project-specific)	Contained in Appendix A
SOP-08	Soil Sample Logging, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-09	Sediment Sampling, 02/11, Rev. 1	Tetra Tech	Stainless steel or disposable trowels	Y (project-specific)	Contained in Appendix A
SOP-10	Management of Investigation-Derived Waste, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-11	Subsurface Soil and Groundwater Sampling Using Direct-Push Technology, 12/10, Rev. 0	Tetra Tech	DPT Rig	Y (project-specific)	Contained in Appendix A
SOP-12	Monitoring Well Installation, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	
SOP-13	Monitoring Well Development, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-14	Measurement of Water Levels, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-15	Low-Flow Well Purging and Stabilization, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-16	Monitoring Well Sampling, 12/10, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-17	Calibration and Care of Water Quality Meters, 12/10, Rev. 0	Tetra Tech	Multi-parameter water quality meter, such as a Horiba U-22	Y (project-specific)	Contained in Appendix A

SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

FIELD EQUIPMENT	ACTIVITY[1]	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (CA)	RESPONSIBLE PERSON	SOP REFERENCE[2]	COMMENTS
Water Quality Meter (YSI 600 Series or Equivalent)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or replacement	Tetra Tech FOL or designee	GH-2.8, SA-1.1, SA-1.2, Manufacturer's Guidance Manual	None.
Turbidity Meter (LaMotte 2020 or equivalent)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance; calibrations must bracket expected values; Initial Calibration Verification (ICV) must be <5 Nephelometric Turbidity Units (NTUs).	Operator correction or replacement	Tetra Tech FOL or designee	Manufacturer's Guidance Manual	To be used to determine the need to collect Dissolved Metals samples (if >5 NTUs).
Electric Water Level Indicator and Oil/Water Interface Probe	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Tetra Tech FOL or designee	GH-1.2, Manufacturer's Guidance Manual	None.
Photoionization Detector	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator correction or replacement	Tetra Tech FOL or designee	SA-1.1, SA-1.2, SA-1.3, GH-1.2, GH-1.3, GH-1.5, GH-2.8, Manufacturer's Guidance Manual	To be used to determine the soil boring depth that is most impacted for biased sample collection.

Notes:

1 Activities may include: calibration, verification, testing, maintenance, and/or inspection.
2 Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet No.21).

SAP Worksheet No. 23 – Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 21, 09/21/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Metals Digestion	NA/Preparation	Empirical	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique, Methods USEPA SW846 7470A and 245.1, CLP-M 4.1 (Revision 18, 04/11/10)	Definitive	Groundwater, and aqueous QC samples/ Mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique, Methods SW846 7471A, 7471B, 245.5, and CLP-ILM 4.1 (Revision 19, 04/11/10)	Definitive	Soil and sediment/ Mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Metals	ICP-AES	Empirical	N
Empirical SOP-164	Distillation of Aqueous/Solid Samples for Cyanide, Total and Amenable, SW846 Method 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM4500-CN C, G, 18th and 19th ED/ USEPA CLP ILMO 4.1 (Revision 15, 03/25/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Cyanide Digestion	NA/ Distillation	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-175	Post-Distillation Analysis for Cyanide by Lachat Flow Injection Analyzer, Methods 335.4, SW846 9012A, USEPA-CLP 4.1; Addendum for USEPA CLP ILM 05.2 Aqueous/Soil/ Sediment (Revision 11, 09/07/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Cyanide	Automated Ion Analyzer	Empirical	N
Empirical SOP-201	Gas Chromatography Mass Spectrometry (GC/MS) semivolatiles and Low-Concentration PAHs using USEPA Method 625 and SW846 Method 8270C and 8270D, Including Appendix IX Compounds (Revision 20, 04/26/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ SVOCs	GC/MS	Empirical	N
Empirical SOP-202	GC/MS Volatiles using USEPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 23, 09/09/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ VOCs	GC/MS	Empirical	N
Empirical SOP-211	Gas Chromatography/ Electron Capture Detector (GC/ECD) Organochlorine Pesticides/ PCBs using USEPA Method 608608.2 or SW846 Method 8081A/8082 or 8081B/8082A (Revision 22, 07/07/10)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ PCBs	GC/ECD	Empirical	N
Empirical SOP-221	TOC SM5310C, SW846 Method 9060/9060A and Lloyd Kahn Method (Revision 9, 07/12/10)	Definitive	Sediment/ TOC	TOC Analyzer	Empirical	N
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction using SW-846 Method 5035 for 8260B Analysis (Revision 9, 9/07/10)	Definitive	Soil and sediment/ VOCs Extraction	GC/MS	Empirical	N
Empirical SOP-300	GC/MS- Semivolatile BNA-Aqueous Matrix Extraction using SW-846 Method 3510C for 8270/625 Analysis (Revision 18, 04/26/10)	Definitive	Groundwater, and aqueous QC samples/ SVOCs Extraction	NA/ Extraction	Empirical	N

LAB SOP NUMBER	TITLE, REVISION DATE, AND/OR NUMBER	DEFINITIVE OR SCREENING DATA	MATRIX AND ANALYTICAL GROUP	INSTRUMENT	ORGANIZATION PERFORMING ANALYSIS	MODIFIED FOR PROJECT WORK? (Y/N)
Empirical SOP-302	Pesticide/PCBs, Aqueous Matrix Extraction for USEPA 608/608.2 and SW846 Method 8081A/8082 Using SW846 Method 3510C (Revision 17, 04/26/10)	Definitive	Groundwater, and aqueous QC samples/ PCBs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-343	BNA, Pesticides/PCBs, and TPH non-Aqueous Matrix (Microwave Extraction) using SW-846 3546 (Revision 01, 09/09/10)	Definitive	Soil and sediment/ SVOCs and PCBs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-QS-10	Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures (Revision 14, 09/07/10)	NA	Log-in	NA/ Log-in	Empirical	N
Empirical SOP-QS-14	Analytical Laboratory Waste Disposal (Revision 6, 08/31/10)	Definitive	Disposal	NA / Disposal	Empirical	N
CFA CF-OA-E-001	SOP for Dioxin/ Furan/ PCB Congener Sample Processing (Revision 2, February 2010) (not in Appendix B)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Dioxins/Furans	NA/ Extraction	CFA	N
CFA CF-OA-E-002	Standard Operating Procedure for the Analysis of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans using High Resolution GC/MS (USEPA SW-846 Method 8290, USEPA Method 1613B, USEPA SW-846 Method 0023A) (Revision 4, September 2009) (not in Appendix B)	Definitive	Soil, sediment, groundwater, and aqueous QC samples/ Dioxins/Furans	High Resolution GC/MS	CFA	N
CFA CF-SR-E-001	Sample Receipt, Login, and Storage (not in Appendix B)	Definitive	Log-in	NA/ Log-in	CFA	N
CFA CF-LB-G-001	Laboratory Waste Management Plan (not in Appendix B)	Definitive	Disposal	NA/ Disposal	CFA	N

Copies of laboratory SOPs listed are included in Appendix B, except for proprietary SOPs, which are noted as not included, but are available for review.

SAP Worksheet No. 24 – Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS VOCs	Bromofluoro-benzene (BFB) Tune	Prior to each Initial Calibration (ICAL) and at the beginning of each 12-hour period.	Must meet the ion abundance criteria required by the method (SW8260B; Section 7.3.1; Table 4).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst/ Supervisor	Empirical SOP-202
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, for major instrument changes, or when continuing calibration verification (CCV) does not meet criteria.	The average response factor (RF) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent relative standard deviation (%RSD) for RFs for calibration check compounds (CCCs) must be ≤ 30%; and %RSD for each target analyte must be ≤ 15%, or the linear regression correlation coefficient (r) must be ≥ 0.995; or the coefficient of determination (r^2) must be ≥ 0.99 (6 points are required for second order).	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst / Supervisor	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor	
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for all target analytes must be within 80-120% of true values.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS VOCs (continued)	CCV	Perform one per 12-hour analysis period after tune and before sample analysis.	The minimum RF for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target analytes and surrogates must be ≤ 20%.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	Empirical SOP-202
GC/MS SVOCs and Low Level PAHs	Tune Verification – decafluoro-triphenyl-phosphine (DFTPP)	Prior to each ICAL and at the beginning of each 12-hour analytical sequence.	Must meet the ion abundance criteria required by the method (SW8270D; Section 7.3.1; Table 3), dichlorodiphenyltrichloroethane (DDT) degradation ≤20% (exclusion for LOW PAH analysis).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst/ Supervisor	Empirical SOP-201
	Breakdown Check (DDT only)	At the beginning of each 12-hour analytical sequence.	The degradation must be ≤ 20% for DDT to verify inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is ≤20% for DDT.	Analyst/ Supervisor	
	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria.	The average RF for SPCCs must be ≥ 0.050. The %RSD for RFs for CCCs must be ≤ 30%; and %RSD for each target analyte must be ≤ 15%, or r must be ≥ 0.995; or r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
	ICV – Second Source	Perform after each ICAL, prior to beginning a sample run.	The %R of all target analytes must be within 75-125% of the true value. SPCC RFs must be ≥ 0.050; CCCs must be ≤ 20%D.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	RT Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst / Supervisor	
	Evaluation of RTs	With each sample.	RT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/MS SVOCs and Low Level PAHs (continued)	CCV	Analyze a standard at the beginning of each 12-hour shift after tune and before sample analysis.	SPCC RFs must be ≥ 0.050; All target analytes and surrogates must be ≤ 20%D.	If %D is high and sample result is non detect (ND), qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	Empirical SOP-201
GC/ECD Pesticides	Breakdown Check – Endrin/DDT	At the beginning of each 12 hour period, prior to analysis of samples.	The degradation must be ≤ 15% for both DDT and Endrin to verify the inertness of the injection port.	Correct the problem then repeat breakdown check. No samples shall be run until degradation is ≤15% for both DDT and Endrin.	Analyst/ Supervisor	Empirical SOP-211
	ICAL – A minimum of a 5-point calibration of individual pesticides, with a mid-point calibration of toxaphene and chlordane, is prepared for all target analytes	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria, prior to sample analysis.	The % RSD for RFs for each target analyte must be ≤ 20%, or r must be ≥ 0.995, or r^2 must be ≥ 0.99 (minimum of 6 points required for second order).	Correct problem and repeat ICAL. If a single-point calibration for toxaphene or chlordane is used and toxaphene or chlordane is identified in analysis of a sample, a minimum of a 5-point ICAL of the identified compound with reanalysis of sample must be performed. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R of all target analytes must be within 80-120% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %D of all target analytes must be ≤20%.	If %D is high and sample result is nondetect, qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/ECD PCBs	ICAL - A minimum of a 5-point calibration of Aroclor 1660 (1016/1260 mixture) is prepared	Upon instrument receipt, major instrument change, when CCV does not meet criteria.	Option 1: %RSD must be ≤20% for Aroclor 1016/1260. If not met, Option 2: r must be ≥ 0.995; or Option 3: r^2 must be ≥ 0.99 for 6-point calibration. Mid-point calibration of other Aroclors – if an Aroclor is detected in a sample, a minimum of 5-point ICAL must be performed and meet the above criteria.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	Empirical SOP-211
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R of all target analytes must be within 80-120% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %D of all target analytes must be ≤20%.	If %D is high and sample result is ND, qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
ICP-AES Metals	ICAL - a 1-point calibration per manufacturer's guidelines is prepared for all target analytes	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-100/105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R of all target analytes must be within 90-110% of true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R of all target analytes must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	
	Low-Level Check Standard (if using 1-point ICAL)	Daily after 1-point ICAL and before samples.	The %R of all target analytes must be within 80-120% of true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst / Supervisor	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning of an analytical run.	The absolute value of ICS A recoveries for non-spiked analytes must be < LOD; and ICS B recoveries must be within 80-120 %R of true value.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	
Mercury Analyzer Mercury	ICAL – A minimum of a 5-point calibration is prepared	Perform daily prior to sample analysis.	The %RSD for RFs must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	Empirical SOP-103/104
	ICB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	ICV - Second Source	Once after each ICAL and prior to sample analysis	The %R must be within 90-110% of the true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst / Supervisor	
	CCB	After each CCV, after every 10 samples, and at the end of the sequence	No mercury detected > LOD.	Investigate source of contamination, rerun any samples not bracketed by passing blanks.	Analyst / Supervisor	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% of the true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
	CCV (undistilled)	CCV (undistilled) at the beginning and end of each run sequence and after every 10 samples.	The %R must be within 90-100% of true value.	If the CCV (undistilled) fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst / Supervisor	
Lachat Cyanide	ICAL– A minimum of a 6-point calibration is prepared	Perform daily prior to sample analysis.	r must be ≥ 0.995. All calibration standards must be distilled if samples are expected to contain sulfides.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Supervisor	Empirical SOP-164/175
	ICV – Second Source	At the start of every sequence prepared fresh daily (undistilled), prior to sample analysis.	The %R must be within 85-115% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst, Supervisor	
	ICB	After the ICV (undistilled).	Must be < LOQ for the target analyte.	Determine source of contamination, correct problem, and restart sequence.	Analyst, Supervisor	
	CCV	Every 10 samples (undistilled).	The %R must be within 85-115% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Supervisor	
	ICV (distilled, high and low)	Once per ICAL.	The %R must be within 85-115% of true value.	Determine cause for failure, correct problem, and redistill standards.	Analyst, Supervisor	
High Resolution GC/MS Dioxins/ Furans	Tune / Mass Resolution Check	At the beginning and the end of each 12-hour period of analysis.	Static resolving power must be ≥ 10,000 (10% valley) for identified masses per method and lock-mass ion between lowest and highest masses for each descriptor and level of reference must be ≤ 10% full-scale deflection.	Retune instrument and verify. Assess data for impact. If end resolution is less than 10,000, narrate or re-inject, as necessary.	Analyst, Supervisor	CFA CF-OA-E-002

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
	GC Column Performance Check Solution (CPCS)	Prior to ICAL or CCV.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers must result in a valley of ≤ 25% per method; 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 CA is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.	Analyst, Supervisor	
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Prior to sample analysis, as needed by the failure of CCV, and when a new lot is used as a standard source.	Ion abundance ratios must be within limits specified in SOP; and signal to noise ratio (S/N) must be ≥ 10:1 for all target analyte ions; and RSD must be ≤ 20% for RFs for all 17 unlabelled standards and 9 labeled ISs.	Correct problem, then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Supervisor	
	CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios must be in accordance with SOP; and RF (unlabelled standards) must be ≤ 20%D of average RF from ICAL; and RF (labeled standards) must be ≤ 30%D of average RF from ICAL.	Correct problem, repeat CCV. If CCV 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, then use mean RF from bracketing CCVs to quantitate impacted samples instead of the ICAL mean RF value. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labeled), then run a new ICAL within 2 hours, and re-quantitate samples. Otherwise, reanalyze samples with positive detections.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
TOC Analyzer TOC	ICAL – a minimum of a 5-point calibration is prepared	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The RSD for RFs for the target analyte must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Department Manager	Empirical SOP-221
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst, Department Manager	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Department Manager	

SAP Worksheet No. 25 – Analytical Instrument/Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
GC/MS	Check pressure and gas supply daily. Bake out trap and column, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in Equipment Maintenance SOP.	VOCs/ SVOCs (Including Low Level PAHs)	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-201/202
GC/ECD	Check pressure and gas supply daily. Bake out column, change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in Equipment Maintenance SOP.	Pesticides and PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	If % D > 20% and samples are < LOD, narrate. If %D > 20% only on one column, narrate. If % D > 20% for closing CCV, and is likely due to matrix interference, narrate. Otherwise, reanalyze all samples since the last acceptable CCV.	Analyst/ Supervisor	Empirical SOP-211/329
ICP-AES	Clean the torch assembly and the spray chamber when they become discolored or when degradation in data quality is observed. Clean the nebulizer, and check the argon supply. Replace the peristaltic pump tubing as needed.	Metals (Except Mercury)	Inspect the torch, nebulizer chamber, pump, and tubing	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-100/105
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in Equipment Maintenance SOP.	Mercury	Inspect the tubing, filter, and the optical cell	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-103/104

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference[1]
Lachat	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, and change dispensing needle, all as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Cyanide	Tubing and rollers.	Prior to ICAL or as necessary.	Acceptable ICAL and CCV.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing CCV.	Analyst, Supervisor	Empirical SOP-164/175
High Resolution GC/MS	Parameter Setup	Dioxins/ Furans	Physical check.	Initially; prior to daily calibration check.	Correct Parameters.	Reset if incorrect.	Analyst, Supervisor	CFA CF-OA-E-002
	Tune Check		Conformance to instrument tuning.	Initially; prior to daily calibration check.	Compliance to ion abundance criteria.	Correct the problem and repeat tune check.	Analyst, Supervisor	
TOC Analyzer	Replace sample tubing, clean sample boat, replace syringe.	TOC	Tubing, sample boat, syringe.	As needed.	Must meet ICAL and continuing calibration criteria.	Repeat maintenance activity or remove from service.	Analyst/ Supervisor	Empirical SOP-221

Notes:

1 Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet No.23).

SAP Worksheet No. 26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): George Ten Eyck / Tetra Tech
Sample Packaging (Personnel/Organization): George Ten Eyck / Tetra Tech
Coordination of Shipment (Personnel/Organization): George Ten Eyck / Tetra Tech
Type of Shipment/Carrier: Overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/ Empirical and CFA
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical and CFA
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory, Dioxins Preparation Laboratory / Empirical and CFA
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory, Dioxins Laboratory/ Empirical and CFA
SAMPLE ARCHIVING
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/ Empirical and CFA

SAP Worksheet No. 27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures

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

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of samples. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP-05 (Appendix A).

Chain-of-Custody Procedures

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech SOP-04 provides further details on the chain-of-custody procedure (Appendix A). Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratories.

Laboratory Sample Custody Procedures

Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (Empirical SOP-QS-10 [Sample Receiving and Sample Storage] and CFA Sample Receipt, Login, and Storage SOP CF-SR-E-001), which are provided in Appendix B.

Sample Designation System

Each sample collected for analysis will be assigned a unique sample tracking number that will consist of a multi-segment alphanumeric code that identifies the site, sample location, sample type (sample medium or QC sample designation), sample location, and sample depth. SOP-03 addresses sample identification nomenclature (Appendix A).

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method / SOP Reference	**SW-846 8260B Empirical SOP-202**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples	All target analytes must be ≤ ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination and rerun method blank prior to analysis of samples, if possible. Evaluate the samples and associated QC: if blank results are above LOQ, then report sample results that are <LOQ or >10X the blank concentration. Re-prepare and reanalyze blank and those samples that were >LOQ and <10X the blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) (not required)	One per preparatory batch of 20 or fewer samples.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. RPD must be ≤ 30% (for LCS/LCSD, if LCSD is performed).	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and acceptable, then narrate. If the LCS %Rs are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze the LCS and associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**VOCs**
Analytical Method / SOP Reference	**SW-846 8260B Empirical SOP-202**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (IS)	Every field sample, standard, and QC sample - three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Inspect mass spectrometer and gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Surrogates	All field and QC samples - four per sample- Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then re-prepare and reanalyze sample for confirmation of matrix interference when appropriate.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs and Low Level PAHs**
Analytical Method / SOP Reference	**SW-846 8270C, 8270C-Low Empirical SOP-201**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination. Rerun method blank prior to analysis of samples if possible. Evaluate the samples and associated QC: if blank results are above RL, report sample results that are < RL or > 10X the blank concentration. Reanalyze blank and samples >RL and < 10X the blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except low level PAHs that are provided in Appendix B).	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12-hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze the LCS and associated samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except low level PAHs that are provided in Appendix B). The RPD between MS and MSD should be ≤ 30%.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met, unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**SVOCs and Low Level PAHs**
Analytical Method / SOP Reference	**SW-846 8270C, 8270C-Low/ Empirical SOP-201**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every field sample, standard, and QC sample - six per sample 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Re-analyze affected samples.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Surrogates	All field and QC samples - six per sample 2-Fluorophenol Phenol-d6 Nitrobenzene-d5 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d14	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM (except low level PAHs that are provided in Appendix B).	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, then check instrument performance; if problem is found, then correct and reanalyze sample. (3) If still out, then re-extract and reanalyze sample. (4) If reanalysis is out, then flag data.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**Pesticides/ PCBs**
Analytical Method / SOP Reference	**SW-846 8081B, 8082A Empirical SOP-211**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, then report sample results that are <LOQ or > 10X the blank concentration. Otherwise, re-prepare a blank and samples >LOQ and <10X LOQ.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix Pesticide: Spike with single component pesticide mix PCB: Spike with Aroclor 1016/1260 mix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If an MS/MSD was performed and is acceptable, then narrate. If a LCS/ LCSD were performed and only one of the set was unacceptable, then narrate. If the LCS recovery is high, but the sample results are <LOQ, then narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix (spike same as LCS)	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	Evaluate the samples and associated QC and if the LCS results are acceptable, then narrate. If both the LCS and MS/MSD are unacceptable, then re-prepare the samples and QC.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**Pesticides/ PCBs**
Analytical Method / SOP Reference	**SW-846 8081B, 8082A Empirical SOP-211**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	All field and QC samples - two per sample Tetrachloro-m-xylene Decachlorobiphenyl	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	No corrective will be taken when one surrogate is within criteria. If surrogates recoveries are high and sample is <LOQ, then no CA is taken. If surrogates recoveries are low, then the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. For Method 8082, report the higher of the two concentrations, unless there is interference.	None. Apply "J" flag if RPD >40% and discuss in the case narrative.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury), Total and Dissolved**
Analytical Method / SOP Reference	**SW-946 6010C, 7470A, 7471A Empirical SOP-103, 104, 105**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ, except common lab contaminants, which must be < LOQ.	If the blank value > LOQ, then report sample results. If the blank value < LOQ or > 10x the blank value, then redigest. If blank value is less than negative LOQ, then report sample results. If > 10x the absolute value of the blank result, then redigest and reanalyze.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%R must be within 80-120% of true value.	Evaluate and reanalyze, if possible. If the LCS recoveries are high, but the sample results are < LOQ, then narrate. Otherwise, re-digest and reanalyze all associated samples for failed target analyte(s).	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%R should be within 80-120% of true value (if sample is < 4x spike added).	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits
Serial Dilution (ICP Only)	One per preparatory batch with sample concentration(s) >50x LOD	The 5-fold dilution result must agree within ±10%D of the original sample result if result is >50x LOD.	Perform post spike addition.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**Metals (Including Mercury), Total and Dissolved**
Analytical Method / SOP Reference	**SW-946 6010C, 7470A, 7471A Empirical SOP-103, 104, 105**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post Spike (ICP Only)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with "J".	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, Groundwater, and Aqueous QC Samples**
Analytical Group	**Cyanide**
Analytical Method / SOP Reference	**SW-946 9010B, 9012A Empirical SOP-164, 175**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	Cyanide must be ≤ ½ LOQ.	Stop analysis, correct problem, and recalibrate.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Re-prepare and reanalyze the LCS and all associated samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Flag data with an "N", unless recovery is > 4x the spike added; If the sample results exceed 4x the spike added, then spike the un-spiked aliquot of the sample at 2x the indigenous level or 2x the LOQ.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	RPD should be ≤ 20%, if concentration is > 5x LOQ; or within ± the LOQ, if the concentration is < 5x LOQ.	Flag data for associated samples with an "*".	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits

Matrix	**Soil, Sediment, and Aqueous QC Samples**
Analytical Group	**Dioxins/Furans**
Analytical Method / SOP Reference	**SW-846 8290A/ CFA CF-OA-E-002**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples	All target analytes must be ≤ LOQ.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank. "Totals" are not considered "target analytes" – no CA or flagging is necessary for "totals".	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
IS	Every field sample, standard and QC sample	The %R for each IS must be within 25-150%, per method.	Correct problem, then re-prepare and reanalyze the samples with failed IS.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed target analytes, if sufficient sample material is available.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	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.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits

Matrix	**Sediment Samples**
Analytical Group	**TOC**
Analytical Method / SOP Reference	**Lloyd Kahn Empirical SOP-221**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparatory batch of 20 or fewer samples	The target analyte must be ≤ ½ LOQ.	Correct problem, re-prepare and reanalyze along with associated samples.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
Calibration Blank	At the beginning of analytical sequence, after every 10 samples, at the end of the sequence	The target analyte concentration must be < LOD.	Correct problem, re-prepare, and reanalyze along previous 10 samples.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples	%R must be within 80-120% of true value.	Correct problem, re-prepare, and reanalyze along with associated samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix	%R should be within 80-120% of true value. RPD should be ≤ 20%.	Contact client for guidance.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits

SAP Worksheet No. 29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook (and sampling notes) Field Sample Forms forms (e.g. boring logs, sample log sheets, drilling logs, etc.) Chain-of-Custody Records Sample Shipment Air Bills Sampling Instrument Calibration Logs Photographs FTMR Forms This SAP Field Sampling SOPs Health and Safety Plan	Field documents will be maintained in the project file located in the Tetra Tech Cincinnati, Ohio office.
Laboratory Documents Sample Receipt, Custody, and Tracking Record Equipment Calibration Logs Analysis Run Logs Corrective Action Forms Reported Results for Standards, QC Checks, and QC Samples Raw Data Data Completeness Checklist	Laboratory documents will be included in the hardcopy and Portable Document Format (PDF) deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Cincinnati, Ohio project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings All versions of the SAP All letter and e-mail correspondence with regulatory agencies, including approvals and comments Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Cincinnati, Ohio project file.
Reports SWMU 11 RFI Report.	All versions of the RFI Report, and all support documents (e.g., Data Validation Reports) will be stored in hardcopy in the Tetra Tech Cincinnati, Ohio project file and electronically in the server library.

SAP Worksheet No. 30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

MATRIX	ANALYTICAL GROUP	SAMPLE LOCATIONS/ IDENTIFICATION NUMBERS	ANALYTICAL METHOD	DATA PACKAGE TURNAROUND TIME	LABORATORY / ORGANIZATION (name and address, contact person and telephone number)	BACKUP LABORATORY/ ORGANIZATION (name and address, contact person, and telephone number)
Groundwater, soil, and sediment	VOCs	See Worksheet No.18	SW-846 8260B	21 calendar days	Kim Kostzer kkostzer@empirlabs.com Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 615-345-1115	NA
	SVOCs (Including Low Level PAHs)		SW-846 8270C and 8270C-Low			
	Pesticides		SW-846 8081B			
	PCBs		SW-846 8082A			
	Metals, Total and Dissolved (Including Mercury)		SW-846 6010C/ 7470A/ 7471A			
	Cyanide		SW-846 9012A			
	TOC		Lloyd Kahn			
Soil and sediment	Dioxins/Furans	See Worksheet No.18	SW-846 8290A	21 calendar days	Chris Cornwell ccornwell@cfanalytical.com Cape Fear Analytical, LLC 3306 Kitty Hawk Road Suite 120 Wilmington, NC 28405 910-795-0422	NA

SAP Worksheet No. 31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

ASSESSMENT TYPE	FREQUENCY	INTERNAL OR EXTERNAL	ORGANIZATION PERFORMING ASSESSMENT	PERSON(S) RESPONSIBLE FOR PERFORMING ASSESSMENT (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR RESPONDING TO ASSESSMENT FINDINGS (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR IDENTIFYING AND IMPLEMENTING CA (title and organizational affiliation)	PERSON(S) RESPONSIBLE FOR MONITORING EFFECTIVENESS OF CA (title and organizational affiliation)
Laboratory System Audit[1]	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical and CFA	Laboratory QAM or Laboratory Manager, Empirical and CFA	Laboratory QAM or Laboratory Manager, Empirical and CFA

[1] Empirical and CFA are DoD ELAP accredited by a recognized Accrediting Body. The DoD ELAP accreditation letter is included in Appendix B.

SAP Worksheet No. 32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

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, organization)	**Timeframe for Response**
Laboratory System Audit	Written audit report	Randy Ward, Laboratory QAM, Empirical Mike Larkins, Laboratory QAM, CFA	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet No. 33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per Sample Delivery Group (SDG)	Within 3 weeks after receiving the data from the laboratory	Project Chemist or Data Validator, Tetra Tech	PM, Tetra Tech; project file
Major Analysis Problem Identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem – on the same day	QAM, Tetra Tech	PM, Tetra Tech; QAM, Tetra Tech; Program Manager, Tetra Tech; project file
Project Monthly Progress Report	Monthly for duration of the project	Monthly	PM, Tetra Tech	PM, Tetra Tech; QAM, Tetra Tech; Program Manager, Tetra Tech; project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical and CFA	PM and project file, Tetra Tech

SAP Worksheet No. 34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

VERIFICATION INPUT	DESCRIPTION	INTERNAL/ EXTERNAL	RESPONSIBLE FOR VERIFICATION (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that the samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	The Empirical and CFA Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Empirical and CFA 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Analytical SOPs/ Analytical Data Packages	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, Empirical and CFA
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM verbally or via e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical and CFA
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet No.20 were collected as required.	Internal	FOL or designee, Tetra Tech

VERIFICATION INPUT	DESCRIPTION	INTERNAL/ EXTERNAL	RESPONSIBLE FOR VERIFICATION (name, organization)
EDDs/Analytical Data Packages	Each EDD will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech

Notes: Verification includes field data verification and laboratory data verification. Verification inputs as per Worksheet No.34 will be checked.

SAP Worksheet No. 35 -- Validation (Steps IIa and IIb) Process Table

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/ Sample Log Sheets	Sample Coordinates - Ensure that sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis, the custody records are complete, and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH for chemically preserved samples meet the requirements listed in Worksheet No.19. Ensure that the analyses were performed within the holding times listed in Worksheet No.19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	Accuracy - Ensure that the laboratory QC samples listed in Worksheet No.28 were analyzed and that the MPCs listed in Worksheet No.12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Precision - Check the field sampling precision by calculating the RPD for FD samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet No.12.	
		Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet No.36. Check that all data have been transferred correctly and completely to the final Structured Query Language database.	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in Worksheet No.15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		PSLs - Discuss the impact on reported DLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PSLs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PSLs listed in Worksheet No.15.	
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

SAP Worksheet No. 36 -- Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

STEP **IIa / IIb**	MATRIX	ANALYTICAL GROUP	VALIDATION CRITERIA	DATA VALIDATOR (title and organizational affiliation)
IIa and IIb	Groundwater, surface water, soil and sediment	VOCs, SVOCs (Including Low Level PAHs), Pesticides, and PCBs	Data validation will be performed using criteria for SW-846 Methods 8260B, 8270C, 8270C-Low, 8081B, and 8082A listed in Worksheets Nos.12, 15, 24, and 28 and the current DoD QSM. If not included in the aforementioned, then the logic outlined in the "USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review" USEPA-540/R-99-008, (USEPA, October 1999) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Groundwater, surface water, soil and sediment	Metals and Cyanide	Data validation will be performed using criteria for SW-846 Methods 6010C, 7470A, 7471A, and 9012A listed in Worksheets Nos.12, 15, 24, and 28 and the current DoD QSM. If not included in and the aforementioned, then the logic outlined in the "USEPA CLP National Functional Guidelines for Inorganic Data Review", USEPA 540-R-04-004, (USEPA, October 2004) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech
IIa and IIb	Soil and sediment	Dioxins/Furans	Data validation will be performed using criteria for SW-846 Method 8290A listed in Worksheets Nos.12, 15, 24, and 28 and the current DoD QSM. If not included in the aforementioned, then the logic outlined in the "National Functional Guidelines for Chlorinated Dioxin/Furan Data Validation", (USEPA, September 2005) will be used to apply qualifiers to data.	Data Validation Specialist, Tetra Tech

STEP **IIa / IIb**	MATRIX	ANALYTICAL GROUP	VALIDATION CRITERIA	DATA VALIDATOR (title and organizational affiliation)
IIa and IIb	Sediment	TOC	Limited data validation* will be performed using criteria for Methods Lloyd Kahn listed in Worksheets Nos.12, 15, 24, and 28 and the current DoD QSM.	Data Validation Specialist, Tetra Tech

Notes:

* Limited data validation. Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding Times, Calibrations, Blank Contamination, and Detection Limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal report, similar to a full data validation report, is prepared but the scope is more limited than a full validation report. The data packages provided by the laboratory will be expansive enough to allow future complete formal data validation, if necessary.

SAP Worksheet No. 37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

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

Completeness

- For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the Project Team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and Project Risk Assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

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

Accuracy

- The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet No. 28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, MS, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

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

Comparability

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

analysis is required.

Sensitivity

- The Tetra Tech Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in Worksheet No. 15 were achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Tetra Tech Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., Normal versus log-normal) and estimates of data variability. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

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

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the Tetra Tech PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

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

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the Navy ERSM, and the IDEM RPM. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

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

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

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FIGURES

10-1 Base and Site Location Map
10-2 Site Layout – 2005 Aerial Photograph
10-3 Site Plan
10-4 IA-6 Site Layout – 2005 Aerial Photograph, Building 2981 Concrete Tank
10-5 Conceptual Site Model Schematic
10-6 Human Conceptual Exposure Model Diagram
10-7 Ecological Conceptual Exposure Model Diagram
17-1 IA-1, IA-2, IA-3, and IA-5 Soil Sampling Locations
17-2 IA-4 Soil Sampling Locations – Downwind Areas
17-3 IA-6 Soil Sampling Locations – Building 2981 Concrete Tank
17-4 IA-7 Sediment and SWMU 11 Groundwater Sampling Locations

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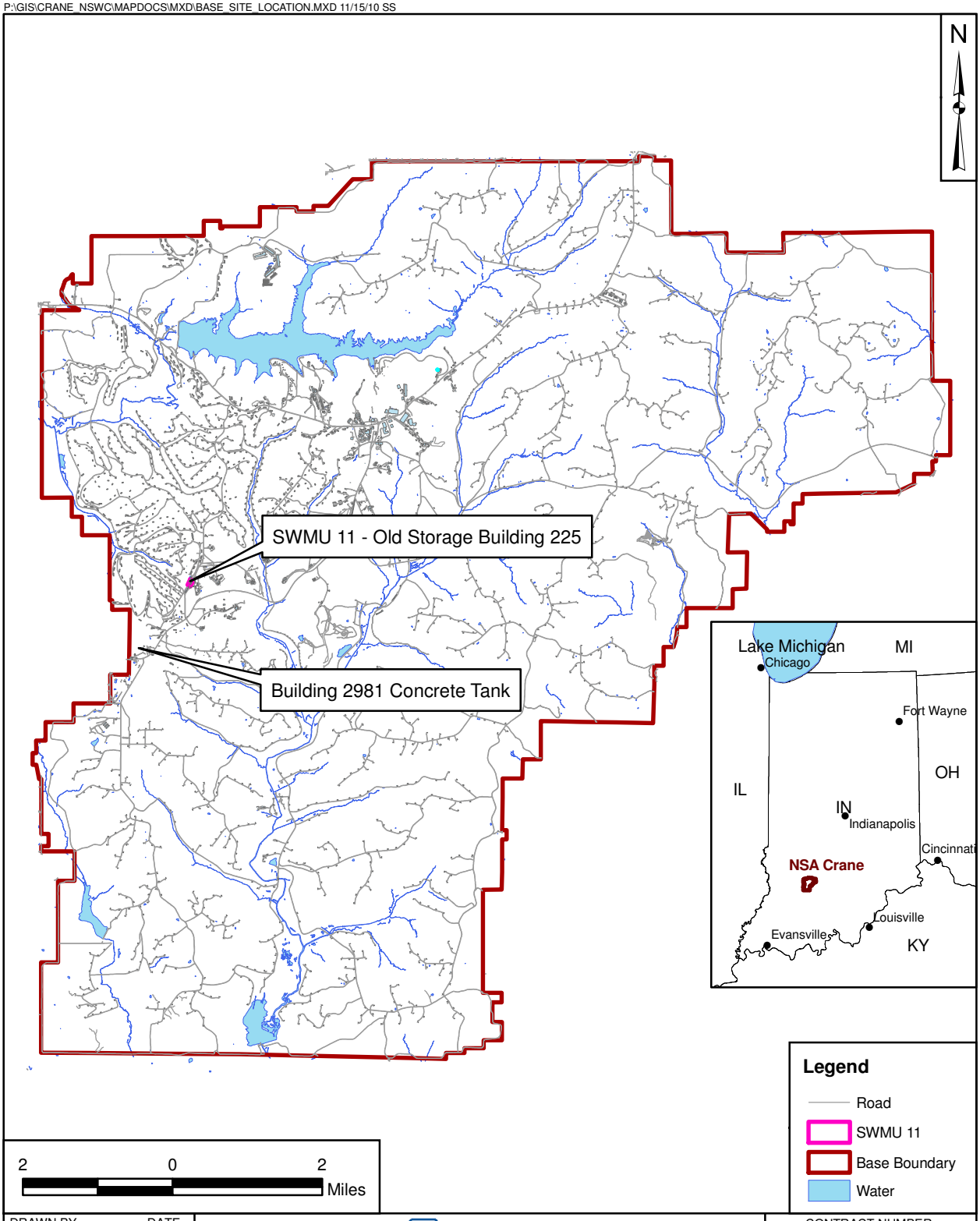

DRAWN BY S. STROZ	DATE 02/08/10	TETRA TECH	CONTRACT NUMBER CTO F273
CHECKED BY G. TEN EYCK	DATE 11/15/10	BASE AND SITE LOCATION MAP SWMU 11 - OLD STORAGE BUILDING 225 NSA CRANE CRANE, INDIANA	APPROVED BY ___ DATE ___
REVISED BY S. STROZ	DATE 11/15/10		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. 10-1 REV 0

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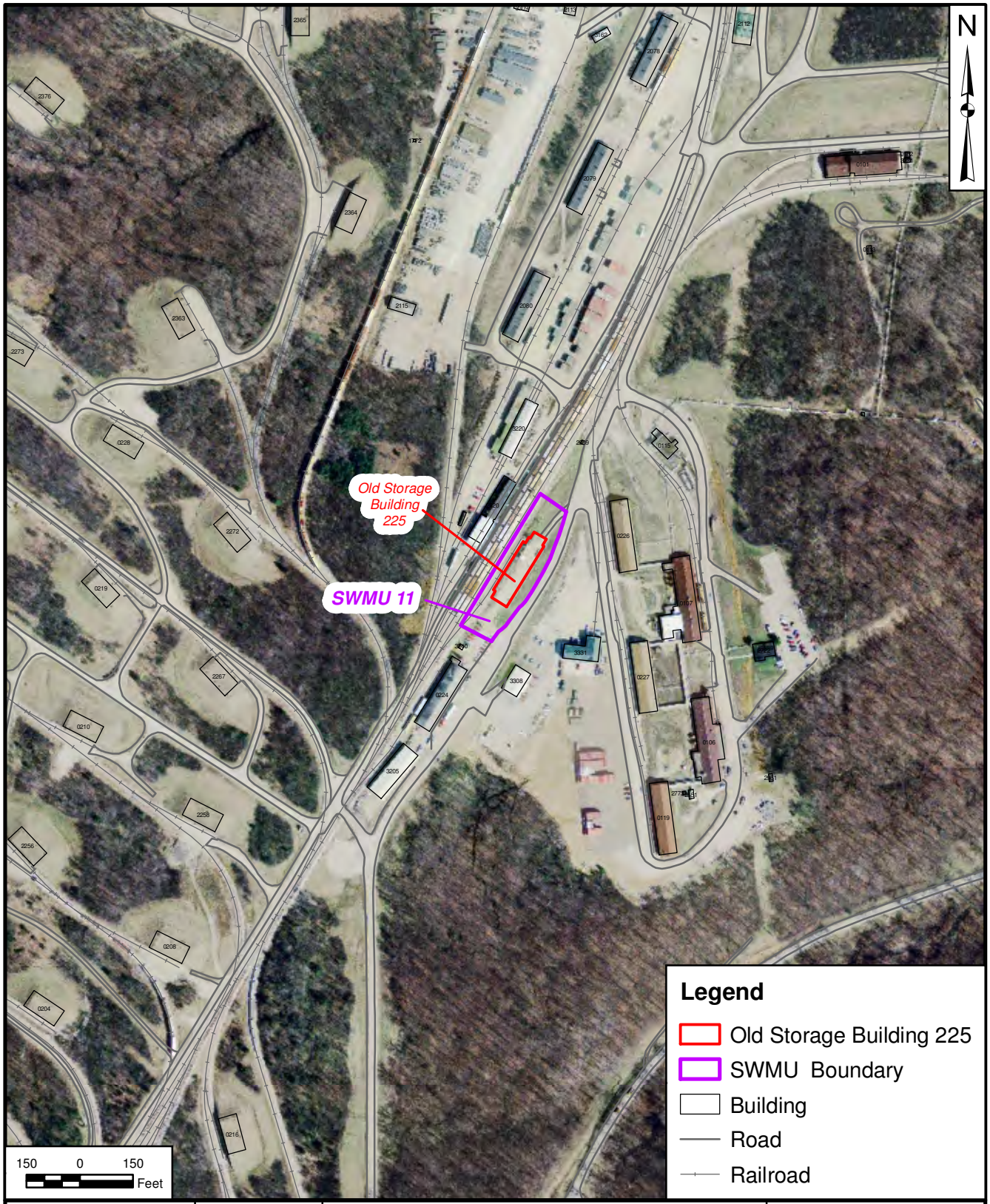

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CHECKED BY: K. LOSEKAMP	DATE: 12/20/10	
REVISED BY: S. STROZ	DATE: 12/20/10	
SCALE: AS NOTED		

NAVFAC

SITE LAYOUT - 2005 AERIAL PHOTOGRAPH
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER: CTO F27E	
APPROVED BY: A. KLIMEK	DATE: 04/12/10
APPROVED BY: ____	DATE: ____
FIGURE NO.: 10-2	REV: 0

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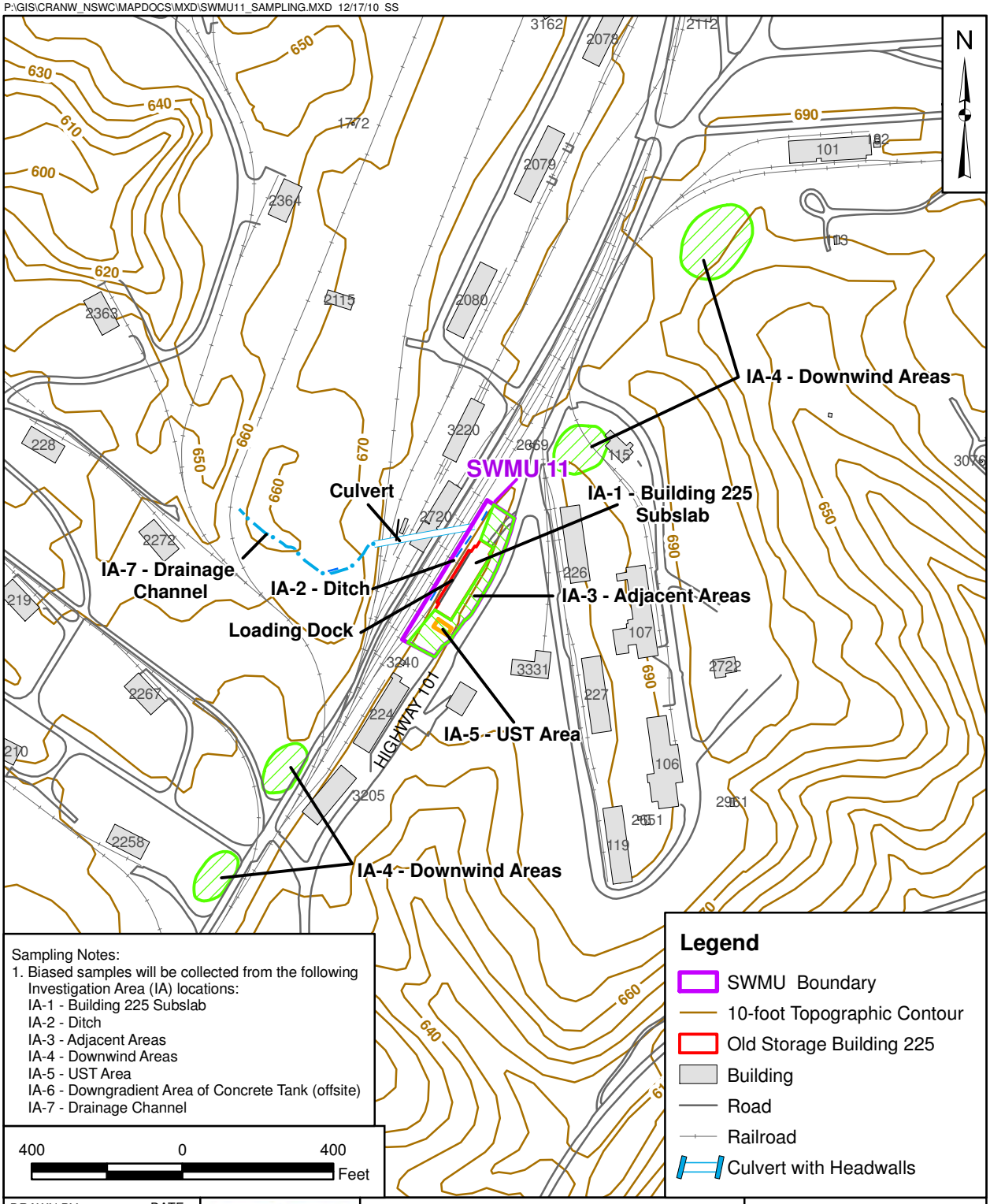

Sampling Notes:
1. Biased samples will be collected from the following Investigation Area (IA) locations:
   IA-1 - Building 225 Subslab
   IA-2 - Ditch
   IA-3 - Adjacent Areas
   IA-4 - Downwind Areas
   IA-5 - UST Area
   IA-6 - Downgradient Area of Concrete Tank (offsite)
   IA-7 - Drainage Channel

DRAWN BY / DATE	S. STROZ 02/08/10
CHECKED BY / DATE	K. LOSEKAMP 12/17/10
REVISED BY / DATE	S. STROZ 12/17/10
SCALE	AS NOTED

NAVFAC

SITE PLAN
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	CTO F27E
APPROVED BY / DATE	A. KLIMEK 04/14/10
APPROVED BY / DATE	— —
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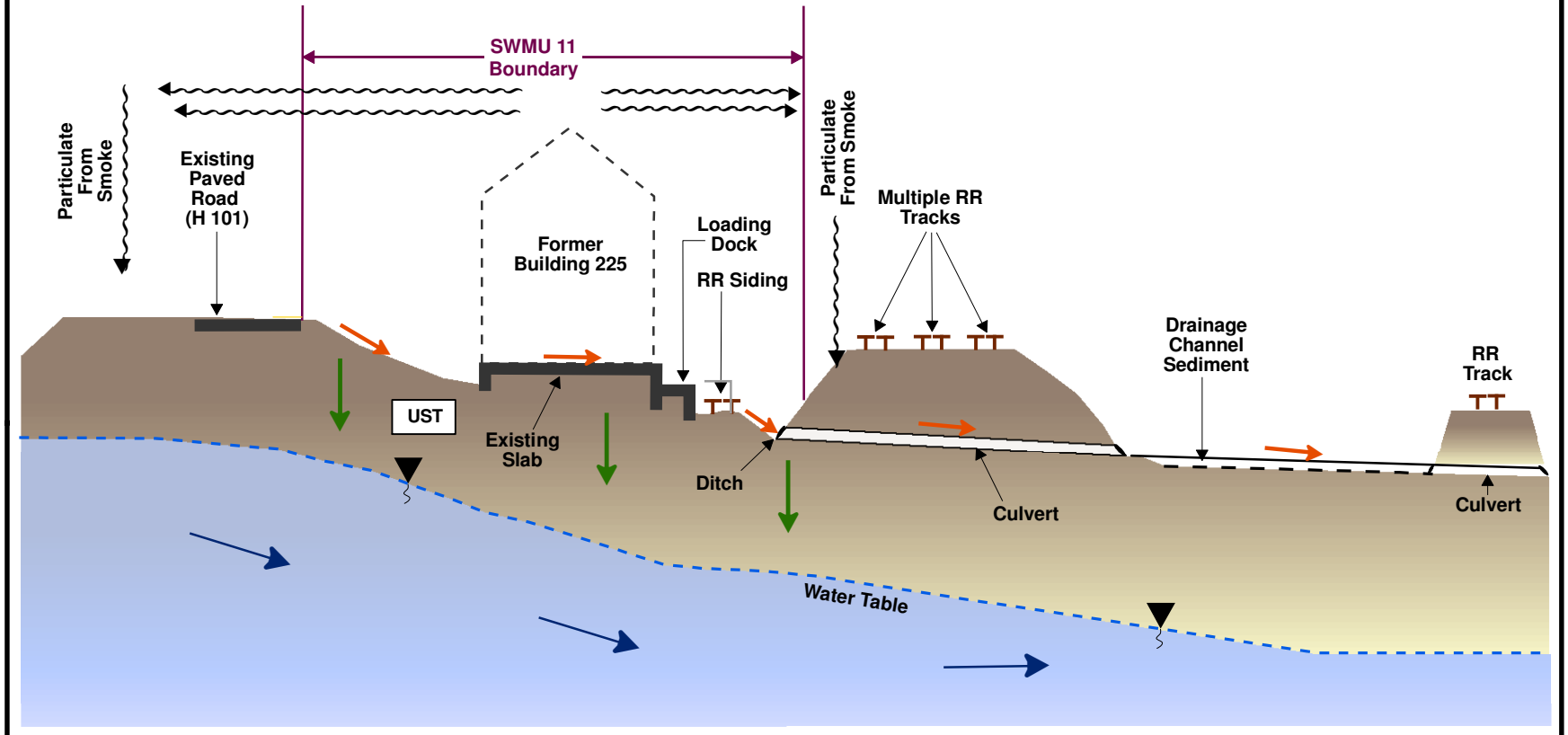
REV	0

P:\GIS\CRANW_NSWC\MAPDOCS\MXD\SWMU11_BLDG2981_AERIAL.MXD 3/16/11 SS

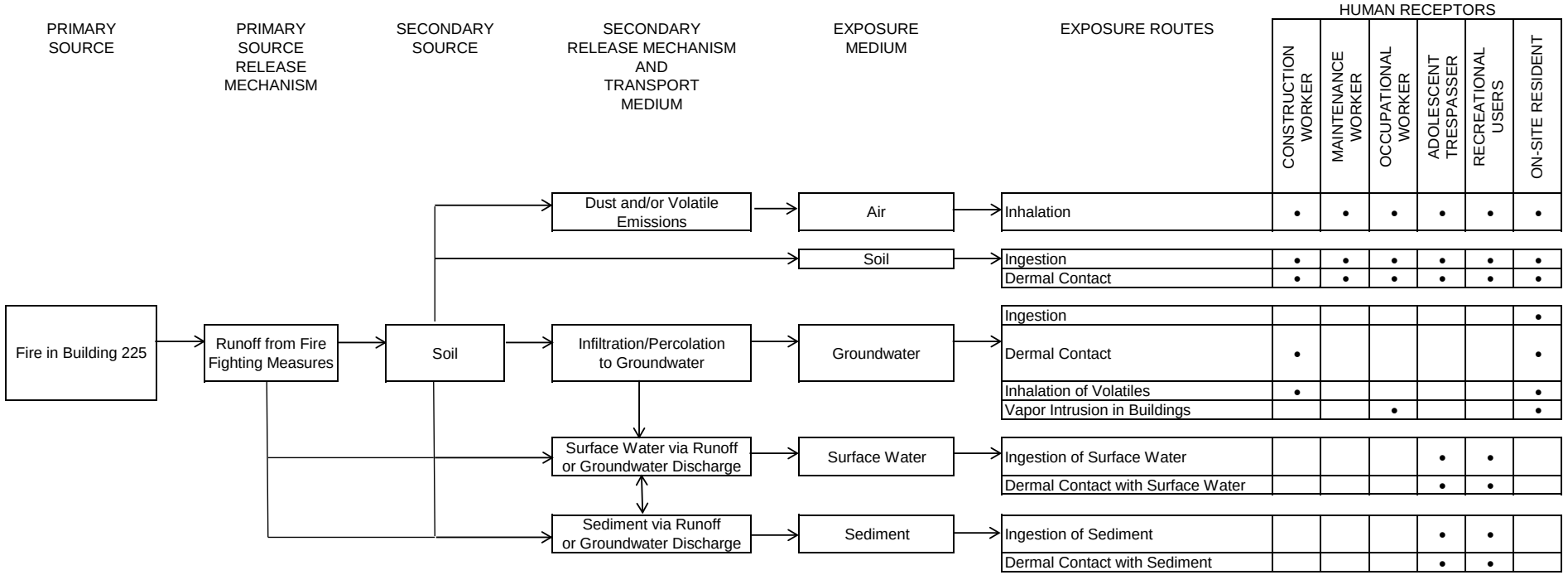
DRAWN BY: S. STROZ — DATE: 11/17/10		IA-6 SITE LAYOUT -	CONTRACT NUMBER: CTO F27E
CHECKED BY: K. LOSEKAMP — DATE: 03/16/11		2005 AERIAL PHOTOGRAPH	APPROVED BY: A. KLIMEK — DATE: 04/14/10
REVISED BY — DATE		BUILDING 2981 CONCRETE TANK	APPROVED BY — DATE
SCALE: AS NOTED	NAVFAC	OFFSITE AREA FOR SWMU 11	FIGURE NO.: 10-4 — REV: 0
		NSA CRANE	
		CRANE, INDIANA	

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMW11_CONCEPTUAL_SITE_MODEL.MXD 11/29/10 SS



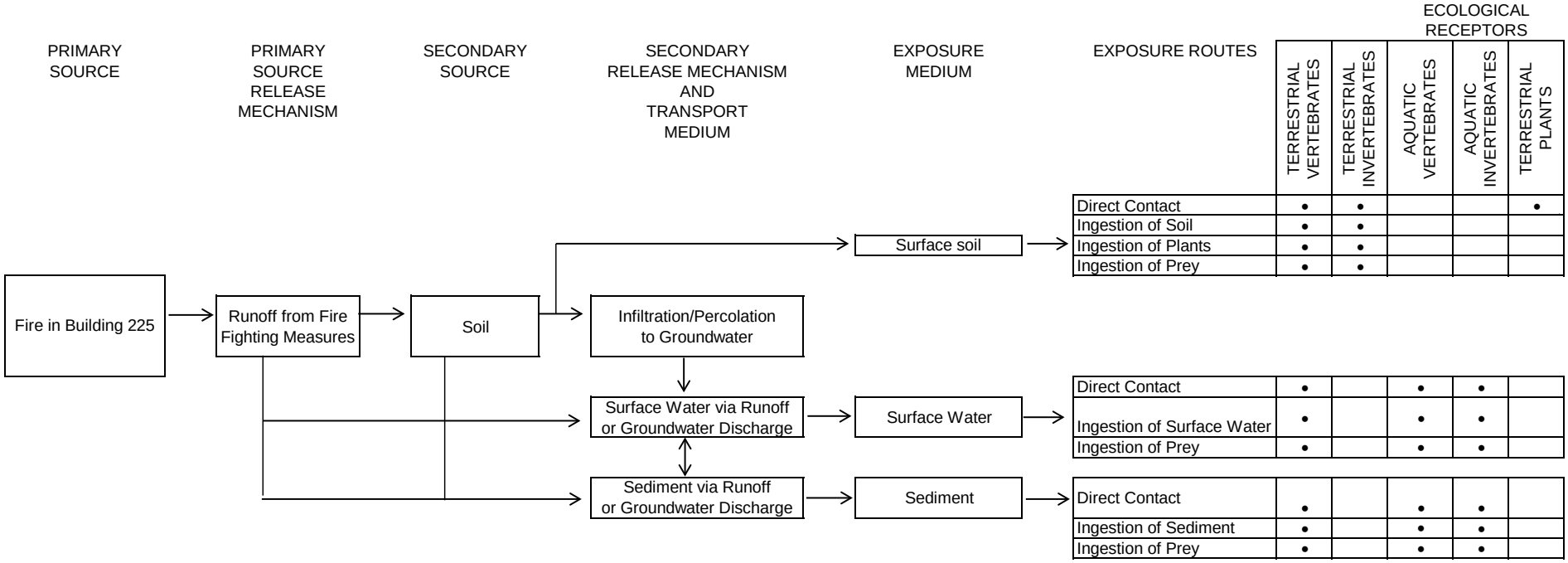
DRAWN BY M. MAGUIRE	DATE 04/09/10	CONCEPTUAL SITE MODEL SCHEMATIC SWMU 11 - OLD STORAGE BUILDING 225 NSA CRANE CRANE, INDIANA	CONTRACT NUMBER ——
CHECKED BY G. TEN EYCK	DATE 11/16/10		APPROVED BY —— DATE ——
REVISED BY S. STROZ	DATE 11/29/10		APPROVED BY —— DATE ——
SCALE NOT TO SCALE			FIGURE NO. FIGURE 10-5 / REV 0

FIGURE 10-6
HUMAN CONCEPTUAL EXPOSURE MODEL DIAGRAM
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE, INDIANA



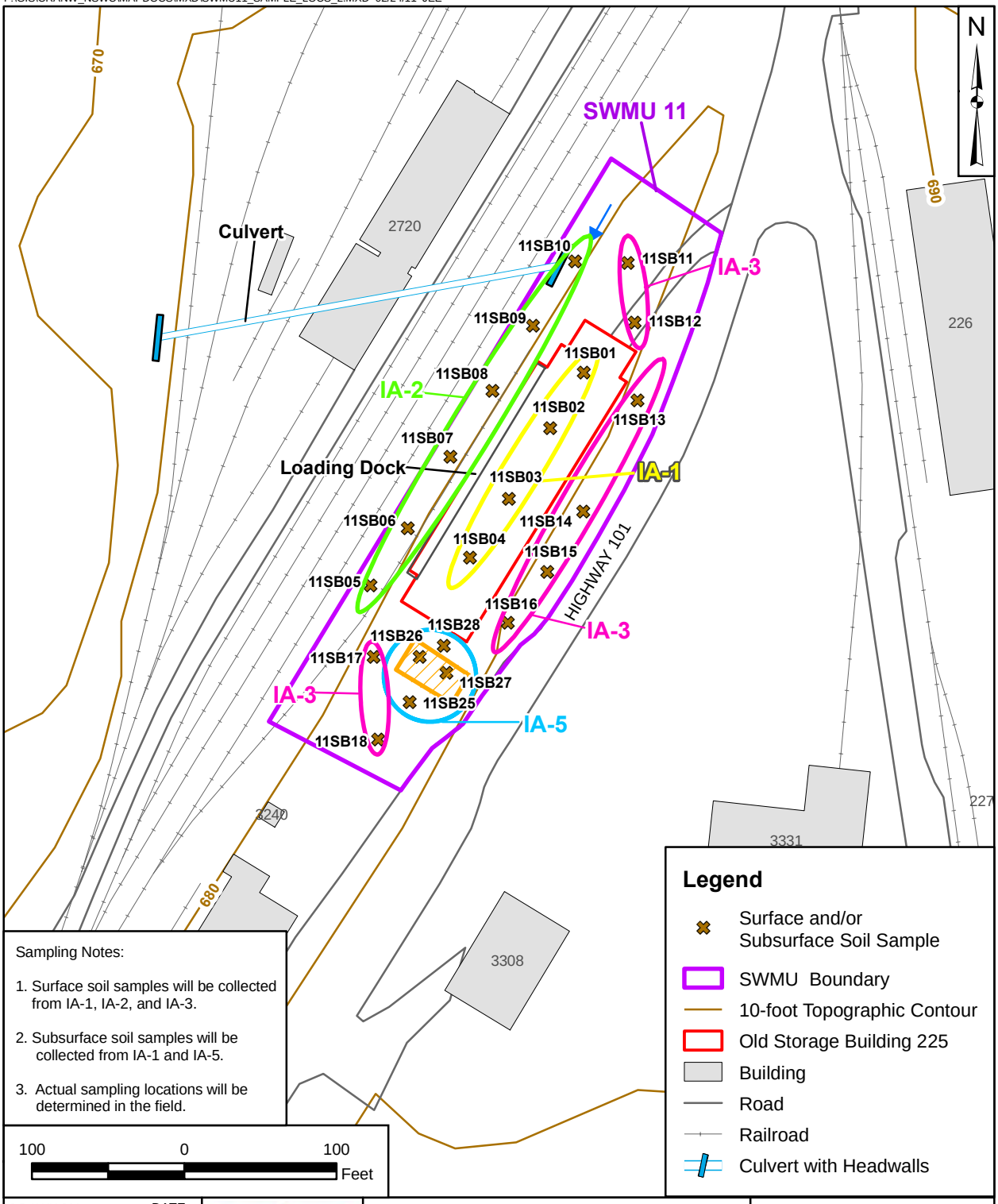
- Indicates receptor for evaluation

FIGURE 10-7
ECOLOGICAL CONCEPTUAL EXPOSURE MODEL DIAGRAM
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE, INDIANA



- Indicates receptor for evaluation

P:\GIS\CRANW_NSWC\MAPDOCS\MXD\SWMU11_SAMPLE_LOCS_2.MXD 02/24/11 JEE



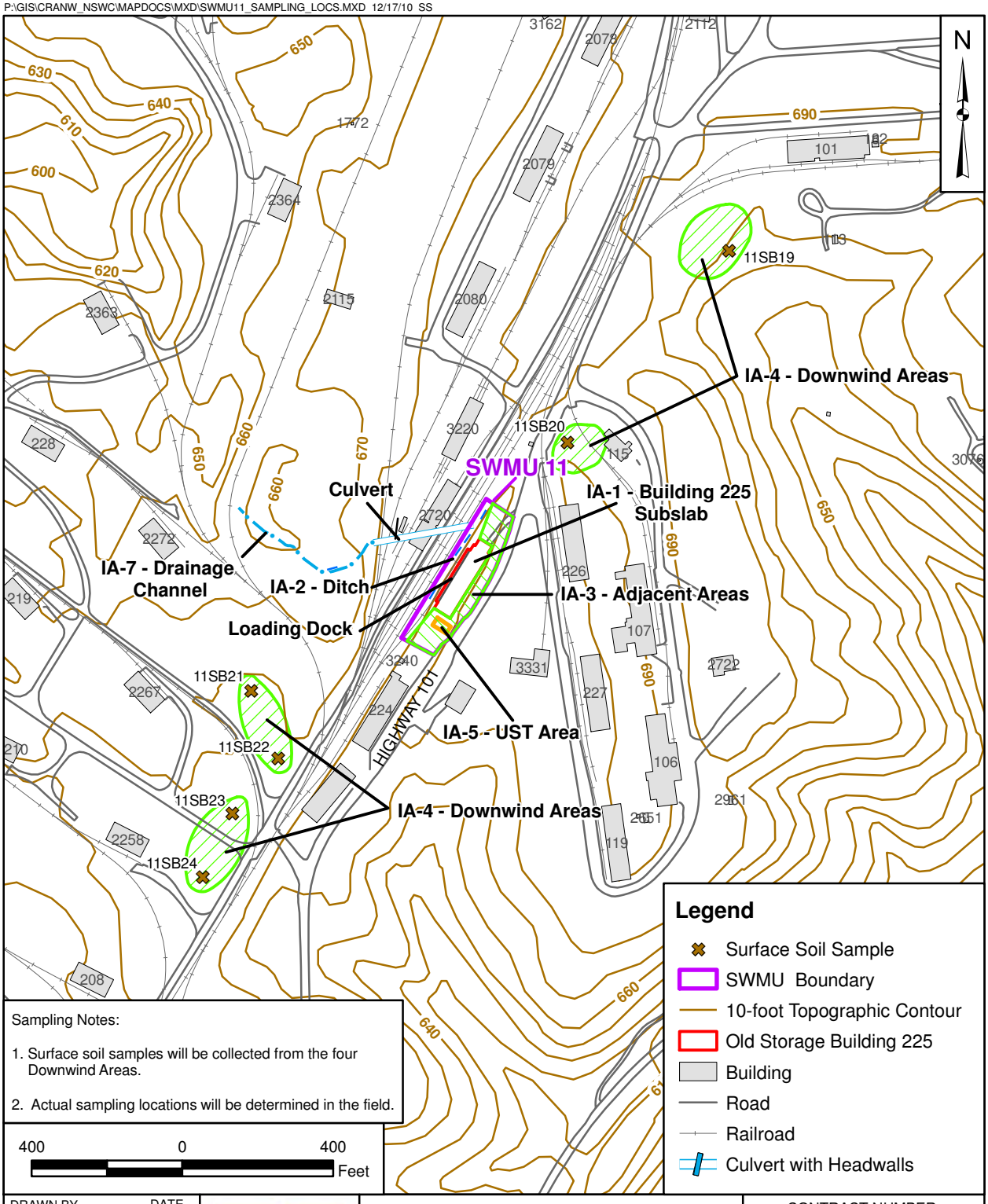
DRAWN BY	S. STROZ
DATE	02/08/10
CHECKED BY	L. FOSTER
DATE	02/24/11
REVISED BY	J. ENGLISH
DATE	02/24/11
SCALE	AS NOTED

NAVFAC

IA-1, IA-2, IA-3, AND IA-5 SOIL SAMPLING LOCATIONS
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	CTO F27E
APPROVED BY	T. KLIMEK
DATE	02/24/11
APPROVED BY	—
DATE	—
FIGURE NO.	17-1
REV	0

P:\GIS\CRANW_NSWC\MAPDOCS\MXD\SWMU11_SAMPLING_LOCS.MXD 12/17/10 SS



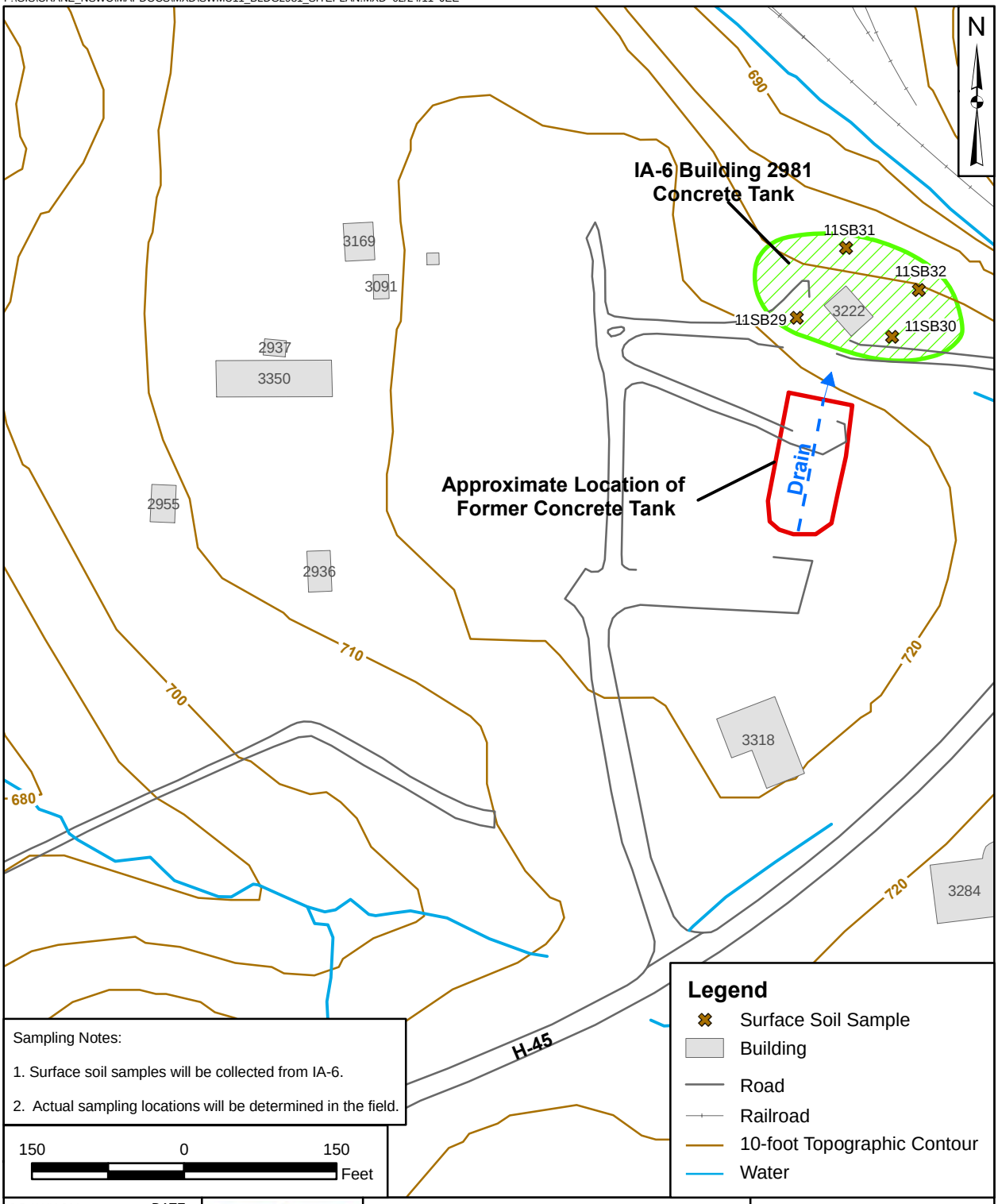
DRAWN BY	DATE
S. STROZ	02/08/10
CHECKED BY	DATE
L. FOSTER	02/24/11
REVISED BY	DATE
J. ENGLISH	02/24/11
SCALE	
AS NOTED	

NAVFAC

IA-4 SOIL SAMPLING LOCATIONS
SWMU 11 - OLD STORAGE BUILDING 225
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO F27E	
APPROVED BY	DATE
A. KLIMEK	02/24/11
APPROVED BY	DATE
—	—
FIGURE NO.	REV
17-2	0

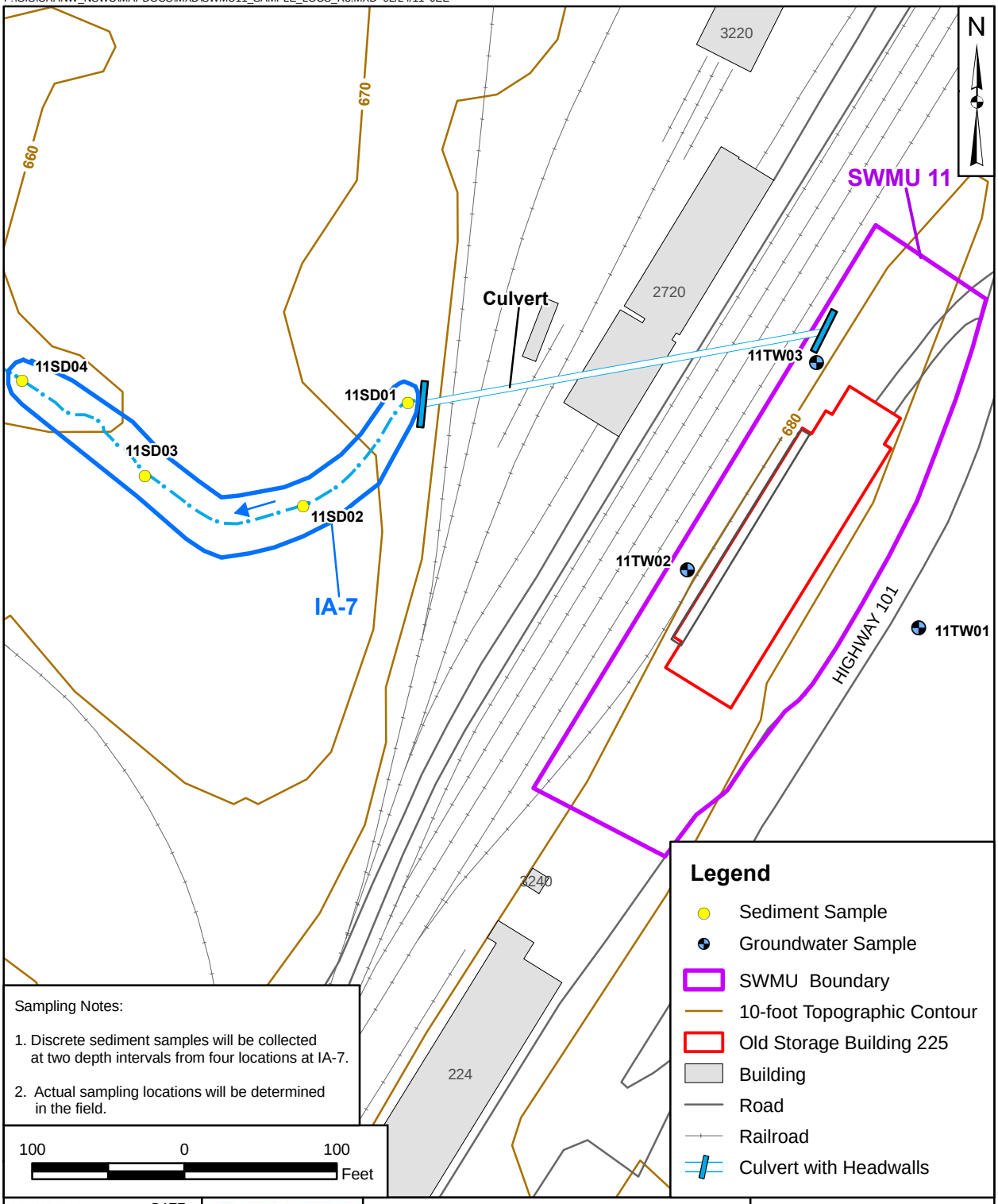
P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU11_BLDG2981_SITEPLAN.MXD 02/24/11 JEE



DRAWN BY: S. STROZ	DATE: 11/17/10	IA-6 SOIL SAMPLING LOCATIONS BUILDING 2981 CONCRETE TANK OFFSITE AREA FOR SWMU 11 NSA CRANE CRANE, INDIANA	CONTRACT NUMBER CTO F27E
CHECKED BY: L. FOSTER	DATE: 02/24/11		APPROVED BY: A. KLIMEK — DATE: 02/24/11
REVISED BY: J. ENGLISH	DATE: 02/24/11		APPROVED BY: — — DATE: —
SCALE: AS NOTED			FIGURE NO.: 17-3 — REV: 0

NAVFAC

P:\GIS\CRANW_NSWC\MAPDOCS\MXD\SWMU11_SAMPLE_LOCS_R3.MXD 02/24/11 JEE



DRAWN BY: S. STROZ	DATE: 12/01/10	IA-7 SEDIMENT AND SWMU 11 GROUNDWATER SAMPLING LOCATIONS SWMU 11 - OLD STORAGE BUILDING 225 NSA CRANE CRANE, INDIANA	CONTRACT NUMBER CTO F27E
CHECKED BY: L. FOSTER	DATE: 02/24/11		APPROVED BY: T. KLIMEK DATE: 02/24/11
REVISED BY: J. ENGLISH	DATE: 02/24/11		APPROVED BY: — DATE: —
SCALE: AS NOTED			FIGURE NO.: 17-4 REV: 0

APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

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STANDARD OPERATING PROCEDURE

SOP-01

GLOBAL POSITIONING SYSTEM

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and update existing Geographic Information System (GIS) data. This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXM or GeoXH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific shape files, data points, background images, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS, Inc.
Attn: John Wright
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 Required GPS Hardware

- Hand-held GPS Unit capable of sub-meter accuracy (i.e. Trimble GeoXM or Trimble GeoXH). This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer.

Optional Accessories:

- External antenna
- Range pole
- Hardware clamp (for mounting Geo to range pole)
- GeoBeacon

- Indelible marker
- Non-metallic pin flags for temporary marking of positions

2.2 Required GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)

- Microsoft ActiveSync version 4.2 or later. Download to personal computer from: http://www.microsoft.com/windowsmobile/en-us/downloads/eulas/eula_activesync45_1033.mspx?ProductID=76

- Trimble Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from: http://www.trimble.com/datatransfer.shtml

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

Start Up:

1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.

2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.

3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.

4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.1 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup.

1) Coordinate System
   a. Tap on the Coordinate System.
   b. Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu. **Note:** It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
   c. Tap on the Units.
   d. Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
   e. Tap Real-time Settings.
   f. Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
   g. The GPS unit is now configured correctly for your specific project.

4.0 ANTENNA CONNECTION

1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
2) At this point the GPS unit is ready to begin collecting data.

5.0 COLLECTING NEW DATA IN THE FIELD

1) From the Main Menu select Data.
2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
3) An auto-generated filename appears and should be edited for your specific project. If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.
4) After entering the file name, tap Create to create the new file.
5) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet).
6) The Choose Feature screen appears.

5.1 Collecting Features

1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**
3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
4) Upon arriving at the specific location, tap on Point_generic as the Feature Name.
5) Tap Create to begin data logging.
6) In the Comment Box enter sample ID or location-specific information.
7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.
9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2.

5.2 Viewing Data or Entering Additional Data Points to the Current File

1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

5.3 Viewing Data or Entering Data Points from an Existing File

1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
4) Highlight the file you want to view and select Map from the Main Menu.
5) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

6.0 NAVIGATION

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

1) From the Main Menu select Map.
2) Using the Select tool, pick the point on the map to where you want to navigate.
3) The location you select will have a box placed around the point.
4) From the Options menu, choose the Set Nav Target (aka set navigation target).
5) The location will now have double blue flags indicating this point is you navigation target.
6) From the Main Menu select Navigation.
7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.

8) Follow the navigation guide until you reach the point you select.
9) Repeat as needed for any map point by going back to Step 1.

7.0 PULLING IN A BACKGROUND FILE

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.
2) Select the project-specific background file from the list of available files.
3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/-> functions at the bottom of the screen.
4) In operating mode, the operator's location will show up on the background file as a floating "x".

8.0 DATA TRANSFER

This section provides instructions on how to transfer stored data on the handheld GPS unit to a personal computer. Prior to transferring data from the GPS unit to a computer, Microsoft ActiveSync and Trimble Data Transfer Utility software must be downloaded to the computer from the links provided in Section 2.2 (Required GPS Software). If a leased computer is utilized in which the operator can not download files, see the Note at the end of Section 8.0.

1) See Attachment A at the end of this SOP for instructions on how to transfer data from the GPS to a personal computer.

Note: If you are unable to properly transfer data from the GPS unit to a personal computer, the unit should be shipped to the project-specific contact listed in Section 1.0 where the data will be transferred and the GPS unit then shipped back to the vendor.

9.0 SHUTTING DOWN

This section provides instruction for properly shutting down the GPS unit.

1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.
2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.

4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

ATTACHMENT A

How to Transfer Trimble GPS Data between Data Collector and PC
original 11/21/06 (5/1/08 update) – John Wright

Remember – Coordinate System, Datum, and Units are critical!!!

Trimble Data Collection Devices:
Standard rental systems include the Trimble ProXR/XRS backpack and the newer handheld GeoXT or GeoXH units. Some of the older backpack system may come with either a RECON "PDA-style" or a TSCe or TSC1 alpha-numeric style data collector.

The software on all of the above units should be Trimble TerraSync (v 2.53 or higher – current version is 3.20) and to the user should basically look and function similar. The newer units and software versions (which should always be requested when renting) include enhancements for data processing, real-time display functions, and other features.

Data Transfer:
Trimble provides a free transfer utility program to aid in the transfer of GIS and field data. The Data Transfer Utility is a standalone program that will run on a standard office PC or laptop.

To connect a field data collector such as a RECON, GeoXM, GeoXT, GeoXH, or ProXH, you must first have Microsoft ActiveSync installed to allow the PC and the data collector to talk to one another. A standard USB cable is also needed to connect the two devices.

A CD or USB drive is provided with the data collector for use in data transfer. If needed, these programs are also available without charge via the web at:

• **Trimble Data Transfer Utility** (v 1.38) program to download the RECON or GeoXH field data to your PC: http://www.trimble.com/datatransfer.shtml

• **ActiveSync** from Microsoft to connect the data collector to the PC. The latest version (v4.5) can be found at: http://www.microsoft.com/windowsmobile/activesync/default.mspx
(see page 2 for data transfer instructions)

To Transfer Data Collected in the Field:

• Install the Data Transfer and ActiveSync software installed on your PC
• Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
• ActiveSync should auto-detect the connection and recognize the data collector
• Make sure the data file desired is CLOSED in TerraSync prior to transfer
• Connect via ActiveSync as a guest (not a partnership)
• Run the Trimble Data Transfer Utility program on your PC
• Select "***GIS Datalogger on Windows CE***" or similar selection
• Hit the green connect icon to the right - the far right area should say "***Connected to ....***" if successful
• Select the "***Receive***" data tab (under device)
• Select "***Data***" from file types on the right
• Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
• Select or browse to a C-drive folder you can put this file for emailing
• When the file appears on the list, hit the "***Transfer All***"
• Go to your Outlook or other email, send a message to: John.Wright@tetratech.com (or GIS department)
• Attach the file(s) you downloaded from your C-drive. For each TerraSync data file created you should have a packet of multiple data files. All need to be sent as a group – make sure you attach all files (the number of files may vary – examples include: ssf, obx, obs, gix, giw, gis, gip, gic, dd, and car)

To Transfer GIS Data from PC to the Field Device (must be converted in Pathfinder Office):

• Obtain GIS file(s) desired from GIS Department and have converted to Trimble extension
• Contact John Wright (John.Wright@tetratech.com) if needed for file conversion and upload support
• The GIS file(s) can be quickly converted if requested and sent back to the field user in the needed "Trimble xxx.imp" extension via email – then quickly downloaded from Outlook to your PC for transfer
• Install the Data Transfer and ActiveSync software installed on your PC
• Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
• ActiveSync should auto-detect the connection and recognize the data collector
• Connect via ActiveSync as a guest (not a partnership)
• Run the Trimble Data Transfer Utility program on your PC
• Select "***GIS Datalogger on Windows CE***" or similar selection
• Hit the green connect icon to the right - the far right area should say "***Connected to ....***" if successful
• Select the "**Send**" data tab (under device)
• Select "**Data**" from file types on the right (you can also send background files)
• Browse to the location of the data on your PC (obtain the file from Pathfinder Office or from the person who converted the data for field use)
• Select the options as appropriate for the name and location of the data file to go on the data collector (usually you can choose main memory or a data storage card)
• When the file(s) appears on the list, hit the "***Transfer All***"
• Run TerraSync on the field device and open the existing data files. Your transferred file should appear (make sure you have selected Main Memory, Default, or Storage Card as appropriate)

STANDARD OPERATING PROCEDURE

SOP-02

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Disposable medical-grade gloves (e.g. latex, nitrile)
Sample log sheets
Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.
Sample labels
Chain-of-custody records
Sealable polyethylene bags
Heavy-duty cooler
Ice

3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label prior to mobilizing for field activities. Additional “generic” labels will also be printed prior to mobilization to be used for field QC and backups.

- Project Number
- Sample Location ID
- Contract Task Order Number (CTO F27E)
- Sample ID
- Matrix

- Preservative
- Analysis to be Performed
- Laboratory Name

3.2 Select the container(s) that are appropriate for a given sample. Select the sample-specific ID label(s), complete date, time, and sampler name, and affix to the sample container(s).

3.3 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.4 Place the sample container in a sealable polyethylene bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** **Location:** **CTO:**	
Sample No:		**Matrix:**
Date:	**Time:**	**Preserve:**
Analysis:		
Sampled by:	**Laboratory**	

STANDARD OPERATING PROCEDURE

SOP-03

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Support Activity (NSA) Crane. The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The NSA Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink
Sample tags
Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Monitoring Samples

All monitoring samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Sample Numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated Solid Waste Management Unit (SWMU) number, sample type, location, and sample depth. For soil samples, the final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample was collected (rounded to the nearest foot). For sediment samples, the final four tracking numbers will identify the depth in units of inches bgs at which the sample was collected. For aqueous samples, where applicable, the sample tracking number will identify whether a sample is filtered and/or the sample round number.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

NN	**AA**	**A and/or N 2 to 5-Characters**	**NN and/or A (Aqueous only)**	**NNNN (Soils and Sediment only)**
SWMU Number	Sample Type	Location Code	Round Identifier and/or Filtered	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha
N = Numeric

SWMU Number (NN):

11 = SWMU 11

Sample Type (AA):

SS = Surface Soil Sample
SB = Subsurface Soil Sample
SD = Sediment Sample
GW = Groundwater Sample

Location:

SB = Soil Boring
SD = Sediment
TW = Temporary Well
MW = Monitoring Well

The sample location code is the well number, soil sample location, or sediment sample location, numbered sequentially by SWMU, beginning with zero one for each sample type (SD01). The location code for each sample is listed on figures and tables in the site-specific work plan.

Note: To keep the sample identification nomenclature to a minimum number of characters and to avoid redundancy, the AA portion of the location code such as "SB" (soil boring) is used to reference the sample location in figures and tables. The AA portion of the location code is replaced with sample type AA in the sample identification and sample tracking number. For example, SB01 (location code) would be SS01 (sample identification) for a surface soil sample collected by DPT.

Existing well numbers are used when unique. New wells will be designated by a SWMU or site number, followed by "MW" for permanent monitoring well, "TW" for temporary well, etc.; a "T" for Tetra Tech NUS for permanent wells; and numbered sequentially, by SWMU, beginning with zero one (TW01).

Note: To keep the sample identification nomenclature to a minimum number of characters and to avoid redundancy, "TW" (temporary well) is used for text, figures, and tables and replaced with "GW" (groundwater) in the sample identification. For example, TW01 would be GW01

Round Identifier and/or Filtered:
This code section will be used for aqueous samples only.

Round Identifier:
A two-digit round identifier will be used to track the number of aqueous samples (GW) taken from a particular aqueous sample location. The first sample collected from a location will be assigned round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells.

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

Depth Interval (NNNN):
This code section will be used for soil and sediment samples only. For soil samples, the final four tracking numbers will identify the depth in units of feet. Surface soil samples will be collected from 0- to 2-feet bgs. Subsurface soil samples will be collected at depths greater than 2-feet bgs. For sediment samples, the final four tracking numbers will identify the depth in units of inches. Sediment samples will be collected from 0- to 6-inches below the sediment/water interface.

The depth code is used to note the depth bgs at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval of the sample depth. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc

3.1.2 Examples of Sample Nomenclature

The location code for the first soil boring at SWMU 11 would be labeled as “11SB01”.

The surface soil sample collected from soil boring 11SB01, at a depth of 0- to 2-feet bgs would be designated as “11SS010002”.

The subsurface soil sample collected from soil boring 11SB01, at a depth of 4- to 6-feet bgs would be designated as “11SB010406”.

The sediment sample collected at location 11SD03, at a depth of 0- to 6-inches bgs would be designated as “11SD030006”.

The first groundwater sample collected from temporary well 11TW02 would be designated as “11GW0201”.

3.2 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the UFP-SAP. They will be designated using a different coding system than the one used for regular field samples.

3.2.1 QC Sample Numbering

The QC code will consist of a four-segment alpha-numeric code that identifies the sample type, QC sample type, the date the sample was collected, and the number of this type of QC sample collected on that date.

NN	AA	AA	MMDDYY	NN	F
SWMU Number	Sample Type	QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

Character Type:

A = Alpha

N = Numeric

M = Month

D = Day

Y = Year

The QC types are identified as:

TB = Trip Blank

RB = Rinsate Blank (Equipment Blank)

FD = Field Duplicate

Note: QC sample tracking numbers for trip blanks will not include sample type (AA), as the same trip blank may be used for samples from more than one type of media.

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be “0000” so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.2.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at SWMU 11 for a surface soil sample collected on collection date (mmddyy) would be designated as 11SSFDMMDDYY01.

The second duplicate of the day taken at SWMU 11 of a subsurface soil sample collected on the same collection date (mmddyy) would be designated as 11SBFDMMDDYY02.

The first rinsate blank associated with surface soil samples collected on the same collection date (mmddyy) would be designated as 11SSRBMMDDYY01.

The second trip blank associated with surface and subsurface soil samples collected on the same collection date (mmddyy) would be designated as 11TBMMDDYY02.

STANDARD OPERATING PROCEDURE
SOP-04

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, labels, and equipment are required.

Writing utensil (preferably black pen with indelible ink)
Site logbook
Field logbook
Sample label
Chain-of-Custody Form
Custody seals
Equipment calibration log
Soil and Sediment Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- Names and responsibilities of on-site project personnel including subcontractor personnel
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Sampling activities and sample log sheet references
- Daily on-site activities performed each day
- Description of subcontractor activities
- Sample pickup information including chain-of-custody form numbers, air bill numbers, carriers, times, and dates
- Descriptions of borehole activities and operations
- Descriptions of monitoring well installation activities and operations, if monitoring wells are deemed necessary
- Description of photographs including date, time, photographer, picture number, location, and compass direction of each photograph
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date
- Name of the person to whom the logbook is assigned

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the Tetra Tech Field Operations Leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the upper right hand corner section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page __ of __" on each COC. The original (top) signed copy of the COC will be placed inside a sealable polyethylene bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Tetra Tech Project Manager (PM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing

environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the Tetra Tech PM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.7 Sample Log Sheets

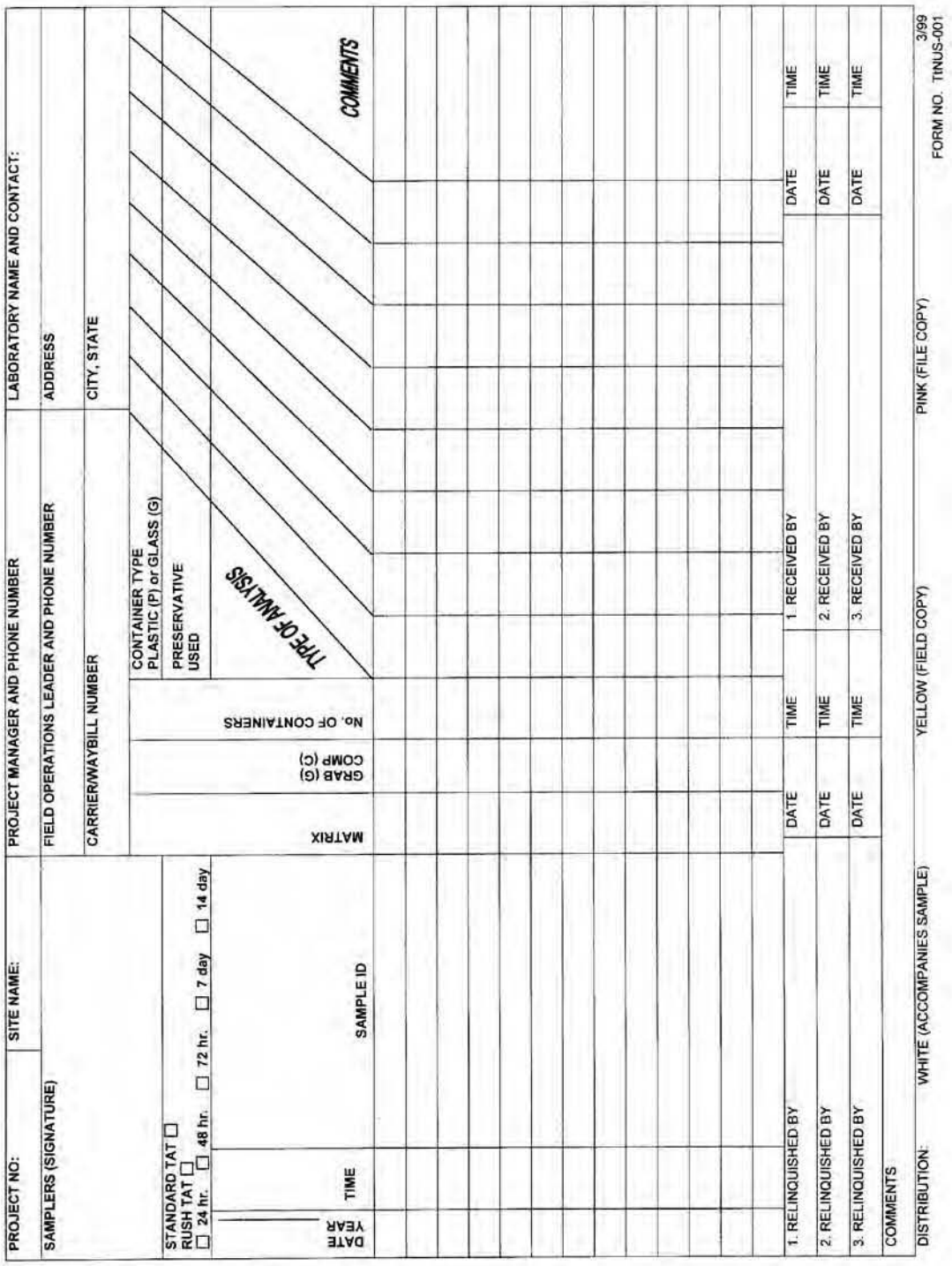
The Soil and Sediment Sample Log Sheets are used to document the sampling of soil and sediment (see SOPs -07, -08, and -09).

4.0 ATTACHMENTS

1. Chain-of-Custody Record
2. Equipment Calibration Log
3. Soil and Sediment Sample Log

ATTACHMENT 1

CHAIN-OF-CUSTODY RECORD



ATTACHMENT 2
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ____________________ INSTRUMENT NAME/MODEL: ____________________

SITE NAME: ____________________ MANUFACTURER: ____________________

PROJECT No.: ____________________ SERIAL NUMBER: ____________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT 3

SOIL AND SEDIMENT SAMPLE LOG SHEET

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE

SOP-05

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling soil, sediment, and aqueous samples.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels

Custody seals

Chain-of-custody (COC) form(s)

Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.

Packaging material: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4 degrees Celcius (°C). This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a polyethylene bag.

3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Line the cooler with large plastic bag, and line the bottom of the cooler with a layer of bubble wrap. Place the sample containers into the shipping cooler in an upright position (containers will be upright, with the exception of any 40-milliliter vials). Continue filling the cooler with ice until the cooler is nearly full and the movement of the sample containers is limited.

3.7 Wrap the large plastic bag closed and secure with tape.

3.8 Place the original (top) signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.

3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.

3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

STANDARD OPERATING PROCEDURE

SOP-06

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Non-latex rubber or plastic gloves
Cotton gloves
Field logbook
Potable water
Deionized water
Isoproponal (optional)
LiquiNox detergent
Brushes, spray bottles, paper towels, etc.
Container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the LiquiNox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-10 and this UFP-SAP.

STANDARD OPERATING PROCEDURE

SOP-07

SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using hand augering techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Stainless Steel Auger Buckets
Stainless Steel Extension Rods
Cross Handle
Required decontamination materials
Bentonite pellets
Sealable polyethylene bags
Sample labels
Shipping containers (containing ice)
Disposable plastic trowels or stainless steel trowels
Stainless steel mixing bowls
Sample containers: Sample containers are certified clean by the laboratory supplying the containers.
Soil Sample Log Forms
Daily Activity Logs
Chain-of-Custody Form

3.0 SOIL SAMPLING USING A HAND AUGER

Hand augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2" long and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), a cross handle.

3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence or rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, letter, etc.)

3.3. Turn hand auger sampler into the ground to a depth of 2 feet. The 0- to 2-feet depth soil interval is considered to be the surface soil.

3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.

3.4 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a sealable polyethylene bag. Note in a field notebook or on a standardized data sheet any changes in the color, texture or odor of the soil.

3.5 Thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker. Collection of the sample material for VOC analysis will be completed prior to homogenization, if VOC analysis is required.

3.6 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.

3.7 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water.

3.8 Decontaminate all soil sampling equipment in accordance with SOP-06 before collecting the next sample.

3.9 Soil samples shipped to a fixed-base laboratory for analysis will be in sample containers supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The

samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-05.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date: / Time: / Method: / Monitor Reading (ppm):	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE

SOP-08

SOIL SAMPLE LOGGING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil samples.

2.0 FIELD FORMS AND EQUIPMENT

Knife
Ruler (marked in tenths and hundredths of feet)
Boring Log: An example of this form is attached.
Writing utensil (preferably black pen with indelible ink)

3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all activities and assuring that each soil sample is properly and completely logged.

4.0 PROCEDURES FOR SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

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

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

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

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

4.2 Color

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

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

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

4.3 Relative Density and Consistency

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

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

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

CONSISTENCY FOR COHESIVE SOILS

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

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

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

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

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

Examples:

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

4.5 Moisture

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

4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	CH	inorganic clays of high plasticity, fat clays.
	OH	organic clays of medium to high plasticity, organic silts.
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity.
	OL	organic silts and organic silty clays of low plasticity.
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils.
Sand	SW	well graded sands, gravelly sands, little or no fines.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
	SP	poorly graded sands, gravelly sands, little or no fines.
	SM	silty sands, sand-silt mixtures.
	SC	clayey sands, sand-clay mixtures.

4.7 Summary of Soil Classification

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

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

5.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

ATTACHMENT 1

FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System

Coarse Grained Soils (more than half of soil > No. 200 sieve)	Gravels (More than half of coarse fraction > no. 4 sieve size)		GW	Well graded gravels or gravel-sand mixtures, little or no fines
			GP	Poorly graded gravels or gravel-sand mixtures, little or no fines
			GM	Sandy gravels, gravel-sand-silt mixtures
			GC	Clayey gravels, gravel-sand-silt mixtures
	Sands (More than half of coarse fraction < no. 4 sieve size)		SW	Well graded sands or gravelly sands, little or no fines
			SP	Poorly graded sands or gravelly sands, little or no fines
			SM	Silty sands, sand-silt mixtures
			SC	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity
Fine Grained Soils (more than half of soil < No. 200 sieve)	Silts and Clays LL = < 50		ML	Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity
			CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays
			OL	Organic silts and organic silty clays of low plasticity
	Silts and Clays LL = > 50		MH	Inorganic silts, micaceous or diatomaceous fine sand or silty soils, elastic silts
			CH	Inorganic silts of high plasticity, fat clays
			OH	Organic clays of high plasticity, organic silty clays, organic silts
Highly Organic Soils			Pt	Peat and other highly organic soils

Grain Size Chart

Classification	Range of Grain Sizes	
	U.S. Standard Sieve Size	Grain Size In Millimeters
Boulders	Above 12"	Above 305
Cobbles	12" to 3"	305 to 76.2
Gravel	3" to No. 4	76.2 to 7.76
coarse	3" to 3/4"	76.2 to 4.76
fine	3/4"to No.4	19.1 to 4.76
Sand	No. 4 to No. 200	4.76 to 0.074
coarse	No.4 to No. 10	4.76 to 2.00
medium	No. 10 to No. 40	2.00 to 0.420
fine	No. 40 to No. 200	0.420 to 0.074
Silt and Clay	Below No. 200	Below 0.074

Relative Density (SPT)

SANDS AND GRAVELS	BLOWS/FOOT
VERY LOOSE	0 – 4
LOOSE	4 – 10
MEDIUM DENSE	10 – 30
DENSE	32 – 50
VERY DENSE	OVER 50

Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 – 2
SOFT	2 – 4
MEDIUM STIFF	4 – 8
STIFF	8 – 16
VERY STIFF	16 – 22
HARD	OVER 3[illegible]

ATTACHMENT 2

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

STANDARD OPERATING PROCEDURE

SOP-09

SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for sediment sampling in streams and other waterways.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

Sediment Sample Log Forms: A copy of this form is attached at the end of this SOP.
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Bound field logbook
Disposable plastic trowels
2-inch diameter, 18-inch long clean plastic sleeves
Survey stakes and flagging: Used to mark sampling locations after completion of sampling.
Labeled sample containers: See SOP-02 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
Sealable polyethylene bags
Shipping containers (containing ice)
Disposable medical-grade gloves (e.g., latex, nitrile)
Chain-of-Custody Form

3.0 SEDIMENT SAMPLE LOCATION SELECTION

In general, sediments composed of fine-grained materials with greater surface area available for adsorption are more desirable for sample selection. The fined-grained materials may act as a sink or reservoir for adsorbing heavy metals and organic contaminants even if surface runoff concentrations are below detection limits. Therefore, it is important to locate the specific sampling points where the sediment

has the greatest percentage of fine particles. The sampling personnel will determine specific sampling locations with these goals in mind.

4.0 SEDIMENT SAMPLING PROCEDURES

4.1 The sampler will wear clean, disposable medical-grade gloves.

- In areas of standing or flowing water, the sampler will push a 2-inch diameter, 18-inch long clean plastic sleeve down into the sediment to the desired depth and cover the open end of the sleeve with a gloved hand to capture the sample in the lower end of the sleeve. Remove the sleeve from the substrate and place the lower end containing the sediment sample inside the sample collection bag. Remove the sampler's gloved-hand from the end of the sleeve allowing the sediment to empty into the bag. Prior to the transfer of sample material from the collection bag to the labeled sample jar, all sediment samples will be thoroughly homogenized.

- In areas where standing or flowing water are not present, the sampler will clear vegetative matter or debris, if present, from the sample location using a disposable sampling trowel or spoon. Use the trowel to dig up and homogenize the sediment in an 18-inch-diameter circular area that is 6 inches deep. Stir the sediment within the circular area; do not move the sediment outside the circle. Also, do not dig or stir sediment that is deeper than 6 inches below the ground surface, until the next depth interval is sampled. Use the same trowel to scoop the homogenized sediment into the requisite labeled sample container(s).

4.2 Record the sample time (using military time) on the Sediment Sample Log Form and sample container labels. Record all other information required on the labels as specified by SOP-02.

4.3 Place the labeled sample container into a sealable polyethylene bag and then place the bag holding the sample container into a cooler containing ice.

4.4 Record date, sampling site, site conditions, location map, and other information (e.g., presence and flow rate of water in channel) on the Sediment Collection Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP-04.

4.5 Using an indelible marker, write the sample identification on a survey stake, and drive the stake into the ground at the sample location. Tack a piece of brightly colored flagging to the stake. In

addition, tie a piece of flagging to an overhead tree branch or other eye-level object to improve the ability to relocate the sampling site in the future.

5.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings				
(Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
SOP-10

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at Naval Support Activity (NSA) Crane. The following types of IDW will be generated during this investigation:

- Soil sampling residues
- Monitoring well development and well purge waters
- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)
Hand augers, plastic or stainless steel trowels
Bucket (with collected development/purge water)
Decontamination equipment
Field logbook
Writing utensil (preferably black pen with indelible ink)
Plastic sheeting and/or tarps
55-gallon drums with sealable lids
IDW labels for drums
Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and containerized in a central location at NSA Crane for proper disposal.

3.2 Solid Wastes

Solid wastes that may be generated during site activities include soil and sediment sampling residues. Excess soil core/sampling materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water. Excess sediment sampling materials will be returned to the point of collection. The disposition of this materials will be carried out in a manner such as not to contribute further environmental degradation or pose a threat to public health or safety.

3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSA Crane.

STANDARD OPERATING PROCEDURE
SOP-11

SUBSURFACE SOIL AND GROUNDWATER SAMPLING USING DIRECT-PUSH TECHNOLOGY

1.0 PURPOSE

This procedure provides general guidance and reference information on direct-push technology (DPT). DPT is designed to collect soil and groundwater samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, ability to sample in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells.

The methods described herein are specific for soil, groundwater, and soil gas samples at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 (Water Well Drillers) of Chapter 310 of the Indiana Administrative Code (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction.

Field Geologist - The Tetra Tech Field Geologist supervises and documents DPT activity performed by the driller, and insures that the soil and groundwater samples collected accurately representative the desired media and sample interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

All Field Personal – All field personal including, the drilling contractor personnel and other field staff, must have all of the health and safety training required to perform the work, as specified in the Health and

Safety Plan (HASP). All field personnel shall be aware of the potential presence of underground utilities. Proper utility clearance must be obtained by the Tetra Tech Project Manager (PM) prior to any DPT activity.

If the potential of Unexploded Ordnance (UXO) is present at the site, UXO clearance to six feet will be required for subsurface samples. Every two feet the drill rig shall be withdrawn and a downhole magnetometer used to check for UXO. If the presence of any UXO is suspected, the drilling location will be shifted two feet and drilling resumed. A trained UXO technician will be part of the Tetra Tech field team.

3.0 REQUIRED EQUIPMENT/ITEMS

The list of equipment and items required for DPT sampling includes, but is not limited to:

Health and safety equipment as required by the HASP and the Site Safety Officer.

DPT Rig is supplied by the drilling subcontractor and may include the following:

- 4-foot x 2-inch diameter macrocore sampler
- Probe sampling adapters
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Stainless steel screen point groundwater sampler (Geoprobe®, HydroPunch™, or equivalent)

55-gallon drums to containerize IDW (supplied by the drilling subcontractor).

Required decontamination materials including distilled water, deionized water, paper towels, and stainless steel clamps.

Writing utensil (preferably black ink), non-latex gloves, bound field logbook, chain-of-custody forms, sample labels, boring log, sample logsheets, engineer's tape (or equivalent), and stainless-steel spoon or trowel.

Required sample containers with appropriate preservative: All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory. Additional sampling equipment as needed, such as photo-ionization detector (PID), flame-ionization detector (FID), Ziplock bags, calculator, wristwatch, and timer, and cooler (containing ice), peristaltic pump, inertial lift pump,

silicon tubing, polyethylene (PE) tubing, water quality meter with a flow through cell, LaMotte 2020 Turbidity Meter (or equivalent), water level indicator, 0.45 micron filter cartridge, trip blanks, and bucket to collect development/purge water.

4.0 GLOSSARY

Direct Push Technologies (DPT) -DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools.

Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photoionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

5.0 DPT SOIL SAMPLING PROCEDURES

General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

DPT Sampling Methodologies

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Tetra Tech PM in accordance with the project-specific plan.

Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

The sampler is advanced continuously in 4-foot intervals, or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit (or equivalent) firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.
- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife. Then remove the strip of acetate from the trough to gain access to the collected soils. **Do not** attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID (according to manufacturer's Standard Operating Procedure [SOP]) and observe/examine the sample. If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organic compounds (VOCs) analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van, truck, or track mounted rig cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling. Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.1 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited to the following:

- 2-foot x 1 -inch diameter mill-slotted (0.005 to 0.02-inch) well point Connecting rods
- Roto-hammer with 1.5-inch bit Mechanical jack
- 1/4-inch outside diameter (OD) PE tubing
- 3/8-inch OD PE tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.2 DPT Sampling Methodologies

Once the water table has been encountered, a stainless steel screen point groundwater sampler (Geoprobe®, HydroPunch™, or equivalent) will be driven by DPT to collect a groundwater sample at the water table.

- Field screening of VOC vapors in the borehole shall be done using a FID or PID.
- The screen point will be allowed to equilibrate for at least 15 minutes.
- Once equilibration occurs, measurement of the static water level will be taken. This initial water level measurement will be used to assess the amount of water present in the screen point and to determine the amount of silt and/or sand infiltration.
- Development of the screen point will be accomplished using a peristaltic pump.

- Insert the intake end of a length of dedicated PE tubing to the bottom of the screen point and attach a length of silicon tubing (approximately 1 foot) to the discharge end of the PE tubing. The silicon tubing will be threaded around the rotor of the pump and out of the pump.
- The PE tubing will be lifted and lowered slightly while the pump is operating. The maximum pump rate will be approximately 2 liters per minute during development; however the yield of the formation will dictate the pumping rate.
- Measurement of pH, specific conductance, turbidity, dissolved oxygen, eH, salinity, and temperature shall be recorded every 5 to 10 minutes during the development process using a water quality meter and flow-through cell, with the exception of turbidity. Turbidity measurements will be taken with a Lamotte Turbidity Meter from water collected from a T-connector with a valve inserted in the pump discharge tubing prior to entering the flow-through cell.
- After removal of sediment from the bottom of the screen point, the screen point will be pumped until discharge water is visibly clear and no further sediments are being generated.
- Stabilization is achieved after two consecutive readings taken at 5 to 10 minutes intervals of the following field parameters has occurred:

    - pH +/- 0.1 standard units
    - Turbidity +/- 10% for values greater than 1 NTU
    - Specific conductance +/- 3%
    - Temperature +/- 3%
    - eH +/- 10 millivolts
    - Dissolved oxygen +/- 10%

- Samples will be collected using the peristaltic pump set at 0.2 liters per minute or lower, depending on the yield of the formation. Samples (with the exception of samples to be analyzed for VOCs) will be collected directly from the pump discharge. The pump shall continuously operate between development, purging, and sampling.

- If the above condition(s) have not been met after three well volumes have been removed, this will be recorded on the field sample form and the groundwater sample will be collected.

- Record the sample date and time (using military time) on a Tetra Tech Groundwater Sample Log Sheet and on a chain-of-custody form.

- Record the sample date and time (using military time) on an adhesive-backed sample label and affix the sample label securely to the sample container.

- With the pump continuing to run, allow the pump discharge to flow gently down the inside of the sample container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled.

- Cap each container immediately after filling.

- Place the sample container into a ziplock bag and then into a cooler containing ice.

- Repeat the last four steps for each sample container collected.

- The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Tetra Tech Groundwater Sample Log Sheet.

- All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. The hierarchy of filling sample containers is as follows:

    - VOCs
    - Explosives
    - Total metals
    - Dissolved metals
    - Perchlorates

- This hierarchy takes into consideration the volatilization sensitivity of groundwater samples. The only deviation from this order will be the collection of samples for VOC analysis. The collection of VOCs will be the final parameter collected due to the fact that VOCs will not be collected using the peristaltic pump.

- A single-use, disposable, in-line 0.45-micron filter cartridge shall be used to collect dissolved metals samples. Attach the filter cartridge to the discharge end of the pump tubing. Prior to filling containers with filtered sample, rinse the filter cartridge with approximately 100 milliliters (mL) of water from the boring to be sampled. Direct the discharge from the filter cartridge into the sample bottle and collect the filtered sample. The laboratory will supply all sample containers, and the laboratory will pre-preserve sample containers where appropriate.

- Once all of the sample containers have been filled (with the exception of those sample containers for VOC analysis), the pump shall be shut off. Record the sample date and time (in military time) on an adhesive-backed sample label and affix the sample label securely to the sample container. Sample containers for VOCs will be filled by crimping the discharge end of the PE tubing (immediately after shutting off the pump). Remove the inlet end of the PE tubing from the well, suspend the inlet tubing above the VOC sample container (pre-preserved 40 mL vial), and slowly allow water to fill each VOC sample container by gravity flow. The discharge of sample from the PE tubing shall be accomplished in a manner that allows the water to gently flow down the inside of the sample container. Sample containers for VOCs must be completely filled so that no headspace exists in the container. Record the end time for sampling on a Tetra Tech Groundwater Sample Log Sheet.

- Once collection of samples is complete, the driller shall remove the screen point and the screen point will be decontaminated in accordance with the procedures outlined in the decontamination SOP.

- If needed, continuous soil and groundwater sampling using DPT below the water table shall be done in accordance with those procedures outlined above.

- After the groundwater samples have been collected, the driller shall retract the screen point sampler from the borehole and proceed to abandon the borehole with a grout pump using a cement bentonite grout mix from the bottom up to the ground surface.

- When advancing a boring using DPT and refusal is encountered, the boring shall be deemed complete, drilling shall cease, and the borehole shall be abandoned with a grout pump using a cement bentonite grout mixture.

STANDARD OPERATING PROCEDURE
SOP-12

MONITORING WELL INSTALLATION

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper design and installation of ground water monitoring wells. The methods described herein are specific for monitoring well construction at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command, (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 Water Well Drillers of Chapter 310 of the Indiana Annotated Codes (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The drilling contractor personnel must have all of the health and safety training required to perform the work, as specified in the Health and Safety Plan (HASP).

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that the screen interval for each monitoring well is properly placed to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the Site Safety Officer.

Well drilling and installation equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineer’s rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink

marker for marking monitoring wells, sample jars, well installation forms, boring logs, soil sample log forms, chain-of-custody records, sample coolers with ice, and a field notebook).

4.0 WELL DESIGN AND CONSTRUCTION

New wells will be installed only with Navy concurrence. Based on observations and information gathered during the drilling of each hole, the total depth of the hole and the placement of the well screen will be determined at the discretion of the field geologist or the Field Operations Leader (FOL). The decision concerning the monitored interval and well depth will be based on the following (and possibly other) information collected while the well bore is being drilled and logged:

- The specific depths where poorly-cemented sandstone units, fractured rock, or other permeable rock zones are encountered,

- The specific depths where above-average rates of ground water were brought to the surface during drilling,

- The specific depth interval where contaminants (i.e., VOCs), if any, are encountered during drilling.

All of this information will be recorded on the borehole log as the hole is drilled.

Overburden drilling followed by diamond coring (if necessary) will be performed at borehole locations. For each well, the coring will proceed to the final depth of the borehole. Once the coring has been completed and the core has been logged, then the hole will be reamed out with a 6 to 8-inch diameter air rotary bit. The air rotary equipment must have a filter on the compressed air line going to the borehole to prevent oil and other organics from being introduced. Once the hole has been completed to depth, the boring shall be cleaned out using the compressed air of the rig. Note: all drilling equipment must be decontaminated before it is placed in a borehole.

A 6-inch diameter steel isolation casing will be installed and pressure grouted in the deep wells to seal the upper groundwater from deep groundwater. The grout will be allowed to cure for a minimum of 24 hours before resuming coring and reaming to the total depth of the borehole.

All monitoring wells will be constructed of schedule-40, flush-joint threaded, 2-inch inside diameter (ID) polyvinyl chloride (PVC) riser pipe and flush joint threaded, factory slotted well screen with a threaded end cap. The well screens will be factory slotted to 0.020-inch size. Each section of well casing and screen shall be National Sanitation Foundation (NSF) approved. Well screens will be 10-feet long, but

may be longer or shorter based on the subsurface conditions encountered. A PVC cap will be placed on the bottom and will also be flush-threaded. Thermoplastic pipe shall comply with ASTM F-480 (1981). Other means of joining casings using glue, gaskets, pop rivets or screws are not allowed. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material.

Monitoring wells will be installed immediately upon completion of drilling. A well screen section with bottom cap and the proper amount of riser pipe will be assembled and lowered down the borehole. Spacers may be used to ensure that the casing and screen are centered and are aligned straight. Clean silica sand pack will be installed through the borehole. The sand pack will be extended from 0.5 feet below the well screen to 2.0 feet above the top of the well screen. Clean silica sand of U.S. Standard Sieve Size No. 20 to 40 will be used.

A minimum 2-foot thick bentonite pellet seal will be installed above the filter pack and allowed to hydrate as determined by field geologist before grout is added above the seal. Only 100-percent, certified pure, sodium bentonite will be used for well construction. The depths of backfill materials will be constantly monitored during well installation using a weighted stainless steel or fiberglass tape measure.

The remaining annulus above the hydrated bentonite seal will be backfilled to the surface using a tremie pipe, with a 20:1 cement/bentonite grout. A maximum of 10 gallons of water per 94-pound bag of Type-1 cement will be used. The grout mixture should be blended in an above-ground rigid container or mixer to produce a thick lump-free mixture.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets extending to the surface. The grout effectively seals the well and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom of the hole upward, to prevent bridging, and to provide a better seal. However, in shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

When the well is completed and grouted to the surface, a protective steel surface casing is placed over the top of the well. The finished well casing shall extend at least 2 ft above the ground level. This casing will have a cap that will be locked to prevent vandalism. A vent hole shall be provided in the cap to allow venting of gases and maintain atmospheric pressure as water levels rise or fall in the well. The protective casing has a larger diameter than the riser pipe and is set into the wet cement grout over the riser upon completion. In addition, one hole is drilled just above the cement collar through the protective casing

which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

Four barrier posts shall be placed at the corner of a 3 foot by 3 foot by 6 inch thick concrete pad located at the ground surface.

5.0 DOCUMENTATION OF FIELD ACTIVITIES

A critical part of monitoring well installation is recording of significant details and events in the site logbook, on field forms, and a field logbook.

All installed wells must be registered with the NSA Crane Environmental Protection Department. The following information must be supplied to NSA Crane for each well as soon as this information is known:

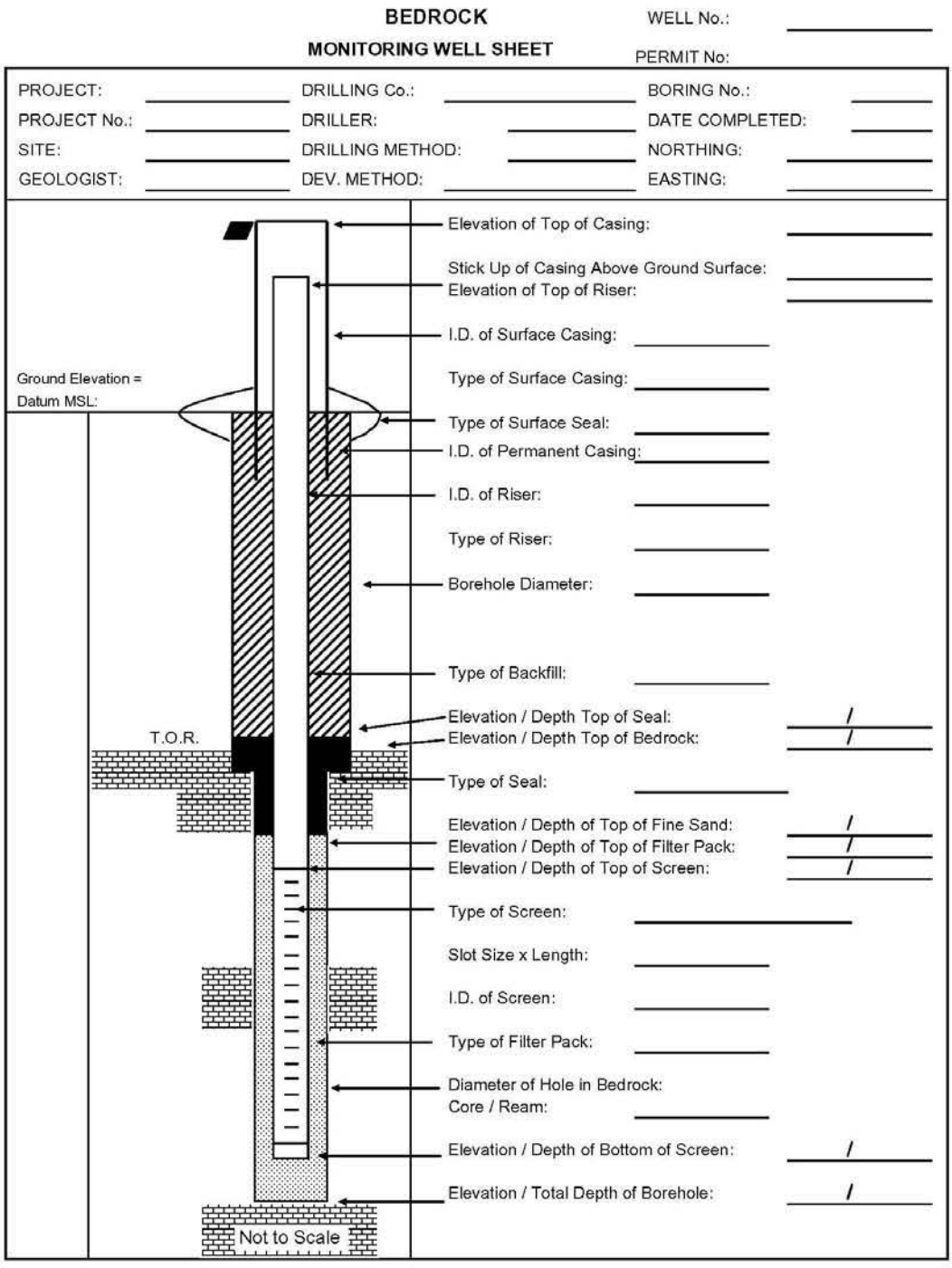
- Tag number
- Installation Name (i.e., NSA Crane)
- Contract Task Order number (CTO F273)
- TtNUS project number
- Well identification number
- Date installed
- Installer (i.e., TtNUS)
- Total well depth
- Screened interval
- Elevation (Top of casing)
- Northing coordinate (ft)
- Easting coordinate (ft)
- Survey coordinate reference system
- Information point of contact.

6.0 ATTACHMENTS

1. Bedrock Monitoring Well Sheet
2. Overburden Monitoring Well Sheet

ATTACHMENT 1

BEDROCK MONITORING WELL SHEET



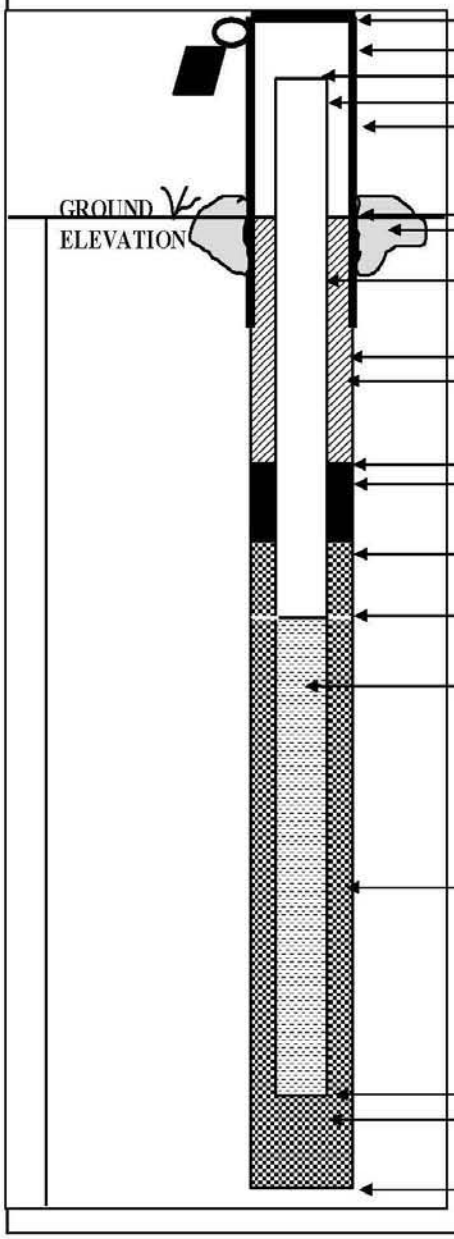
ATTACHMENT 2

OVERBURDEN MONITORING WELL SHEET

BORING NO.: _____

OVERBURDEN MONITORING WELL SHEET

PROJECT:		DRILLING Co.:		BORING No.:	
PROJECT No.:		DRILLER:		DATE COMPLETED:	
SITE:		DRILLING METHOD:		NORTHING:	
GEOLOGIST:		DEV. METHOD:		EASTING:	



ELEVATION OF TOP OF SURFACE CASING: _____
STICK -UP TOP OF SURFACE CASING: _____
ELEVATION OF TOP OF RISER PIPE: _____
RISER STICK-UP ABOVE GROUND SURFACE: _____
I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

GROUND ELEVATION: _____
TYPE OF SURFACE SEAL: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____
TYPE OF SEAL: _____

ELEVATION / DEPTH OF SEAL: _____ / _____
TYPE OF SEAL: _____

ELEVATION / DEPTH TOP OF FILTER PACK: _____ / _____

ELEVATION / DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE X LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF FILTER PACK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____
ELEVATION / DEPTH BOTTOM OF FILTER PACK: _____ / _____
TYPE OF BACKFILL BELOW WELL: _____
ELEVATION / DEPTH OF BOREHOLE: _____ / _____

STANDARD OPERATING PROCEDURE
SOP-13

MONITORING WELL DEVELOPMENT

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper development of new and existing monitoring wells. The methods described herein are specific for monitoring wells located at Naval Support Activity (NSA) Crane. Guidelines by Southern Division, Naval Facilities Engineering Command, (South Div NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 Water Well Drillers of Chapter 310 of the Indiana Annotated Codes (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

The drilling contractor will provide adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing the development of monitoring wells. The drilling contractor personnel must have all of the health and safety training required to perform the work, as specified in the Health and Safety Plan (HASP).

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the Site Safety Officer.

Well development equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).

4.0 WELL DEVELOPMENT METHODS

The development of new wells shall not occur until at least 24 hours after the well has been installed and grouted. This time is required so that the grout in the annulus can set and harden. The purpose of well

development is to stabilize and increase the permeability of the sand pack and the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water, if any, is removed from the well.

Sequential measurements of pH, specific conductance, turbidity, and temperature will be taken during development. Development should proceed until criteria are met as stated in Navy Guidelines.

Vigorous on-and-off pumping or a surge block will be used for development.

A surge block that is approximately the same diameter as the well riser will be used to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. Site-specific conditions will dictate which type will be used. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

Development should proceed until three consecutive pH, specific conductance, and temperature readings are within 10 percent of each other and three consecutive turbidity readings are within 5 Nephelometric Turbidity Units (NTUs) of each other. If these criteria cannot be met after five volumes of water have been removed, then one additional well volume will be removed and well development will be considered complete.

If for any reason the above criteria cannot be met, the site geologist should document the event in writing and consult with the TtNUS Project Manager (PM) regarding an alternate plan of action.

Well development must be completed at least 24 hours before well sampling. The intent of this hiatus is to provide time for the newly installed well and backfill materials to sufficiently equilibrate to their new environment and for that new environment to re-stabilize after the disturbance of drilling.

5.0 ATTACHMENTS

1. Monitoring Well Development Record

ATTACHMENT 1

MONITORING WELL DEVELOPMENT RECORD

MONITORING WELL DEVELOPMENT RECORD

Page ___ of _____

Well: _____ Depth to Bottom (ft.): _____ Responsible Personnel: _____
Site: _____ Static Water Level Before (ft.): _____ Drilling Co.: _____
Date Installed: _____ Static Water Level After (ft.): _____ Project Name: _____
Date Developed: _____ Screen Length (ft.): _____ Project Number: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

STANDARD OPERATING PROCEDURE

SOP-14

MEASUREMENT OF WATER LEVELS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for determining water levels in monitoring wells.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following equipment and field forms are required for determining water levels in monitoring wells.

Ground Water Level Measurement Form: A copy of the Ground Water Level Measurement Form is attached.

Bound Field Log Book

Photoionization Detector (PID)

Well Key

Electronic Water-Level Indicator: The water level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01 feet.

Decontamination Supplies

3.0 WATER-LEVEL MEASUREMENT PROCEDURES

3.1 Check the operation of the electronic water level indicator or interface meter.

3.2 Record the well identification (ID), date, and time (using military time) on the Ground Water-Level Measurement Form.

3.3 Unlock the well and remove the well cap.

3.4 Place the well cap on a clean piece of plastic.

3.5 Check the well for the presence of organic vapors in the 2-inch PVC riser pipe as follows:

1. Calibration of the PID shall be done in accordance with appropriate calibration procedures.

2. Insert the PID sample inlet straw approximately three inches into the riser pipe.

3. Record the PID reading on the Ground Water Level Measurement Form. If the reading is less than concentrations specified in the site-specific Health and Safety Plan (HASP), proceed to step 3.6. If the reading is greater than the concentration specified in the HASP, measure the concentration in the breathing zone. If the concentration in the breathing zone is less than the concentration specified in the HASP, proceed to Step 3.6. If the reading is greater than the specified concentration, allow the riser pipe to ventilate for ten minutes and repeat the measurement of breathing zone concentrations until the concentrations fall below the level specified in the HASP before proceeding to step 3.6.

3.6 Ensure that the water level indicator probe has been decontaminated before use.

3.7 Slowly lower the probe into the well riser pipe (or into the surface water for staff gages) until an audible and/or visible signal is produced, indicating contact with the water surface.

3.8 Read the water level measurement from the top of the inner casing (or from the staff gage reference point) at the surveyed reference point to the nearest 0.01-foot.

3.9 Record the water level measurement on the Water Level Measurement Form.

3.10 Wind the meter cable measuring tape back onto the spool.

3.11 Replace the well cap and lock.

3.12 Decontaminate the meter's probe and cable.

4.0 ATTACHMENTS

1. Water Level Measurement Sheet

ATTACHMENT 1

WATER LEVEL MEASUREMENT SHEET

WATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ___ No ___ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Page ___ of ___

STANDARD OPERATING PROCEDURE

SOP-15

LOW-FLOW WELL PURGING AND STABILIZATION

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for well purging and stabilization utilizing low-flow techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow purging.

Low-Flow Purge Data Sheet: A copy of this form is attached at the end of this SOP.

Ground Water Sample Log Sheet: A copy of this form and instructions for its completion are included in SOP-16.

Bound Field Log Book

Well key

Electronic water level indicator: The water level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01-feet.

Electronic Programmable Controller, model 400 or comparable: This controller regulates air flow in a bladder pump.

Cylinder of compressed nitrogen with regulator: Compressed gas serves as the power source for the bladder pump.

Multiple parameter water quality meter: This unit measures and displays field parameters measured in the field including pH, dissolved oxygen, oxidation-reduction potential (ORP), temperature, and specific conductance.

Flow-through cell adapter for water quality meter

LaMotte Turbidity Meter or comparable: Used to measure turbidity.

Purge water containers

Graduated cylinder and stopwatch: Used to calculate flow rate.

3.0 PROCEDURES FOR WELL PURGING

3.1 Prior to mobilizing to the site, clean, check for proper operation, and calibrate as per manufacturer requirements above equipment as necessary.

3.2 Obtain a static water level measurement of the well to be purged. Record the information on the Ground Water Sample Log Sheet and the Low-Flow Purge Data Sheet. Leave the water level meter suspended in the well casing.

3.3 Calculate one well casing volume as follows:

1. Obtain the total depth of the well.

2. Using the static water level determined in Step 3.2 of this SOP and the total depth of the well, calculate the well casing volume using the following formula:

$$V = (0.163)(T)(r^2)$$

where:

V	=	Static casing volume of well (in gallons).
T	=	Vertical height of water column (linear feet of water).
0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.
r	=	Inside radius of the well casing (in inches).

Note: For wells of 1-inch radius (2-inch diameter) V = 0.163 gallons per foot of water column.

3.4 Connect the pump controller to the well pump air supply (at the well cap) by following the instructions in the pump control manual. The pump controller must be turned off when being connected.

3.5 Connect the nitrogen cylinder to the pump controller. The nitrogen cylinder valve must be closed and the regulator line pressure set at zero pounds per square inch (PSI) when being connected.

3.6 Following the instructions found in the water quality meter manual, connect the flow-through cell to the pump discharge line (at the well cap).

3.7 Place the discharge tubing from the flow-through cell to direct the purge water discharge into the graduated cylinder or purge-water container.

3.8 Following the instructions in the pump controller manual, start pumping water from the well.

3.9 Start with the initial pump rate set at approximately 0.1 liters/minute. Use the graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 feet during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters/minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 Nephelometric Turbidity Units (NTUs) after all other field parameters have stabilized. If ground water is drawn down below the top of the well screen, purging will cease and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be collected from these wells within the same 8-hour workday and no later than 24 hours after the start of purging.

The time to sample any given well will vary greatly due to the many variables associated with low flow purging and sampling, such as:

- Stabilization of parameters
- Possible draw down
- Analytical changes from quarter to quarter
- Varying QA sample requirements from quarter to quarter
- Variable pump rates

Normally, the time from the start of purging to the end of sampling will be between 1 to 4 hours.

3.10 Measure the well water level using the water level meter every five to ten minutes. Record the well water level on the Low-Flow Purge Data Form (attached at the end of this SOP).

3.11 Record on the Low-Flow Purge Data Form every five to ten minutes the water quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, and dissolved oxygen) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned

during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form.

3.12 Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.

3.13 During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections.

3.14 Stabilization is achieved and sampling can begin when a minimum of one casing volume has been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits:

pH $\pm$ 0.1 standard units
Specific conductance $\pm$ 3%
Temperature $\pm$ 1.0 ºC
Turbidity less than 10 NTUs

If the above conditions have still not been met after the well has been purged for four hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data From onto the Ground Water Sample Log Form.

If there is a need to leave a well during purging, there are two options:

- One, if the sampler must move for 30 minutes or less but still has a clear line of sight to the well, the sampler may leave the pump running and watch the well until the sampler is able to return to the well.

- Two, if for whatever reason, the sampler must stop purging for an extended period of time or a clear line of sight cannot be maintained, the pump and cell will be shut-down. All equipment and supplies will be loaded into the sample vehicle, and the well will be secured before departing.

In both cases, the time purging was stopped and restarted will be noted on the Low-Flow Purge Data Form.

3.15 Once sampling activities have been completed, turn the pump off. Remove pump, hoses, cables, and other equipment from the well.

3.16 Decontaminate pumps, hoses, cables, flow-through cell, and other equipment.

4.0 ATTACHMENTS

1. Low-Flow Purge Data Sheet

ATTACHMENT 1
LOW-FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: ______________________ **WELL ID.:** ______________________
PROJECT NUMBER: ______________________ **DATE:** ______________________

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Comments

SIGNATURE(S): ______________________________ **PAGE___OF___**

STANDARD OPERATING PROCEDURE
SOP-16

MONITORING WELL SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for monitoring well sampling. Low-flow sampling techniques will be used for ground water sampling at Naval Support Activity (NSA) Crane.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow sampling of monitoring wells:

Ground Water Sample Log Form: A copy of this form is attached at the end of this SOP.

Bound field log book

Chain-of-Custody Form

Bladder Pump

Surgical Gloves

Labeled sample containers: Sample containers are certified clean by the laboratory supplying the sample containers.

Tag for each sample container

Plastic storage bags

Shipping containers with ice

3.0 MONITORING WELL SAMPLING PROCEDURES

3.1 Ground water sampling may be initiated when the monitoring well has been purged and stabilized.

3.2 Record the sample start time (using military time) on the Ground Water Sample Log Sheet. Record the field measurements for pH, oxidation-reduction potential (ORP), specific conductance, temperature, dissolved oxygen, and turbidity.

3.3 With the pump continuing to run, disconnect the flow-through cell from the pump discharge tube and immediately start filling sample bottles directly from the pump discharge. All sample

containers will be supplied by the laboratory, and the laboratory will pre-preserve all sample containers, where appropriate.

3.4 Allow the pump discharge to flow gently down the inside of the container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled. Sample containers for volatile organic compounds (VOCs) must be completely filled so that no headspace exists in the container. The VOC vials shall be filled to the top so that a convex meniscus is formed. Gently secure the cap, turn the vial upside down, and check to see if any air has been trapped inside the vial. If so, open the cap, reform the meniscus, and attempt again to secure the lid without trapping air in the sample. All other sample containers can have air space included when the container lid is secured.

3.5 Cap each container immediately after filling.

3.6 Record the sample time on the Ground Water Sample Log Form, the sample tag, and on the sample label.

3.7 Secure the associated tag to each sample container.

3.8 Place the tagged sample container into a plastic storage bag and then into a cooler containing ice.

3.9 Enter the proper information on the Chain-of-Custody form for each sample container.

3.10 Repeat steps 3.3 through 3.9 for each sample container collected.

3.11 The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Ground Water Sample Log Form.

3.12 All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. All samples will be collected in the following sequence (where applicable):

Volatile Organic Compounds (VOCs)
Appendix IX Metals

3.13 If the last turbidity measurement prior to the commencement of sampling showed turbidity to be greater than 5 Nephelometric Turbidity Units (NTUs), then filtered aliquots of ground water will be collected and analyzed for dissolved metals and dissolved thorium isotopes. Without turning off the pump, attach a disposable, inline, 0.45-um filter cartridge at the end of the discharge tube. Fill sample containers marked for "dissolved metals" so that the laboratory knows that these aliquots are distinct sample fractions and that the results should be reported as dissolved analytes. Samples scheduled for VOC analysis shall not be filtered.

3.14 Repeat steps 3.5 through 3.9 for the filtered sample containers.

3.15 After completion of sample collection, remove the bladder pump from well and decontaminate.

3.16 Replace the outer protective well cap and lock the well.

3.17 All equipment should be cleaned and packed into the sample vehicle, along with the sample cooler for transport. Disposable gloves and other equipment should be placed in a plastic trash bag and handled as investigation derived waste.

4.0 ATTACHMENTS

1. Ground Water Sample Log Sheet

ATTACHMENT 1

GROUND WATER SAMPLE LOG SHEET

GROUND WATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	Color	pH	S.C.	Temp.	Turbidity	DO	TBD	TBD
	Visual	Standard	mS/cm	^{0}C	NTU	mg/l		
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	TBD	TBD
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

TBD: To Be Determined

STANDARD OPERATING PROCEDURE
SOP-17

CALIBRATION AND CARE OF WATER QUALITY METERS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The YSI 600-Series Environmental Monitoring System or the Horiba U20–Series multi-parameter water quality monitoring system will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), and dissolved oxygen (DO) in water. A LaMotte turbidity meter will be used in conjunction with the water quality meter to measure turbidity. The water quality meter will have a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a ground water discharge or can be immersed in a surface water body such as a stream, pond, or drainage ditch. The LaMotte is a hand held meter that uses a multi-detector optical configuration to assure long term stability and minimize stray light and color interferences. All comparable equipment used in place of the equipment items identified in Section 2.0 below must be comparable in terms of sensitivity, accuracy, and precision.

2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

Site logbook
Equipment calibration log sheet
YSI Model 600 Series and Sonde or Horiba U20 Series, or comparable: multi-parameter water-quality meter with flow through cell.
LaMotte Turbidity Meter, or comparable
Equipment manual
Calibration kit
Deionized water, paper towels, spray bottle, etc.
Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES

This section describes the calibration procedures for the YSI Model 600 series, the Horiba U20 series, and the LaMotte. Each meter is supplied with an instruction manual and will be on site and will be used as the calibration guidance documents. These procedures will list requirements for frequency of calibration and checks to be performed on the meter.

3.1 YSI Model 600 Series and Horiba U20 Series

The YSI Model 600 series and Sonde and the Horiba U20 series are multi-parameter, water-quality meters that may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. With the flow through cell attached, the meters have the ability to measure water-quality parameters in ground water via a pump discharge line. By performing the measurements in the discharge line coming directly from the well, the parameters are measured before the ground water comes in contact with the atmosphere. The parameters measured by the YSI or the Horiba for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP
- Turbidity

3.1.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.1.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the meter will be checked each day that it is used. If the check shows any out-of-specification readings, the specific probe will be recalibrated. Meter specifications can be found in the equipment manual, starting on page 248 (YSI) or page 93 (Horiba). Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

3.1.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.

- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.

- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.

- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.

- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

3.2 Lamotte Turbidity Meter

The Lamotte turbidity meter is a hand held meter that measures the amount of suspended matter in water using the Nephelometric method.

3.2.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks

performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.2.2 Calibration

Turbidity must be calibrated prior to the start of each field effort. After this initial calibration, the LaMotte will be calibrated each day that it is used. If the check shows any out-of-specification readings, the meter will be recalibrated. Meter specifications can be found in the equipment manual. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

3.2.2 Tips for Good Calibration

- Thoroughly clean the standard vial with a chem wipe to remove finger prints.

- Make sure that the vial is properly aligned according the manual recommendations.

4.0 MAINTENANCE

The YSI and/or Horiba Meter and LaMotte will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

4.1 Meter Storage for the YSI and Horiba

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Multi-parameter short term storage key points:

- Use enough water to provide humidity but not enough to cover the probe surfaces.
- Make sure the storage vessel is sealed to minimize evaporation.
- Check periodically to make certain that water is still present.

4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult Section 2.10 on page 89 (YSI) or Section 7.1 on page 86 (Horiba) of the equipment manual.

Note: Reagents that are used to calibrate and check the water quality meter may be hazardous. Review the health and safety plan and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

4.3 Meter Storage for the LaMotte

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. Proper storage of the meter between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Short term storage key points:

- Make sure the storage vessel is moisture free and sealed.

4.4 Sample Vial Cleaning

- Rinse the vial thoroughly with potable water to remove sediments.
- Wipe with chem.-wipes or cotton swabs.

5.0 ATTACHMENTS

1. Equipment Calibration Log

EQUIPMENT CALIBRATION LOG

PROJECT NAME : ______________________ INSTRUMENT NAME/MODEL: ______________________

SITE NAME: ______________________ MANUFACTURER: ______________________

PROJECT No.: ______________________ SERIAL NUMBER: ______________________

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

APPENDIX B
LAB STANDARD OPERATING PROCEDURES
TABLE OF CONTENTS

Empirical Laboratories
Empirical Laboratories DoD Certificate of Accreditation
SOP-100 Metals Digestion/Preparation
SOP-103 Mercury Analysis in Water
SOP-104 Mercury Analysis in Soil/Sediment
SOP-105 Metals
SOP-164 Cyanide
SOP-175 Cyanide
SOP-201 SVOCs and Low-Concentration PAHs
SOP-202 VOCs Aqueous
SOP-208 Herbicides
SOP-211 Pesticides/PCBs
SOP-221 Total Organic Carbon
SOP-225 VOCs Non-Aqueous
SOP-300 SVOCs Aqueous
SOP-302 Pesticides/PCBs Aqueous
SOP-343 BNA and Pesticide/ PCB
SOP-405 Laboratory Waste Disposal
QS10-R14 Sample Receiving, Storage, and Login
QS14-R06 Waste Disposal

Cape Fear Analytical
1 **-** Accreditation
2 - Extraction
3 - Analysis
4 - Quality Assurance plan

LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 ***Certificate Number L2226***

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 30, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 30th of November 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 21 EFFECTIVE DATE: 20100901

METALS DIGESTION/PREPARATION

References:

Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C 21st
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 21, 9/1/10

- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
   a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
   a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
   b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, “Acid Digestion of Soil/Sediment”
   a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. **NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one litter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS

1. Samples are taken in high density polyethylene (CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS

1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.
B. Be certain the exhaust hood is functioning before you begin the digestion procedure.
C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.
B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.
C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.

F. Beckman CS-6R centrifuge.
G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
H. Whatman No. 41 filter paper or equivalent.
I. Whatman No. 42 filter paper or equivalent.
J. Whatman 0.45 micron filter paper or equivalent.
K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
M. Manual Sample Mill
N. Wiley Sample Mill
O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO_3). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H_2SO_4). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in LIMS.

2. PREPARATION

A. Laboratory control sample
   1. Aqueous
      a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIlM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample LIMS.
      b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 and 0.3125 mLs HCl to each 50 mL vessel.
   2. Solids:
      a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. BS1 and then BS2 etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbook and transfered to LIMS.

B. Spiking solution
   1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
   2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
   3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less then 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

IX. PROCEDURE

A. Glassware preparation for digestion or when the hot-block can not be used:
   1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
   2. Rinse glassware with reagent water that contains 5% HNO_3 and 5% HCl followed by a rinse with reagent water.
   3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

B. Aqueous sample filtration (for dissolved metals):
   1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO_3 followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
   2. Rinse a 0.45 micron filter with 1:5 HNO_3 thoroughly, followed by D.I. water.
   3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
   4. Discard the first 50 to 100 mL.
   5. A preparation blank must be taken through the filtration step and analyzed with the sample.
   6. Preserve the sample with HNO_3 to pH<2.
   7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO_3. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

C. Aqueous sample preparation
   1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ".**
      a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
      b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO_3 to the sample.
      c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
      d. Cover the sample with a ribbed watch glass or equivalent source.
      e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, (12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
      f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
h. The sample is now ready for analysis.
i. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".
a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
b. Add 1.0 mL concentrated HNO_3 to the sample.
c. Add 2.50 mL concentrated HCl to the sample.
d. Cover the sample with a ribbed watch glass or equivalent source.
e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes.**
f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
g. Bring sample to its predigestion volume with DI water in the digestion vessel.
h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
i. The sample is now ready for analysis.
j. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
b. Add 1.5 mL concentrated HNO_3 to the sample.
c. Cover the sample with a ribbed watch glass.
d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil**. **This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.
e. Add another 1.5 mL portion of concentrated HNO_3 to the sample.
f. Cover the sample with a ribbed watch glass.
g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
j. Cover the digestion vessel and reflux for an additional 15 minutes.
k. Bring sample to its predigestion volume in digestion vessel.
l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

m. The sample is now ready for analysis.
n. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals"**

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
b. Add 2.5 mL 1:1 HCl to the sample.
c. Heat 15 minutes in a hot bath.
d. Filter through a membrane filter.
e. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry

enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, **"Acid digestion of Soil/Sediment"**
   a. Mix the sample thoroughly to achieve homogenity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
   b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3, replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
   c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
   d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)
   e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivelent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3, mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. The sample is now ready for analysis.
   f. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.
2. Method 3050B, **"Acid digestion of Sediments, Sludges and Soils"**
   a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
   b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

      NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.
   c. Add 5 mL D.I. water and 5 mL concentrated HNO_3(1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for

10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook

d. Allow the sample to cool. Add 5 mL concentrated HNO_3, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO_3. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C ± 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.

e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2.)

f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.

g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling

h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration , all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

j. The sample is now ready for analysis.

k. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank
   a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
   b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.
2. Blanks
   a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
   b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
   c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
   d. Sample is given a unique identifier in the digestion log.
3. Laboratory Control Samples
   a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
   b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
   c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
   d. Sample is given a unique identifier in the digestion log.
4. Duplicates
   a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

      NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.
5. Blank Spike
   a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike
   a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

      NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.
   b. The following metals do not get digested spikes when using CLP spike.

      Calcium
      Magnesium
      Sodium

Potassium

c. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)

d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.

XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.
   1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
   1. Redigest another sample aliquot.
   2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.
   1. All metals glassware must be kept separate from all other laboratory glassware.
   2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
   3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.

F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 103 **REVISION #: 18** **EFFECTIVE DATE: 041110**

MERCURY ANALYSIS IN WATER
BY MANUAL COLD VAPOR TECHNIQUE
METHODS USEPA SW846 7470A and 245.1 CLP-M 4.1
(NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003),
ADDENDUM FOR USEPA CLP ILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary

Revision 18, 04/11/10

- The SOP is an update from Revision 17 dated 03/25/10
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- Tables have been updated to reflect the current limits/processes.

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1.0 Identification of the Test Method

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury, and is compliant with SW846 Method 7470A, USEPA Method 245.1, and USEPA SOW ILM04.1.

2.0 Applicable Matrix or Matrices

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

3.0 Detection Limit

Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	**AQUEOUS MDL/DL (ug/L)**	**AQUEOUS LOD (ug/L)**	**AQUEOUS ERL/LOQ (ug/L)**	**AQUEOUS CRQL ILMO 4.1 (ug/L)**	**AQUEOUS CRQL ILMO 5.2 (ug/L)**
Mercury	0.080	0.16	0.20	0.20	0.20

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	**253.7**

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.

4.4 The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

7.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

7.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).

7.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

8.0 Safety

8.1. Normal accepted laboratory practices should be followed while performing this procedure.

8.2. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.3 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

8.4 The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

9.0 Equipment & Supplies

9.1 Perken Elmer Flow injection Mercury system.

9.2 Mod Block Digester set to maintain 95$\pm$2°C for 2 hours.

9.3 Polypropylene sample digestion vessels with snap or screw caps or equivalent. Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.

10.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra Scientific and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at room temperature.

10.1 REAGENTS

10.1.1 Concentrated sulfuric acid suitable for Hg determination.
10.1.2 Concentrated nitric acid suitable for Hg determination.
10.1.3 Stannous chloride: In a 1000 mL volumetric flask add approximately 500 mLs D.I. water, 30 mLs concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
10.1.4 3% HCl Carrier Solution: Dilute 30 mLs of concentrated metals grade HCl to one liter. Prepare fresh daily.
10.1.5 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.
10.1.6 Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mLs of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring (minimum of 3 hours if absolutely necessary). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.
10.1.7 Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mLs D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

10.2 STANDARDS

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within Element as well as on the container's label.
10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.
10.2.1.3. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All standards, blanks, and samples are taken through the digestion process.
10.2.1.4 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique Element identifier.

10.2.1.5 Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3. This solution is recorded in Element and given a unique Element identifier.

10.2.2 Calibration Standards

Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

10.2.2.1 Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

ug/L Hg	mLs of 200 ug/L standard in 30 mLs
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

10.2.2.2 Appropriate reagents are added as below in the sample preparation section.

10.2.2.3 Prepare one vessel for each.

10.2.2.4 It is necessary to digest the calibration standards.

10.2.3 Calibration Verification Standards

10.2.3.1. Initial calibration verification (ICV) solution – 4.0 ug/L

10.2.3.1.1 Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

10.2.3.1.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.1.3 It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.3.2 Continuing calibration verification (CCV) solution

10.2.3.2.1 Prepared from the primary source standard.

10.2.3.2.2 Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

10.2.3.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.3.2.4 It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.4 Digestion standards

10.2.4.1 Blank Spike

10.2.4.1.1 Prepared from the secondary source standard.

10.2.4.1.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 This solution should be given a unique identifier within Element.

10.2.1.2 Matrix Spikes

10.2.1.2.1 Prepared from the secondary source working standard.

10.2.1.2.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

10.2.1.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.

11.2 The holding time for the mercury digestion is 28 days from time of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BS’s are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 (Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)

12.4 Daily

12.4.1. **The instrument must be calibrated daily for all projects.**

12.4.2 Begin each analysis with an ICV(QCS) second source. The control limits are ±10% and IPC (CCV) for 245.1, limits are ± 5% and subsequent analyses are ± 10%.

12.4.3 Analyze ICB. Control limits (<±MDL for USACE or ±RL/CRDL for others and CLP)., depending on method. **No analyte detected >2xMDL for DOD.**

12.4.4 If the ICV (QCS) is not in control a new curve must be analyzed prior to sample analysis.

12.4.5 If the IPC (initial CCV) for 245.1 is not within the limits of ±5%, try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.

12.4.6 Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are ±20% for SW846-7470 and ± 10% for 245.1.

12.6.7 A CCB must always follow a CCV, the control limit is (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected >2xMDL for DOD.**

12.5 Quarterly or as needed when doing straight CLP work.

12.5.1 IDL's for CLP 4.1.

12.6 Digestion

12.6.1 BS data should be maintained and available for easy reference or inspection.

12.6.2 BLK (<1/2 ±RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP).

12.6.2.1 Employ a minimum of one preparation blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be re-digested and re-analyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.

12.6.3 Laboratory control sample (BS)

12.6.3.1. Employ a minimum of one laboratory control sample (BS) per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are ±20% for SW846-7470 and ±15% for 245.1. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.

12.7 Sample matrix:

12.7.1 Analyze one replicate sample for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. CLP does not allow this. Project specific requirements will take precedence in these situations.

12.7.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within ±25% of the true value (**± 20% for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.

NOTE: For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

12.7.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.

12.7.4 For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be ±10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.7.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

13.0 Calibration and Standardization

Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1 Set up the instrument with proper operating parameters.

13.1.1 Perkin Elmer Flow Injection Mercury System (FIMS).

13.1.1.1. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. **(Perkin-Elmer tygon tubing, waste**

and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

13.1.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

13.1.1.3 Turn on PE 100 spectrophotmeter; (Note: this must be on in order to start up the software on the computer.)

13.1.1.4 Turn on computer and go to icon "AA Win LAB Analyst".

13.1.1.5 Go to method; select "Hg CAL 2" then OK.

13.1.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.

13.1.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.

13.1.1.8 Save entered sample list under "Save ….sample info file" Note: description and batch ID are normally the date of analysis.

13.1.1.9 Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.

13.1.1.10 Turn the printer on.

13.1.1.11 Connect all tubing to the pump and blocks.

13.1.1.12 Start the pump by going to "FIAS" and click the pump 1 Icon (120).

13.1.1.13 The pump will start, then lock down and tighten the tubes onto the pump.

13.1.1.14 Turn on the nitrogen tank, it should be above 500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.

13.1.1.15 The pressure gauge on the PE100 should be just below 100.

13.1.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

13.1.1.17 Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

13.1.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl2. (Click the valve fill inject and make sure flow is correct and the line is rinsed).

13.1.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

13.1.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.

13.1.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".

13.1.1.22 "Select Location" enter #'s to be ran, and then press "OK". Samples are done in increments of 10 samples

13.2 Analyze the calibration standards as below.

13.2.1 New calibration points must be analyzed when the ICV analysis is not within ± 5%. **A curve must be analyzed daily for all projects especially USACE and CLP projects.**

13.2.2 The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of $\geq$0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

14.0 Procedure

14.1 Glassware preparation

14.1.1 After use, samples are neutralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.

14.1.2 Acid clean the glassware used for mercury prep as follows:

14.1.2.1 Rinse with low Hg content 1:1 HCl.

14.1.2.2 Rinse with D.I. water.

14.2 Label the vessels indicating which sample will be in each.

14.3 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below.

14.4 Sample preparation

14.4.1. Transfer 30 mLs, or an aliquot diluted to 30 mLs of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

NOTE: Normally, **a**n automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix gets spiked. Check with your manager.

14.4.2 Add 1.5 mLs of concentrated sulfuric acid to each vessel and mix.

14.4.3 Add 0.75 mL of concentrated nitric acid to each bottle and mix.

14.4.4 Add 4.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mLs). If the purple color does not persist after the addition of 7.5 mLs $KMnO_4$ the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

14.4.5 Add 2.4 mLs of potassium persulfate to each vessel and mix. Cover.

14.4.6 Heat for 2 hours in the block digester at 95$\pm$2°C (the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool.

14.4.7 Samples may be saved at this point if there is not time to run the whole set that day.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

14.5 Sample analysis

14.5.1 Set up the instrument as described in the calibration section above.

14.5.2 When ready to run samples, add 1.8 mLs of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of $\pm$10% for SW846-7470 and ±5 % for 245.1. Followed by the ICB (< ±MDL for USACE or ±RL/CRDL for others and CLP).

14.5.3 Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of $\pm$20% for SW846-7470 and ±10% for 245.1.

14.5.4 CCB must always follow the CCV. Control limits are (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected >2xMDL for DOD.**

14.5.5 The auto-sampler log is set up to analyze 106 samples at a time.
Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ-ICV
10	SEQ-ICB
11	BS

AS LOC	Sample ID
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB

23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.6 Data Reporting

14.6.1 Reduce data to result which will be reported.

14.6.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Apply a least squares fit to the calibration standards plotting µg Hg/L versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume (the 0.1 µg Hg standard will be input as 1.0 µg Hg/L)
(0.1µg Hg / 0.030 L solution).

15.3 Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.

15.4 Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.

15.5 Report the data as µg Hg/L of sample.

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.3 mL of the second source standard to 30 mLs with reagent water in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

19.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

19.2.1 ICV (**QCS for 245.1**)- second source not within ± 10%.

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within ± 20% for SW846 and ±10% for (**245.1, ± 5% for initial IPC and + 10% for subsequent IPCs**)

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.

1. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
2. If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 CORRECTIVE ACTION: DIGESTION RELATED

19.3.1 The preparation blank less than <1/2 RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP.

A. If the problem is with the instrument or stannous chloride.
Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, re-prepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.

B. If the problem is with the digestion.
All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be re-digested/re-analyzed or reported as qualified. The project manager or QA manager will make this determination.

C. LCS not within control limits (or ±20%, ± 15% for **245.1**).

1. If the problem is with the instrument, reanalyze when instrument is in control if further sample bottles are available.
2. Is the problem is with the digestion.
   a. If biased low, associated samples must be re-digested.
   b. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

19.4.1 Replicate analysis RPD not within ±20%
The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within ±25%(**± 20% for DOD projects**)

A. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

B. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

19.4.3 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage

during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 7470A.

21.2 *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B.*

21.3 *USEPA Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

ADDENDUM FOR USEPA SOW ILM05.2

1. The CCV concentration must be different from the ICV.

2. The same CCV shall be used throughout analysis for an SDG.

3. Calibration standards must be within 5% of the standard concentration.

4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is ±30%.

5. Spike samples at 1 ug/L for water.

Table 1

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	**AQUEOUS MDL/DL (ug/L)**	**AQUEOUS LOD (ug/L)**	**AQUEOUS ERL/LOQ (ug/L)**	**AQUEOUS CRQL ILMO 4.1 (ug/L)**	**AQUEOUS CRQL ILMO 5.2 (ug/L)**
Mercury	0.080	0.16	0.20	0.20	0.20

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level	• If more than one calibration standard is used, r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Must be within $\pm$ 10% of true value	• Re-run ICV • Repeat ICAL
Continuing calibration verification (CCV)	• After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% of true value	• Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.
Method Blank (BLK)	One per prep batch	No analytes detected > ½ RL and greater that 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanlyzed.
BS	One per prep batch	Most stringent criteria listed within the LIMS.	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year	• Calculated value must be less than the Spike level .	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	**Sequence ID:**
Method:	**7470A/245.1 (Mercury)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation Blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?				

10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

_____

_____

_____

_____

_____

_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 104 **REVISION #: 19** **EFFECTIVE DATE: 041110**

MERCURY ANALYSIS IN SOIL/SEDIMENT BY MANUAL COLD VAPOR TECHNIQUE METHODS SW846 7471A 7471B, EPA 245.5 AND CLPILM 04.1 (NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003), ADDENDUM FOR USEPA CLP ILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary
Revision 19, 04/11/10

- The SOP is an update from Revision 18 dated 03/25/10.

Revision 18, 03/08/10

- The SOP is an update from Revision 17 dated 01/29/09.
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.
- Numerous improvements/modifications were made to this SOP. Details/specifications were added that require evaluation from start to finish.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with USEPA method 245.5, SW-846 method 7471A&B and CLP SOW ILM04.1.

2.0 Applicable Matrix or Matrices

2.1 This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.

3.0 Detection Limit

3.1 The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

3.2 Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	**SOLID/SOIL MDL/DL (mg/Kg)**	**SOLID/SOIL LOD (mg/Kg)**	**SOLID/SOIL ERL/LOQ (mg/Kg)**	**SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)**	**SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)**
Mercury	0.013	0.026	0.033	0.10	0.10

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	**253.7**

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood.

These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 A weighed portion of the sample is acid digested for 2 minutes at 95±2°C, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

7.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

7.3 Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.

7.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.2 Normal accepted laboratory practices should be followed while performing this procedure.

8.3 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.4 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

9.0 Equipment & Supplies

9.1 Perkin Elmer Flow Injection Mercury System (FIMS).
9.2 Perkin Elmer AS 90.
9.3 Mercury lamp.
9.4 Environmental Express Mod-Block digestion block capable of holding 95+2°C for 2 hours.
9.5 A scale or balance capable of weighing to 0.01 + 0.02 gram.
9.6 Snap cap digestion polypropylene vessels for use with the mod block digester. Five vessels of each lot must be taken through analysis to check for mercury.
9.7 Polypropylene watch glasses suitable for use with the above vessels in F above.
9.8 Manual Sample Mill
9.9 Wiley Sample Mill
9.10 Clippers for cutting vegetation

10.0 Reagents and Standards

10.0.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.0.2 Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Spex, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

10.1 REAGENTS

10.1.1 Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

10.1.2 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3. Both HNO_3 and HCl must be of the reagent grade suitable for mercury determinations.
NOTE: This reagent is required for use when USACE project samples are being digested.

10.1.3 Concentrated HCl.

10.1.4 Concentrated HNO_3.

10.1.5 Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H_2O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.

10.1.6 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note: this is normally made up 2 liters at a time.

10.1.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.

10.1.8 3 % HCl carrier solution: 30 mLs HCl – 1 L DI H20; Prepare fresh daily.

10.1.9 Potassium persulfate 5% solution: Dissolve 100g in 2 liters of D.I. water. Used with digestion of CLP soils.

10.2 STANDARDS

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number within Element as well as on the container's label.

10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

10.2.2 Preparation

10.2.2.1. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All Standards, blanks, and samples are taken through the digestion process.

10.2.2.2 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

10.2.2.3 Primary source and secondary source mercury standard solutions: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3 (200 ug/L).

10.2.3 Calibration standards:

Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.

10.2.3.1 Dilute the volumes below to 5 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 5 mLs for the initial step of the digestion.)

ug/L Hg	mLs of 200 ug/L standard in 50 mL

0.20	0.050
0.50	0.125
1.0	0.25
ug/L Hg	mLs of 200 ug/L standard in 50 mL
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

10.2.3.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.3 Prepare one vessel of each.

10.2.3.4 It is necessary to digest the calibration standards when following all mercury methods.

10.2.4. Calibration verification standards:

10.2.4.1. Initial calibration verification (ICV) solution – 4.0 ug/L.

10.2.4.1.1 Prepared from the secondary source mercury standard (200 ug/L).

10.2.4.1.2 Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 It is necessary to digest the ICV standards when using all mercury methods for soil.

10.2.4.2 Continuing calibration verification (CCV) solution:

10.2.4.2.1 Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.

10.2.4.2.2 Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mLs with reagent water in a polypropylene digestion vessel.

10.2.4.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.2.4 It is necessary to digest the CCV standards when following all mercury methods for soil.

10.2.5 Digestion standards:

10.2.5.1 Laboratory control sample:

10.2.5.1.2 The Laboratory Control Sample (BS) is prepared from the secondary source mercury standard (200 ug/L) and added to ~ 0.3 grams of teflon chips.

10.2.5.1.3 Prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of teflon chips.

10.2.5.1.4 Appropriate reagents are added as below in the sample preparation section.

10.2.5.1.5 This solution is given a unique identifier in Element.

10.2.5.2 Matrix Spikes

10.2.5.2.1 Prepared from the primary or secondary source mercury standard (200 ug/L).

10.2.5.2.2 Prepared by adding 0.50 mL of the mercury standard (200 ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.

C10.2.5.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.

11.3 Refrigerate solid samples at 4°C (±2°C) upon receipt until digestion and analysis.

11.4 The sample should be analyzed without drying. A separate percent solids determination is required

11.5 The holding time for digestion of mercury samples is 28 days.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QUALITY CONTROL (Reference SW-846, 7471A Update III, 7471B Revision 2 February 2007, USEPA CLP ILMO 4.1 or EPA 245.5 for further clarification)

12.3.1 Daily

12.3.1.1 The instrument must be calibrated daily for all projects.

12.3.1.2 Begin each analysis with an ICV(concentration at or near mid range). The control limits are +10% for 7471A and 245.5, ±20% for 7471B and ±5% for 245.5..

12.3.1.3 Analyze ICB. Control limit is <± MDL or ±RL/CRDL for other or CLP. For DOD, no analyte detected > 2x MDL.

12.3.1.4 If the ICV is not in control a new curve must be analyzed prior to sample analysis.

12.3.1.5 Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. The control limits are +20% for SW846-7471A, SW846 7471B and ±10% for 245.5. If an exceedance occurs, analyze another CCV, if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.

12.3.1.6 Follow each CCV with a CCB. Control limit is < ±MDL or ±RL/CRDL for others or CLP. For DOD, no analyte detected > 2x MDL.

12.3.2 Quarterly

12.3.2.1 IDLs for CLP (Follow SOP - 414).

12.3.3 Annually

A. MDLs must be analyzed for all matrixes (Follow SOP - 414).

12.3.4 Digestion

12.3.4.1 BS data should be maintained and available for easy reference or inspection.

12.3.4.2 BLK (< ±½ RL or ±RL for common contaminates or ±RL/CRDL for others or CLP)

12.3.4.2.1 Employ a minimum of one BLK per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be < ± ½ RL for USACE or ±RL/CRDL for others or CLP. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit or if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit, but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA officer will make this determination.

12.3.4.3 Laboratory control sample (BS).

12.3.4.3.1 Employ a minimum of one BS per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are +20% for SW846-7471A, 7471B and 245.5 solid samples. A BS will accompany each batch of soil samples. If the BS is not in control, the Inorganic Manager and QA Officer must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the

data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This corrective action is documented in a CAR.

12.3.5 Sample matrix

12.3.5.1 Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations.

12.3.5.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within +25% for 7471A and ±20% for 7471B of the true value (+20% for DOD projects). If results do not fall within the control limit redigestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your supervisor. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of + 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.4 For 245.5 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be 10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.3.5.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6

13.0 Calibration and Standardization

13.0.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.0.2 Set up the instrument with proper operating parameters.

13.0.3 Perkin Elmer Flow Injection Mercury System (FIMS).

13.0.3.1 Prepare the instrument for calibration by the following steps:

13.0.3.1.1 Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the seperator cover must not have any moisture in it, if it does replace it. (Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

13.0.3.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

13.0.3.1.3 Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)

13.0.3.1.4 Turn on computer and go to icon "AA Win LAB Analyst"

13.0.3.1.5 Go to method; select "Hg CAL 2" then OK.

13.0.3.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time = 18 sec.; BCC time = 2 sec.

13.0.3.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.

13.0.3.1.8 Save entered sample list under "Save …sample info file" Note: description and batch ID are normally the date of analysis.

13.0.3.1.9 Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information." File. The other is to select a results library. Double click on heading and choose.

13.0.3.1.10 Turn the printer on.

13.0.3.1.11 Connect all tubing to the pump and blocks.

13.0.3.1.12 Start the pump by going to "FIAS" and click the pump 1 Icon (120).

13.0.3.1.13 The pump will start, then lock down and tighten the tubes onto the pump.

13.0.3.1.14 Turn on the nitrogen tank, it should be >500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.

13.0.3.1.15 The pressure gauge on the PE100 should be just below 100.

13.0.3.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

13.0.3.1.17 Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

13.0.3.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl2. (click valve fill inject and make sure flow is correct and the line is rinsed)

13.0.3.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

13.0.3.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.
13.0.3.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".
13.0.3.1.22 "Select location" enter the #'s of the samples to be analyzed, then "OK".

13.0.3.2 Analyze the calibration standards as below.
13.0.3.2.1 A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within ±10% for SW846 7471A and ±5 % for 245.5 methods, or ±20% for 7471B.

13.0.3.2.1 The curve should be linear with a calculated intercept with a minimum correlation coefficient of >0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.
13.0.3.2.2 CLP requires a blank + 5 calibration standards (0, .02, .05, .1, .5 and 1.0 µg). (One standard must be at CRDL or IDL whichever is greater.)

14.0 Procedure

14.1 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.

14.2 Sample preparation:

14.2.1 It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

14.2.1.1 The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
14.2.1.2 Two quarters should then be mixed to form halves.
14.2.1.3 The two halves should be mixed to form a homogenous matrix.
14.2.1.4 This procedure should be repeated several times until the sample is adequately mixed.
14.2.1.5 NOTE: Samples that are clay type materials must be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

14.2.2 Grinding of Vegetation Samples

14.2.2.1 Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to

fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

14.2.2.2 Transfer 0.30 g (for USACE work use anywhere from 0.20 to 1.0 g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and on the Element Batch Sheet. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. (Record in specific balance calibration log.)

14.2.2.3 Add 2.5 mLs of reagent water, and 2.5 mLs of aqua regia and mix for samples. Add 2.5 mLs of aqua regia to standards and mix.

14.2.2.4 Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at 95+2°C (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature).

14.2.2.5 Cool, bring to 30 ml with D.I. water.

14.2.2.6 Add 7.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mLs).

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

14.2.2.7 Heat for 30 minutes on the hot block at 95+2°C (The temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool. Samples may be saved at this point if there is not time to run the whole set that day.

14.2.2.8 Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.

14.2.2.9 Bring to 50 mLs with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. The samples are now ready for analysis.

14.2.2.10 NOTE: Stannous Chloride (10.1.5) and 3% HCl (10.1.8) are added by the instrument during analysis.

14.2.3 Sample analysis

14.2.3.1 Set up the instrument as described in the calibration section above.

14.2.3.2 When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step “3”. NOTE: When aqua-regia is added assume that all samples and standards have chlorine and treat accordingly. Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The

pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.

14.2.3.3 Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits of +10% for SW846-7471A or ±20% for 7471B and ±5% for 245.5 methods.

14.2.3.4 The ICB must follow the calibration standards (<±MDL (USACE) or ±RL/CRDL for other or CLP), but not before the ICV. No analyte must be detected > 2xMDL for DOD.

14.2.3.5 Each set of ten samples must be followed by a CCV with control limits of +20% for SW846-7471A and B and ±10% for 245.5 method. The run must also end with a CCV, then CCB.

14.2.3.6 Analyze CCB after calibration and each CCV. The CCB frequency is 10% or every 2 hours whichever is more frequent. (control limit is < ±MDL or ±RL/CRDL for other or CLP). For DOD, CCB at beginning and end of sequence and after every 10 samples. No analyte detected > 2xMDL.

14.2.3.7 Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ- ICV
10	SEQ-ICB
11	BS
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.2.3.8 Sample analysis:

14.2.3.8.1 Go to "Analyze", "select location" and type in the range of numbers needed to complete analysis. (ie. 9-54). Press enter and the autosampler will proceed to enter the selected range. NOTE: Check standards are loaded as part of the tray.

14.2.3.8.2 Make sure that the sample wash beaker is filled with 3% HCl.

14.2.3.8.3 Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are

made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL are reported flagged as estimated, ("B" flag).

14.2.4 Data reporting

14.2.4.1 Reduce data to result which will be reported using the soil spreadsheet found on the network..

14.2.4.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Pull up the blank spreadsheet at V: lab\metals\tests\mercury and transfer all the information pertinent to the current analysis. Save as the date of analysis. This information can be obtained from your mercury batch sheet.

15.3 Transfer the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

15.4 Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

15.5 The spreadsheet should divide the result which is the µg Hg obtained from the sample mass by the sample mass in grams. This will yield a result of µg Hg/g sample on a wet weight basis. Calculations in the spreadsheet should be checked occasionally to make sure that they are working correctly.

15.6 If available, divide the result by the %solids to obtain the result on a dry weight basis.

15.7 Report the data as µg Hg/g of sample (mg/kg wet or mg/kg dry when % solids are available).

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.5 mL of the second source standard (200 ug/L) add to ~0.3g to 5 mLs with reagent water/aqua-regia in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

17.0 Pollution Prevention

14.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

14.2Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

19.2 **CORRECTIVE ACTIONS: INSTRUMENT RELATED**

19.2.1 ICV not within + 10% (SW846) and (245.5)

19.2.1.1 If the problem is with the solution, re-prepare, obtain new stock if necessary.

19.2.1.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within + 20% (SW846) and (245.5)

19.2.2.1 If the problem is with the solution, reprepare, obtain new stock if necessary.

19.2.2.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

19.2.2.2.1 If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.

19.2.2.2.2 If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 **CORRECTIVE ACTION: DIGESTION RELATED**

19.3.1 The preparation blank less than ± ½ RL for DOD or ±RL/CRDL for others or CLP.

19.3.1.1. If the problem is with the instrument or stannous chloride.

19.3.1.1.1 Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.

19.3.1.1.2 If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.

19.3.1.2 If the problem is with the digestion, all associated samples which are below the method detection limit (MDL) or have a level of mercury

greater than 10X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

19.3.2 BS not within control limits.

19.3.2.1 If the problem is with the instrument, reanalyze when instrument is in control with another aliquot of the sample.

19.3.2.2 If the problem is with the digestion.

19.3.2.2.1 If biased low, associated samples must be redigested.

19.3.2.2.2 If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

19.4 **CORRECTIVE ACTION: SAMPLE MATRIX RELATED**

19.4.1 Replicate analysis RPD not within +20%

19.4.1.1 The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within +25% 7471A and ±20% 7471B (+20% for DOD projects)

19.4.2.1 If the analyte level in the sample is greater than 4X the spiking level, the % recovery can not be evaluated and no action is taken.

19.4.2.2 If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

21.0 References

21.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III/IV); Method 7471A, 7471B

21.2 USEPA Code of Federal Requlations, 40, CH 1,PT 136; Method 245.1; APX-B

21.3 USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

Addendum for USEPA CLP ILM 05.2

1. CCV concentration must be different from ICV.

2. The same CCV shall be used throughout analysis for a sample delivery group.

3. Calibration standards must be within 5% of the standard concentration.

4. 0.2 grams of sample must be used for the sample aliquot, add enough reagent water to each sample to make a total volume of 10 mL. Proceed with method as in the water method SOP 103.0 Revision 9.

5. The ICV and CCV must be at ±20% recovery.

6. A CRA must be analyzed at the beginning and end of each batch of 20 samples. Right after the ICV/ICB and right before the final CCV/CCB. The control limit is ±30%.

7. The matrix spike must be analyzed at the concentration of 0.5 mg/Kg.

Table 1

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	SOLID/SOIL MDL/DL (mg/Kg)	SOLID/SOIL LOD (mg/Kg)	SOLID/SOIL ERL/LOQ (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Mercury	0.013	0.026	0.033	0.10	0.10

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level	• If more than one calibration standard is used, r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must be within $\pm$ 10% for SW846 7471A,±20% for 7471B, or ±5% for 245.5 of true value	• Re-run ICV • Repeat ICAL
CCV	• After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% for SW846-7471A&B, ±10% for 245.5of true value	• Follow guidelines for SOP QS05
Closing CCV	• At the end of every sequence	• $\pm$ 20% for SW846-7471A&B, ±10% for 245.5of true value	• Follow guidelines for SOP QS05
BLK	One per prep batch	No analytes detected > ½ RL and greater that 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
BS	One per prep batch	Most stringent criteria listed within the LIMS.	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported • Follow guidelines from SOP QS05
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	• Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year	• Calculated value must be less than the Spike level •	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: SW846 7471A/B, EPA245.5 (Mercury)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did blank spike(BS) meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				

11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

1.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 105 REVISION #: 16 EFFECTIVE DATE: 041110

METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE

References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments

utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.

6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1 Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high- concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods

using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.

7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The

interference effects must be evaluated for each individual instrument since the intensities will vary.

7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.

7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

7.1.8 When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs.

Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized

7.4 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.

8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.

8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.

8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.

9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.

9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.

9.4. Auto-sampler: Thermo Fisher or equivalent.

9.5. Printer capable of printing results every 4 minutes.

9.6. Cooling Water recycler.

9.7. *Iteva* software.

9.8. Argon gas supply – Liquid Argon

9.9. Class A volumetric flasks

9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).

9.11. Variable Eppendorf Pipettes 1000µL; 5000µL

9.12. Disposable beakers 10, 20 and 50 mL size.

9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.4. Nitric acid (concentrated), HNO_3. A method blank is digested and analyzed before a new lot number of HNO3 is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.

10.5. Calibration standards

10.5.1. All standards have an acid matrix of 2% HNO_3 and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).

10.5.2. CAL1 is the calibration blank: Reagent grade water **matrix matched as in 10.5.1. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**

10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.

10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.

10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element**.**

10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.

10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO3 and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.

10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500

ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO3 are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1. Stock Multi-element Spiking Solutions: High Purity <u>CLP-CAL-1 solution A</u>: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; <u>solution B</u>: 250 ug/mL Ag; <u>CLP-CAL-2</u>: 1000 ug/L Sb; <u>CLP-CAL-3</u>: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low

level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:

2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL . Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:

0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO_3 prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods 19th Edition Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO3, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1**. Daily run and batch QC**

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within ±5% for 200.7 and within ±10% for all other methods.

12.1.4. ICB/CCB less than two times ± MDL or less than ± LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within ±20% initially and client specific requirement of ±30% at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times ±MDL or less than ±LOD unless verified standard contamination for DOD. The IFB must recover within ±20% for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an "Q" on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within ±10% or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times ±MDL or <±LOD for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75 to 125% for 6010B or 80 to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-10% of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

12.3.1. All quality control data should be maintained and available for easy reference or inspection.

12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than ± ½ RL for DOD or ±RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.

12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are $\pm$15% method 200.7 - aqueous and soil samples or $\pm$20% for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are >5x RL) or ± the RL (if either are <5X RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.

12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project

specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within ±20% of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply "J" flag for DOD if acceptance criteria are not met. Apply "N" flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO_3 / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.

13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV (± 10%) for 200.7 (± 5%) and ICB (< ±2xMDL, <±LOD-DOD or ±RL/CRDL for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within ±20% for DOD projects and ±30% for samples analyzed for 6010C. Then reanalyze the

highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±20%. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes < ±2xMDL or <±LOD for DOD beginning of sequence**.** Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be <LOD (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within ±20% of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

1) Cal Std 1 (blank)
2) Cal Std 2 (Low Cal)
3) Cal Std 3 (Mid Cal)
4) Cal Std 4 (Ba @ 5000 ppb)
5) Cal Std 5 (QC5)
6) Cal Std 6 (QC 21)
7) Cal Std 7 (NAK 100)
8) Cal Std 8 (QC3)
9) Cal Std 9 (Ag)
10) Al IEC-(correction using ITEVA software)
11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

1) SEQ-ICV
2) SEQ-ICB
3) SEQ-CRL1-reporting limit standard 1
4) SEQ-CRL2-reporting limit standard 2
5) Ba@ 5000 ppb (readback)
6) QC5
7) NAK High-(readback)
8) QC 21 High-(readback)
9) Salt Cal at 500 ppm (readback)
10) Rinse
11) SEQ-IFA1
12) SEQ-IFB1
13) Rinse
14) SEQ-CCV
15) SEQ-CCB
16) Method Blank (*Batch #* -BLK1)
17) Blank Spike (*Batch #* -BS1)
18) Sample 1
19) Sample 2
20) Sample 3
21) Sample 4
22) Sample 5
23) Sample 6
24) Sample 7
25) Sample 8
26) Sample 9
27) Sample 10
28) SEQ-CCV
29) SEQ-CCB
30) Sample 11
31) Sample 12
32) Sample 13
33) Sample 14
34) Sample 15
35) Sample 16
36) Sample 17
37) Sample 18
38) Sample 19
39) Sample 20
40) Sample matrix spike (*batch#*- MS1)
41) Sample matrix spike duplicate (*batch#* -MSD1)
42) Sample post digestion spike (*batch#* -PS1)
43) Sample serial dilution (*batch#* -DUP1)
44) SEQ-CCV

45) SEQ-CCB
46) Preparation Blank (*batch#* -BLK1)
47) Blank Spike (*batch#* -BS1)
48) Sample 1
49) Sample 2
50) Sample 3
51) Sample 4
52) Sample 5
53) Sample 6
54) Sample 7
55) Sample 8
56) Sample 9
57) Sample10
58) SEQ-CCV
59) SEQ-CCB
60) Sample 11

RACK 2

1) Sample 12
2) Sample 13

Etcetera…

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

14.1. Once the instrument has been calibrated, begin the analysis of samples.

14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**

14.3. Flush the system with 2% HNO_3 / 5% HCl for at least 1 minute before the analysis of each sample.

14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm$ 10% of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**

14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

14.5.1. The results of the CCV are to agree within ±10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be < 2x ±MDL, for CLP <RL, for **DOD no analytes detected >±LOD**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 "General and commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$.

15.2. The instrument will generate data results in mg/L or µg/L (labeled appropriately). Each result represents an average of three individual readings per metal channel.

15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

SR (ug/g or mg/kg) = IR*DF*FED/SM

SR = Sample result
IR = Instrument result (µg/L)
DF = Dilution factor (post digestion)
FED = Final volume of digestate (L)
SM = Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manuel is completed by each analyst and then provided to the supervisor for further processing and approval.
DOC LCS Preparation: See BS preparation under 10.7.1 through10.7.3 above.
DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within ±50%.

17. Pollution Prevention:
Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data
Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

19.1.1. ICV not within ± 10% or ± 5% for 200.7
a. Is the problem with the solution?
i. Re-prepare or obtain new stock.

b. Is the problem with the calibration?
   i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**

a. Is the problem with the solution?
   i. Re-prepare
b. Is the problem with the calibration?
   i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards not within ± 5%
a. Is the problem with the solution?
   i. Re-pour, re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. CLP only-CRI not within ± 20% (Internal QC, only required for CLP work).
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes <±LOD.**
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. IFB not within ± 20%
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.7. CCV not within ± 10%
a. Is the problem with the solution?
   i. Re-prepare or obtain new stock.
b. Is the problem with the calibration?
   i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.8.. CCB not ±2xMDL or CRDL for CLP, DOD no analytes detected >±LOD.
   a. Is the problem with the solution?
      i. Re-prepare
   b. Is the problem with the calibration?
      i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

19.2.1. Preparation blank (BLK) not within ± ½ RL and ± RL for common contaminants DOD or RL/CRDL for other or CLP
   a. Is the problem with the instrument?
      i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
   b. Is the problem with the digestion?
      i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.

19.2.2. BS not within control limits
   a. Is the problem with the instrument?
      i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
   b. Is the problem with the digestion?
      i. If biased low, associated samples must be re-digested.
      ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

19.3.1. Replicate analysis RPD not within ±20% (if both are >5X CRDL) or ± the CRDL (if either are <5X CRDL).
   a. The associated sample data must be qualified on the final report.

19.3.2. Spike analysis recovery not within ±20%.
   a. Is the analyte level in the sample greater than 4X the spiking level?
      i. If yes, the spike recovery is not evaluated.
      ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

19.3.3. When required, post digestion spike analysis recovery not within ±25% for SW6010B, DOD or ± 20% SW6010C.
   a. The associated sample data must be qualified on the final report.
   b. For USACE analysis by MSA is required.

19.3.4. Serial dilution analysis percent difference not within ±10%
   a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

i. If no, the serial dilution data can not be evaluated.

iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. **Waste Management**

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 6010B and Method 6010C.

21.2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 200.7; APX-B.

21.3. USEPA *Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.1. (Based on NELAC Voted Revision June 5, 2003. 4/22/09

22. **Tables, Diagrams, Flowcharts and Validation Data**

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L

Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
Interference Check	• once per calibration	• IFA less than LOD if not verified contamination of standard. IFB must be within ±20%.	• Check IEC corrections for metals in the IFA.
Calibration Curve	• Prior to analyzing any samples • A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. • Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C).	• Linear calibration Corr. of 0.998 • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICB	At the beginning of every sequence	Must meet the <±LOD for DOD or < 2xMDL	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	• Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
Closing CCV	• At the end of every sequence	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
BLK	One per prep batch	• Must be less than ½ ±RL.	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	• Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy	• Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):
Batch Number(s):
Method: 6010B or 6010C (ICP)

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did BS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

Comments on any "No" response:

Analyst: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 164 REVISION #: 15 EFFECTIVE DATE: 20100325

DISTILLATION OF AQUEOUS/SOLID SAMPLES FOR CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

APPROVALS:

Lab Director: _____ Date: 3/30/10

Data Quality Manager: _____ Date: 3/30/10

Section Supervisor: _____ Date: 3/30/10

Changes Summary:

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness. No changes have been made.

DISTILLATION OF CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

I. SCOPE AND APPLICATION

A. This method is applicable to the distillation of cyanide from drinking, surface, and saline waters, domestic and industrial wastes, and soil/sediments.

B. The limit of detection for waters is 0.0050 mg/L and the limit of quantitation is 0.010 mg/L. The limit of detection for soils is 0.13 mg/kg and the limit of quantitation is 0.25 mg/kg.

II. SUMMARY OF METHOD

A. The cyanide as hydrocyanic acid is released from cyanide complexes by means of a reflux distillation operation, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

B. Cyanide is defined as cyanide ion (CN^-) and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

III. SAMPLE HANDLING AND PRESERVATION

A. The sample should be collected in plastic or glass bottles of 100 mLs. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.

B. Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.06 g of ascorbic acid for each liter of sample volume.

C. Samples (not including soils/sediments) must be preserved with 2 mL of ten normal sodium hydroxide per liter of sample (pH $\geq$12) at time of collection.

D. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator to maintain temperature at 4°C.

E. The holding time for cyanide is 14 days counting the day of sampling as the first day. This applies for soils as well as waters. Samples must be distilled and analyzed within 14 days of sampling.

IV. INTERFERENCES

A. Interferences are eliminated or reduced by using the distillation procedure.

B. Sulfides adversely affect the colorimetric procedures. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation, must be treated by adding lead acetate, or if the sulfide concentration is too high, add powdered lead carbonate [$Pb(CO_3)_2$] to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Filter sample before raising pH for stabilization.

C. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. Nitrate and nitrite interference is eliminated by adding 0.5 ml of sulfamic acid solution (VII.A.7) before distillation.

D. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser.

V. SAFETY

A. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions.**

B. If an alkaline solution containing simple cyanide is acidified, the simple cyanide will be released.

C. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

D. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

VI. EQUIPMENT/APPARATUS

A. Reflux distillation apparatus is a midi-distillation apparatus which uses Hammett Scientific Glassware Note: This glassware is made to fit our specific block digester for cyanide. Glassware orders must be made in advance since they are not made until ordered.

VII. REAGENTS AND STANDARDS PREPARATION

A. Reagents

1. **Sodium Hydroxide, 0.25N**: Dissolve 10 g grams of NaOH in DI water and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

2. **Sulfuric Acid 1:1:** Slowly add 500 mL of concentrated H_2SO_4 to 500 mL DI water. Use caution because solution will become extremely hot!! Allow to cool while continuing to stir. Store in a labeled container at room temperature.

4. **Magnesium Chloride Solution**: Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1 liter flask; dissolve and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

5. **Chlorine Bleach Made with Sodium Hypochlorite**, such as Chlorox.

6. **Rhodanine Indicator**:
   Dissolve 20 mg of p-dimethyl-amino-bensalrhodanine in 100 mL acetone.

7. **Sulfamic Acid:** Dissolve 40 g of sulfamic acid in DI water, dilute to 100 mL (saturated solution). **NOTE:** When making this reagent for use with the maxi-distillation system the 40 g of sulfamic are diluted to one liter with DI water

8. **Phenolphthalein solution:** Commercially prepared.

9. **Lead Acetate Test Paper**: Commercially prepared.

10. **Acetate Buffer Solution, pH 4.0:** Dissolve 146g anhydrous $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2.3H_2O$, in 400 mL distilled water, add 480 g conc. acetic acid, and dilute to 1 L with chlorine-demand-free water.

11. **Lead Acetate**

12. **Powdered Lead Carbonate [$Pb(CO_3)_2$]**

B. Standards

1. Traceability

a. All reference materials must be recorded in Element and given an element identification number. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in Element as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number from Element with complete information on preparation date, concentration of

each compound, solvent, preparer's name, expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded on the bench sheet. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

2. **Preparation**

a. Stock Cyanide Solution at 1000 mg/L: Commercially prepared.

b. Working Standard Cyanide Solution: Prepare fresh daily by diluting 1 mL of stock cyanide solution to 100 mL in a volumetric flask with using 0.25N NaOH as diluent. 1 mL - 10 µg CN. Record the preparation in Element.

c. Performance Evaluation (PE) samples which we have analyzed and received true values for are used for the Laboratory Control Samples. These PE samples are given a unique identifier and all information such as expiration date (typically one year from the date made), source, concentration and preservation are recorded in Element and the Element # assigned is used to label this PE sample and is used on all data to trace it back to its source and true concentration.

VIII. CALIBRATION

A. Not applicable. See SOP-175 "**POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT"** for analysis.

IX. PROCEDURE

A. Pretreatment for cyanides amenable to chlorination

1. Two sample aliquots are required to determine cyanides amenable to chlorination. Place one 25 mL aliquot or a volume diluted to 25 mL, in a 100 mL beaker, under the hood, add a stirring bar, and place on a magnetic stirrer, and start stirring bar turning.

2. Check pH and adjust to between 11 and 12 units, with 1.25N NaOH. Use NaOH pellets if solution is highly buffered.

3. Add sodium hypochlorite chlorine bleach (Chlorox) solution dropwise until a drop of well-mixed sample turns KI starch paper a distinct blue color. Add about 10 drops excess bleach. (Caution: the initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride.) Maintain this excess for one hour, continuing agitation. Check periodically during the hour (15 minute intervals) to make sure an excess is maintained. If necessary, add additional hypochlorite bleach solution. Also check the pH and adjust back up to 11 to 12 units if necessary.

4. After one hour, add 0.025 g portions of ascorbic acid until KI starch paper shows no residual chlorine. Add an additional 0.025 g of ascorbic acid to ensure the presence of excess reducing agent.

5. Turn off stirring plate and remove stirring bar. Proceed with distillation as in the procedure.

6. Test for total cyanide in both the chlorinated and unchlorinated aliquots. (The difference of total cyanide in the chlorinated and unchlorinated aliquots is the cyanide amenable to chlorination.)

B. Spikes and high and low check standards

1. **Spikes**: Matrix spikes are prepared by diluting 2.5 mL of the High DCV solution in #3 below, to 25 mL with sample (adding 22.5 mL of sample and 2.5 mL of DCV high to the distillation tube). The concentration of this spike is 0.10 mg/L.

2. **Low Distilled Check standard (low DCV).** Dilute 2.0 mL of the 10 mg/L working standard to 100 mLs in a volumetric flask. Then dilute 6.0 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.048 mg/L standard.

3. **High Distilled Check standard (high DCV).** Dilute 10 mLs of the 10 mg/L working standard to 100 mLs in a volumetric flask with DI water. Then dilute 5 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.20 mg/L standard.

C. Distillation

1. For Waters: Place 25 mL of sample, or an aliquot diluted to 25 mL, in a sample tube. 0.25 N NaOH must be used for all sample dilutions following the distillation.

2. For Soils or Sludges:

   a.) It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

   - The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
   - Two quarters should then be mixed to form halves.
   - The two halves should be mixed to form a homogenous matrix.

   This procedure should be repeated several times until the sample is adequately mixed.

   NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a

representative sub-sample aliquot from the center or middle section of the sample container.

b.) Accurately weigh a representative 1-g minimum (record actual weight) portion of wet-weight sample (unless there are project-specific requirements where a lower detection limit is required) and transfer it to a sample tube. Add 25 mL of 0.25 N NaOH. Shake or stir the sample so that it is dispersed.

3. Add one drop of Phenolphthalein solution to verify preservation. A positive test is indicated by a bright pink color. If not add a small amount of NaOH.

4. To test for sulfide, place a drop of sample on lead acetate paper, previously moistened with acetic acid buffer solution (pH 4). A positive test is indicated by a black color on the paper.

   a.) If positive the sample will need to be filtered through 934AH filter paper. (4.5cm size) The filtrate will be treated and the filter added back to the treated filtrate before distillation.

   b.) Treat filtrate with powdered lead carbonate [$Pb(CO_3)_2$]. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper.

   c.) Filter the resulting precipitate (should form a lead sulfide which will be black in appearance).

   d.) Once the filtrate is treated and filtered, the filter with the original solids from the pretreated sample will be added back to the treated filtrate and then distilled.

5. Add 25 mL of sodium hydroxide solution 0.25N to the absorber and assemble the apparatus as shown in Fig. 1.

6. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum such that air bubbles from the thistle tube in the flask at a rate of 3 to 5 per second.

   Note: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally to prevent the solution in the sample tube from boiling and spewing out through the thistle tube.

7. Turn on condenser water.

8. Add 0.5 mLs of sulfamic acid through the thistle tube.

9. Slowly add 2 mL 1:1 H_2SO_4 through the thistle tube.

10. Add 2 mL 2.5 M $MgCl_2$ through the thistle and rinse it down with deionized water.

11. Reflux at 100°C for 90 minutes, turn off heat, allow to cool with pump on for 15 minutes.

12. Disconnect absorber and transfer the solution from the absorber into a storage container.

14. Record distillation information in the distillation log. Include date, analyst, sample number, client, matrix, position, sample mass / volume, distillation time, cool down time, when transferred to analyst, when analyzed.

X. CALCULATIONS

A. Sample volumes / masses must be recorded accurately in the distillation log to allow the analyst to correctly calculate the final concentration of cyanide correctly.

XI. DISTILLATION GLASSWARE CLEANING & LABELING

A. The distillation glassware is cleaned thoroughly with hot soapy water between distillations. 1:1 HCl is aerated through the frits on the block to ensure that the frits are clean. Deionized water is forced up through the glassware to rinse it. The cold fingers are dipped in mild acid, rinsed and wiped off.

B. All glassware must be labeled and each position's label must be recorded in the distillation log with the sample that was distilled in that particular vessel.

C. When sample concentrations are at or exceed 1000 mg/L a deionized water blank must be distilled in the exact glassware used for that sample before processing any other client's sample to ensure that the glassware is cleaned and has no residual cyanide to carryover.

XII. QUALITY CONTROL

A. With each distillation batch of samples (maximum of 20 samples) a preparation blank (BLK) must be distilled.

B. Each distillation batch of aqueous samples **requires a laboratory control sample (BS) each day or per 20 samples whichever is more frequent.** Under CLP the BS serves as the distilled ICV.

C. With each distillation batch of soil samples a soil BS (boiling chips are used) **each day or per 20 samples whichever is more frequent.**

D. A high and low check standard (DCV/ICV) must be distilled each day or per 20 samples whichever is more frequent. A non-distilled check standard must be prepared daily. This will be used to verify the preparation of the DCV if the DCV results are outside the specification limits. Under CLP the aqueous BS serves as the ICV.

E. A sample spike (MS) and sample spike duplicate (MSD) are performed with each set of twenty samples. CLP requires 1 duplicate and 1 spike per batch. All QC samples should be taken through the whole procedure including distillation. Spikes are added to the boiling flask in the same manner as standards adding 25 mL of samples or a dilution as used for the unspiked

sample. Spike concentration is listed in the method. The addition of the spiking solution must be witnessed and such must be noted in the digestion log.

XIII. CORRECTIVE ACTION

A. If any problems, such as low or high recovery for check standards or BSs occur, contact the Group Leader. Problems most frequently encountered (for the distillation procedure) are listed below.

B. Recoveries are affected in various ways.

1. If the spiking solution is not prepared properly (e.g., making the proper dilutions), the check standard will be out of control. Caution should be used in calculating the dilution and performing the pipetting.

2. Also, the air flow from the vacuum pumps can cause low recovery. Pay attention to the air flow (it should be in the area of 1 to 2 bubbles per second). If it is too great the HCN does not have an adequate amount of time to be absorbed by the NaOH solution.

3. If the heating block is not heating properly, this will also lead to low recovery. During the distillation, periodically check the sample tubes for the presence of heat (tubes should be very hot to the touch).

4. The addition of all reagents, including the spiking solution, is essential. Without the $MgCl_2$, 1:1 H_2SO_4, or spiking solution, the percent recovery will suffer greatly for complex cyanide. The recovery of simple cyanides will usually be unaffected.

5. If the volume of 0.25 N NaOH and reagent water is not accurate in the NaOH absorber tubes, low recoveries will usually occur.

B. Spike recoveries are affected in similar ways. Refer to the above suggestions. Also, if possible, do not spike a sample that is known to be high in cyanide. This may cause recovery and calculation problems.

XIV. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.

2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is $\geq$ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

XV. DEFINITIONS

See SOP-431 for a list of definitions

EMPIRICAL LABS, LLC.
Record of SOP Review and Implementation

TRAINING TOPIC SOP164 Rev 15 - Distillation of Aqueous/Solid Samples for Total and Non-Amenable Cyanide Analysis Methods 335.1/335.4/ Standard Methods SM 4500-CN C,G 18th, 19th ED./ (SW846) 9012A/USEPA CLP ILMO 4.1

Group: Inorganic

ATTENDEES: NAMES (print)	SIGNATURE	REMARK	DATE	TIME	INSTRUCTOR
1 Veronica Mullen	Veronica Mullen		8-3020	1118	
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EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP175 **REVISION #: 11** **EFFECTIVE DATE: 20100907**

POST-DISTILLATION ANALYSIS FOR CYANIDE USING LACHAT FLOW INJECTION ANALYZER METHODS 335.4;(SW846) 9012A, Standard Methods 21st Edition USEPA-CLP 4.1, (NJDEP does not accept CLPILM 04.1 after June, 2003) Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 11, 09/07/10

- The SOP is an update from Revision 10 dated 12/15/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1.0 Identification of the Test Method

1.1 This SOP is compliant with EPA method 335.4 and SW9012A/B.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to the determination of Cyanide in Aqueous and Soil / Sediment matrices.

3.0 Detection Limit

3.1 All limits reported for various programs are listed in Table 1 of this SOP.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte.

5.0 Summary of the Test Method

5.1 Cyanide from alkaline distillates is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH lower than 8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The color is read at 570 nm.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides are removed by treatment with lead acetate or powdered lead carbonate. See Empirical Laboratories Method SOP-164 for distillation procedure.

Note: method 9012A/B says that sulfides "should" be treated with bismuth nitrate. Empirical Laboratories attempted to use that procedure and found that bismuth nitrate also removes cyanide from standard solutions as well. This sulfide treatment is not used. See Empirical Laboratories Method SOP-164 for distillation procedure.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment & Supplies

9.1 Lachat Quick Chem AE Automated Ion Analyzer
9.2 Cyanide manifold (Lachat)
9.3 Instrument Information

Analyst should confirm the following:

A. Timing:

Sample throughput: 80 samples/h; 40 s/sample
Pump speed: 35 RPM

Cycle Period: 45 s
Inject to start of peak period: 28 s
Inject to end of peak period: 61 s

B. QuikChem AE Settings:

1) Parameter, Data Window:

Top Scale Response: 0.50 abs
Bottom Scale Response: 0.00 abs

2) Segment/Boundaries

A - 0.50 mg CN/L
F - 0 mg CN/L

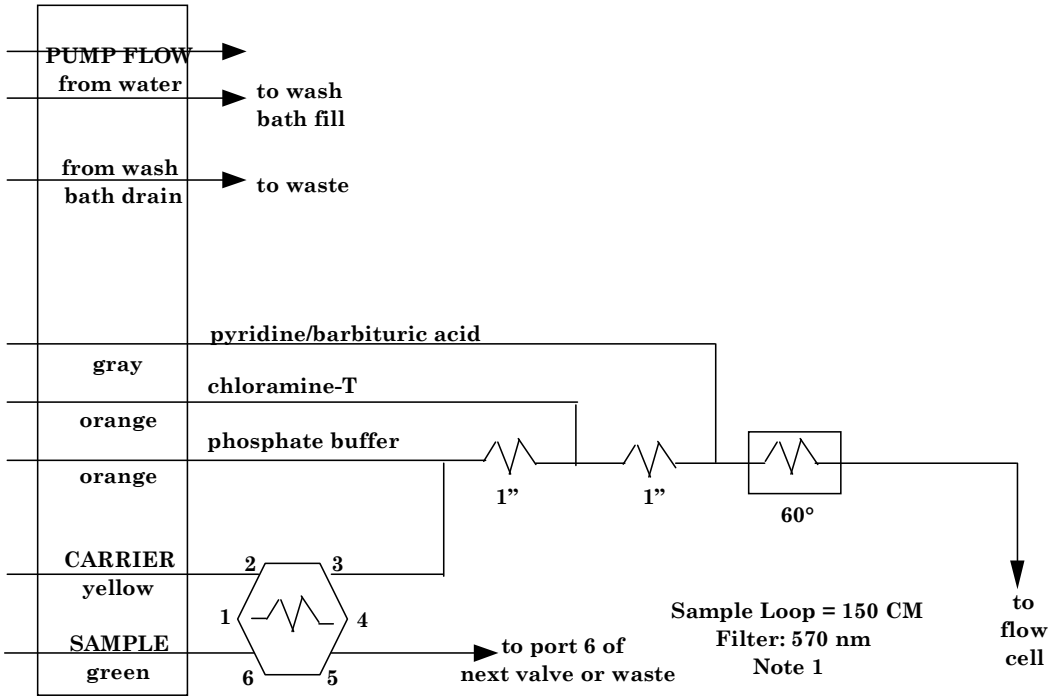
3) Results/Approval, Reports

In the default Report definition file (RDF), change:
-Set Default Chord 0 to
-Set Default Chord 3
(Peak should be centered in chord 3)
This change must be made to both the sample and the calibration RDF's.

C. System Notes:

1. Allow enough time for heating unit to warm¬ up to 60°C with D.I. water pumping through all reagent lines at full speed. (approximately 30 min.)

MANIFOLD DIAGRAM



CARRIER is 0.25 M Sodium Hydroxide

1" is 70.0 cm of tubing on a 1 in coil support

Note1: A 3.0 m x 0.022" id back pressure loop is placed at the exit of the flow cell.

Manifold Diagram Revision Date: 13 Ma

APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650 cm of heated tubing.

All manifold tubing is 0.8 mm (0.032 in) i.d., This is 5.2 µL/cm.

10.0 Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4 ° C.

10.2 Use deionized water (<10 megaohm) for all solutions.

10.3 **Reagent 1. Carrier, 0.25 M Sodium Hydroxide:** In a 1L vol. flask add 200 mL of 1.25N NaOH (which is used in the SOP 164 distillation method) to approximately 600 mL of D.I. water. Dilute to 1 L, invert to mix. This will ensure that the concentration of the NaOH used for the distillates will be the same concentration used for the carrier solution.

10.4 **Reagent 2. Phosphate Buffer, 0.71 M**: In a 1 L volumetric flask, dissolve 97 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous, KH2PO4) in approximately 800 mL water. Dilute to the mark and invert to mix. Prepare fresh monthly.

10.5 **Reagent 3. Chloramine-T Hydrate:** To a 500 mL volumetric flask add about 250 mL water, then add 2.0 g chloramine-T [CH3C6H4SO2N(Cl)Na x H2O]. Dilute to the mark and invert to mix. Prepare fresh daily. The shelf life of the chloramine-T can be a critical factor in the Lachat run. New Chloramine-T should be ordered every 6 months. (Analyst discretion should be used).

10.6 **Reagent 4. Pyridine-Barbituric Acid Reagent:** In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100.0 mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine (C5H5N) with stirring and mix until the barbituric acid dissolves. Add 15 mL concentrated hydrochloric acid (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert to mix. This reagent is stable for approximately six months if stored in a cool, dark place.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 The color reaction is pH sensitive. Therefore, distillates and standards should be carefully matched with respect to NaOH concentration.

11.3 The distillates are 0.25 N NaOH.

12.0 Quality Control

12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.2 An initial demonstration must be performed by each analyst performing this method. Four LCSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QA/QC, distilled:

12.3.1 PB (preparation blank):

25 mL of DI water is distilled. When brought up to 25 mL volume following the distillation process a 0.25 M NaOH solution is obtained. A blank is distilled every batch of samples. If the absolute value of the PBW is not below the CRDL or the RL or ½ the CRDL for Navy Projects the analyst's supervisor should be consulted before proceeding.

12.3.2 DCV (distilled calibration verification)/ICV (distilled/CLP)

a. The DCV high and low are check standards generally of a concentration of about 0.200 mg/L for the high and 0.050 mg/L for the low. A DCV high and low are distilled with every batch of distilled samples. There is a maximum of 20 samples in a batch. If the DCV is not within +15 % of the

undistilled value the analyst's supervisor should be consulted before proceeding. A NCR will be required if the limit is exceeded.

b. CLP requires the ICV to be distilled and from a second source. Must be analyzed immediately after calibration. Since the matrix for the soil LCS is Teflon chips the LCS serves as a distilled ICV. The control limit is + 15%.

12.3.3 Blank Spike (BS or laboratory control sample)

The laboratory control sample is a second source check on the calibration and must be run once every 20 aqueous samples. The control limits are 80 to 120%. If the BS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log.

12.3.4 BS (BS or laboratory control sample-**soil)**

The LCSS is a second source check with a soil matrix (Teflon chips are used for this purpose) and must be run once every 20 solid phase samples. If the BS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in Element.

12.3.5 MS/MSD (matrix spike/matrix spike duplicate)

A spike and spike duplicate (CLP requires 1 duplicate and 1 matrix spike) are done with each batch or 20 samples. The spike concentration is 0.10 mg/L. The analyst's supervisor should be consulted before proceeding if the percent recovery for the MS or MSD is not within ±20 percent of the spike concentration,.

12.3.6 Documentation of Capability (DOC) - Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS05 for guidance.

12.4 QA/QC, undistilled:

12.4.1 ICV (initial calibration verification)

The ICV is an undistilled second source check standard (except when analyzing by CLP methods and then the ICV is distilled) made from a second source working standard solution. The concentration is usually around the midpoint of the calibration curve (about 0.500 mg/L). The ICV is analyzed at the beginning of each Lachat run. The analyst's supervisor should be consulted before proceeding, if the ICV is not within ±10 percent of its true value. This calibration verification standard must be prepared fresh daily.

12.4.2 ICB (initial calibration blank)

The ICB is 0.25 M NaOH solution and follows the ICV at the beginning of each Lachat run. If the absolute value of the ICB is not <± 2x MDL or < + CRDL or ½ the RL for Navy Projects, the analyst's supervisor should be consulted before proceeding.

12.4.3 CCV (continuing calibration verification) from primary std. that curve is made from.

The CCV will be analyzed after every 10 samples and at the end of the tray. The sampling of the CCV's will be done automatically by the Lachat if it is included in the tray definition. The CCV scheduling can be done under the Data Quality Management option in the Tray Definition screen. If the CCV is not within +10 percent of its true value a CHK STD failure signal will be given by the Lachat and the analyst's supervisor should be consulted before proceeding.

12.4.4 CCB (continuing calibration blank)

This sample verifies the instrumental baseline and must be analyzed immediately after every ICV/CCV. If the absolute value of the ICB/CCB is not below the LOD the analyst's supervisor should be consulted before proceeding.

12.5 Calibration Approval:

12.5.1 For a cyanide calibration to be approved, segment (A-F) of the curve must have a correlation of at least 0.995 in chord 3.

12.5.2 Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength: (MDLS are performed annually to meet specific state requirements or whenever a change in the method is made.)

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Traceability: All reference materials must be assigned Element # and labeled accordingly. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded in the batch sheet in Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

13.3 Standard 1.

1000 mg CN/L [~1,000 mg CN-/L (Stock)]: The Stock Standard is purchased from a vendor. CAUTION: KCN is highly toxic. Avoid contact with standard solutions. Use gloves and protective equipment when handling standards and samples. This solution is given a unique identifier. Record preparation in Element and label standards with appropriate Element #'s.

13.4 Standard 2. Working Standard (10.0 mg CN/L)

Pipette 2.00 mL Standard 1 into a 200 mL volumetric flask. Dilute to the mark with Reagent 1, 0.25 M Sodium Hydroxide. Invert to mix.

13.5 Set of six calibration Standards:

10 mL of 10 mg/L diluted to 100 mL = 1.0 mg/L std
5.0 mL of 10 mg/L diluted to 100 mL = 0.500 mg/L std
2.0 mL of 10 mg/L std diluted to 100 mL = 0.200 mg/L std
1.0 mL of 10 mg/L std diluted to 100 mL = 0.100 mg/L std
4 mL of 0.500 mg/L std diluted to 100 mL = 0.020 mg/L std
2 mL of 0.500 mg/L std diluted to 100 mL = 0.010 mg/L std
Blank consists of 0.25 M NaOH
10 mL of 10.0 mg/L std diluted to 200 mL = 0.500 mg/L chk std

13.6 The diluent for all standards is 0.25 M NaOH.

14.0 Procedure

14.1 The samples for analysis come directly from the midi scale distillation procedure.
14.2 See "Equipment and Supplies" for how to set up the manifold for use for cyanide analysis.
14.3 Be sure to read through all components of this SOP for procedural instructions.

14.4

ANALYTE	WAVELENGTH
Cyanide	570.0

Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. The ICV shall be distilled.

2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is ≥ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Aqueous Samples: The Lachat reports the concentration of the final distillate in mg/L. The method detection limit (MDL) is 0.005 mg/L and the reporting limit (RL) is 0.010 mg/L.

15.3 Solid Samples: The Lachat reports the concentration of the final distillate in mg/L. Results for solid samples should be in mg/kg and can be obtained by entering the appropriate dilution factor when defining the tray. Conversion from mg/L to mg/kg can be accounted for by the following formula:

From this formula a dilution factor can be obtained. For example if 2 grams of soil were used then this would be a x 250 dilution. When the x 0.50 correction factor is included a final manual dilution factor of x 125 is obtained.

N would be the value obtained from the Lachat without a correction factor. The minimum value for N is 0.01 mg/L soln.

15.4 Data reporting

1. Reduce data to the result which will be reported.
2. Complete the data review checklist (attached). This must be completed and attached to each set of USACE data.

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

16.2 The IDOC is analyzed at 4 times the LOQ or 0.080 mg/L for waters and 3.0 mg/kg. All standard results should be in the range of 80 to 120%. The continuing DOC is four LCS's analyzed and a completed precision and accuracy sheet with all LCS within the control limits of 80 to 120%.

16.3 LOD/LOQ are analyzed quarterly and an MDL study is performed annually for specific state requirements. The LOD must be three times the noise and is analyzed at 0.010 mg/L for waters and 0.25 mg/kg for soils. The LOQ must be in the range of 50 to 150% and is analyzed at a concentration of 0.020 mg/L for waters and 0.75 mg/kg for soils.

17.0 Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

18.2 **Instrument Related**

1. ICV not within ± 10%
    a. If the problem is with the solution.
        i. Re-prepare, obtain new stock if necessary.
    b. If the problem is with the calibration.
        i. Recalibrate thru analysis of appropriate standards and recheck ICV.
2. CCV not within ± 10%
    a. If the problem is with the solution.
        i. Re-prepare, obtain new stock if necessary.
    b. If the problem is with the calibration.
        i. Recalibrate thru analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.
            a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.
            b. If the CCV was biased low, the previous ten samples must be re-analysed.
3. Ending CCB not ± RL or CRDL **or ½ the CRDL for Navy Projects**
    a. If the CCB is biased high.
        i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.

ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.

b. If the CCB is biased low.

i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
ii. All other samples must be reanalyzed after the problem is corrected.

18.4 Distillation Related

1. The preparation blank is not less than the ± RL or CRDL **or ½ the CRDL for Navy Projects**.

a. If the problem with the instrument.

i. Analyze a CCB to determine this.
ii. If the problem was with the instrument correct the situation and reanalyze the preparation blank.

b. If the problem is with the distillation.

i. All associated samples which are <MDL or have a level of cyanide greater than 10X the level found in the preparation blank can be reported. If the level of cyanide in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redistilled/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits (±20%).

a. If the problem is with the instrument.

i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

3. Distilled check standard not within control limits of ±15%.

a. If the problem is with the instrument.

i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

18.5 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%

    i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±20%

    i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

    ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

19.0 Contingencies for Handling out-of-control or unacceptable data

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 **References**

21.1 Lachat manual; Quick Chem method 10-204-00-1-A.
21.2 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 335.4.
21.3 Standard Methods for the Examination of Water and Wastewater, 21st Edition, APHA-AWWA-WCPC, Park 413D, pp. 370-372.
21.4 SW-846, 9012A, Revision 1, December 1996.
21.5 SW-846, 9012B, Revision 2, November 2004.
21.6 U.S. Environmental Protection Agency, C.L.P. S.O.W. ILMO 4.1.
21.7 QuickChem AE, Automated Ion Analyzer Training Manual, Lachat Instruments.
21.8 QuickChem AE, Automated Ion Analyzer Software Reference Manual, Lachat Instruments.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, with the applicable RL and lowest calibration standard.
22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.
22.3 Table 3, Technical Completeness / Accuracy Checklist
22.4 Table 4, Data Reviewers Checklist
22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

Table 1
Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Analytes by EPA 335.1, 335.3, 9012A/B, SOW 4.1 & 5.2	AQUEOUS MDL(ug/L)	AQUEOUS ERL(ug/L) And lowest calibration standard	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/kg) And lowest calibration standard	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	OLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Cyanide	5.0	10	10	10	0.125	0.25	0.25	0.25

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • Low standard at the RL/LOD level	• For Linear fits a RF of 0.995 • Average RSD ≤ 20% • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must meet 85 to 115%	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• ± 10% difference	• Follow guidelines for SOP QS05
Closing CCV	• At the end of every sequence	• ± 10% difference	• Follow guidelines for SOP QS05
BLK/CCB	One per prep batch/ after every CCV	• Must be less than ½ RL/Must be less than the LOD	• Re-analysis to confirm the positive value • Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers • If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action • Re-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	• ±20% difference	• Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	• ±20% difference	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
MS	One per prep batch, if sample volume available.	• ±20% difference	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	• ±20% difference	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year for specific state requirements	• Calculated value must be greater than 10% of the Spike Level • Calculated value must be less than the Spike level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • 2nd column / detector confirmation is required • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**9012A/B (Cyanide)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?				
2. Was initial calibration curve QC criteria met?				
3. Was all continuing calibration criteria in control?				
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)				
5. Did LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was distillation temperature monitored/documented and did you apply the thermometer correction factor?				
10. Sample preparation information is correct and complete.				
11. Analytical results are correct and complete.				
12. The appropriate SOP's have been used and followed.				
14. "Raw data" including all manual integration's have been correctly interpreted.				
15. "Special" sample preparation and analytical requirements have been met.				
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.				

ANALYST DATA REVIEW CHECKLIST
9012A/B (Cyanide)

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 201 REVISION #: 20 EFFECTIVE DATE: 042610

GC/MS SEMIVOLATILES and LOW-CONCENTRATION PAHs BY EPA METHOD 625 AND SW846 METHOD 8270C AND 8270D INCLUDING ADDITIONAL APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: _____ Date: 4/27/10

Data Quality Manager: _____ Date: 4/27/10

Section Supervisor: _____ Date: 4/27/10

Changes Summary

Revision 20, 4/13/10

- The SOP is an update from Revision 19 dated 4/11/2010
- The SOP is formatted to simplify the text and place all method/program specifications in the SOP tables.

Revision 19, 4/11/10

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

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1.0 Identification of the Test Method

This SOP is based primarily on SW-846 Methods 8000B/8000C/8270C/8270D. Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This SOP is used for the analysis of semi-volatile organic compounds (including low concentration PAHs) in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits – Reporting Limits

See Table 1

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is routinely analyzed and reported under the scope of this SOP is listed in the Appendix of this SOP. This table also lists the associated Detection Limit, Limit of Detection and Reporting Limit (also defined as the Limit of Quanititation).

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

6.0 Definitions –

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

9.0 Equipment & Supplies

a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for split-less injection.

b Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column.

c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

g Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified times or scan-number limits.

h Micro syringes – gas tight 5uL and larger.

i Liners – 2mm or 4mm single goose-neck.

j Septa 11mm.

k Seals- dual vespel stainless steel or gold plated 0.8mm.

l Vials- 2ml and larger amber.

m Volumetric flasks- 10ml and larger class A with glass stopper.

10.0 Reagents and Standards –

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory.

10.2 Reagent grade chemicals shall be used in all tests unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.3 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.

10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature are monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook.

10.5 Individual standard makeup is recorded in LIMS with specific details concerning the standard being used, concentration, amount, solvent and expiration date.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

12.1 Internals - All samples and QC are spiked with internal standards prior to analysis.

12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.

12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Initial Calibration - An initial multi-point calibration curve must be analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve range from 1.0ug/mL to 100ug/mL for regular SVOCs and 0.1µg/mL to 50µg/mL for low-concentration PAHs.. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.3 Initial Calibration Verification (ICV) - A second source standard at the continuing calibration verification (CCV) level must be analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

13.4 Continuing Calibration Verification (CCV) - Every 12 hours, a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.0 Procedure

Prior to analysis the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, 3546 3550, 3580, EPA method 625 or CLP).

14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.

14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard. The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port . See **Table 2** for criteria and corrective action.

14.3 Extracts - Prior to analysis, 1.0 mL extracts are prepared by verifying volume and spiking with 20uL of the internal standard solution.

14.5 Instrument sequence-The instrument sequence log is filled out prior to sample analyses. An example of a typical instrument sequence log follows:

1-SEQ-TUN1 (12:00 am)
2-SEQ-CCV1
3-SEQ-BS1
4-SEQ-BLK1
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-SEQ-MS1
15-SEQ-MSD1
16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
17-SEQ-CCV2
18-Sample
19-Sample
20-Sample

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need reanalysis or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Internal Standard Area Counts and Retention Times

14.6.2 Surrogate Recoveries and Retention Times

14.6.3 Analyte concentration.
14.6.4 Analyte identification based on spectrum and retention time.
14.6.5 Analyte quantitation verification.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 The RF is calculated as follows:

$$RF = \frac{A_s \text{ x } C_{is}}{A_{is} \text{ x } C_s}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.
C_{is} = Concentration of the internal standard.

15.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where:
Calculated concentration is determined from the initial calibration.
Theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where:
CCV RF is the response factor from the analysis of the verification standard
Average RF is the average of the response factors from the initial calibration.

15.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:
A_s = Area (or height) of the peak for the analyte in the sample.
A_{is} = Area (or height) of the peak for the internal standard.
C_{is} = Concentration of the internal standard in the volume extracted in ug/L.
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s,

A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, Technical Director and/or Data Quality Manager.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Demonstration of Capability (DOC)
16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

40 CFR, Part 136; Appendix A
Test Methods for Evaluating Solid Waste, SW-846
National Environmental Laboratory Accreditation Conference; CH. 5, 2003
USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
DOD Quality Systems Manual for Environmental Laboratories,

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).
22.2 Table 2, QA/QC summary table
22.3 Table 3, Technical Completeness / Accuracy Checklist
22.4 Table 4, Data Reviewers Checklist(s)
22.5 Table 5, 625 QC Limits
22.6 Table 6, Standards Used
22.7 Table 7, INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis
22.8 Table 8, LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS
22.9 Figure 1, Tailing Factor Calculation
22.10 Table 9, DFTPP Tuning Criteria

TABLE 1

Analyte (Water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3´-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L

Table 1 (Continued)

Analyte (Water)	DL	LOD	MRL/LOQ	Units
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
Analyte (Soil)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3´-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg

Table 1 (Continued)

Analyte (Soil)	DL	LOD	MRL/LOQ	Units
Benzo (a) pyrene	83.3	167	333	ug/Kg
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg
Analyte Low PAH (Water)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1-Methylnaphthalene	0.0500	0.100	0.200	ug/L
2-Methylnaphthalene	0.0500	0.100	0.200	ug/L
Acenaphthene	0.0500	0.100	0.200	ug/L
Acenaphthylene	0.0500	0.100	0.200	ug/L
Anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) pyrene	0.0500	0.100	0.200	ug/L
Benzo (b) fluoranthene	0.0500	0.100	0.200	ug/L
Benzo (g,h,i) perylene	0.0500	0.100	0.200	ug/L
Benzo (k) fluoranthene	0.0500	0.100	0.200	ug/L
Chrysene	0.0500	0.100	0.200	ug/L
Dibenz (a,h) anthracene	0.0500	0.100	0.200	ug/L
Fluoranthene	0.0500	0.100	0.200	ug/L
Fluorene	0.0500	0.100	0.200	ug/L
Indeno (1,2,3-cd) pyrene	0.0500	0.100	0.200	ug/L
Naphthalene	0.0500	0.100	0.200	ug/L
Phenanthrene	0.0500	0.100	0.200	ug/L
Pyrene	0.0500	0.100	0.200	ug/L
Analyte Low PAH (Soil)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg

Table 1 (Continued)

Analyte Low PAH (Soil)	DL	LOD	MRL/LOQ	Units
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg
Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
Analyte (TCLP)	**DL**	**LOD**	**MRL/LOQ**	**Units**
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C of DoD QSM 4.1. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 8 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2. [Method 625 – benzidine and pentachlorophenol tailing limits are 3 and 5, respectively, when benzidine or acids are target analytes. Benzidine tailing is specific to benzidine analysis and pentachlorophenol tailing is specific to acid analyte analyses according to 625.]	Correct problem then repeat breakdown checks.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%. **Not applied when low concentration PAHs are the only target analytes.**

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	**1.** Average response factor (RF) for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs **2.** RSD for RFs for CCCs: SVOCs ≤ 30% and one option below: **Option 1:** RSD for each analyte ≤ 15%; [≤ 20% for non-DoD 8270D; or, ≤35% for non-DoD 625] **Option 2:** linear least squares regression r ≥ 0.995 or $r^2 \geq 0.990$; [r ≥ 0.990 for non-DoD analyses] **Option 3:** non-linear regression–coefficient of determination (COD) $r^2 \geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value [± 25% for non-DoD 8270C; or, ± 30% for non-DoD 8270D]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	**1.** Average RF for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs **2.** %Difference/Drift for all target compounds and surrogates: SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [± 20% for CCCs only non-DoD 8270C]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data should be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8270C, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem should be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL/LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL/LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. AFCEE 4.0.02 limits are applied for low concentration PAHs as they are not addressed by DoD. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. Low concentration PAH limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix	Use LCS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

<table>
<tr><th>QC Check</th><th>Minimum Frequency</th><th>Acceptance Criteria</th><th>Corrective Action</th><th>Flagging Criteria</th><th>Comments</th></tr>
<tr><td>Surrogate spike</td><td>All field and QC samples.</td><td><table><tr><th>Surrogate</th><th>Water</th><th>Solid</th></tr><tr><td>Nitrobenzene-d5</td><td>40-110</td><td>35-100</td></tr><tr><td>2-Fluorobiphenyl</td><td>50-110</td><td>45-105</td></tr><tr><td>Terphenyl-d14</td><td>50-135</td><td>30-125</td></tr><tr><td>Phenol-d6</td><td>10-115</td><td>40-100</td></tr><tr><td>2-Fluorophenol</td><td>20-110</td><td>35-105</td></tr><tr><td>2,4,6-Tribromophenol</td><td>40-125</td><td>35-125</td></tr></table>QC acceptance criteria specified by DoD (above) or Client. Low PAH surrogate limits are 14%-129% soil and 34%-167% water. Otherwise, in-house control limits may be used. No limits specified for Method 625.</td><td>For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.</td><td>Apply qualifier to all associated analytes if acceptance criteria are not met. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.</td><td></td></tr>
<tr><td>Results reported between DL and LOQ</td><td>NA.</td><td>NA.</td><td>NA.</td><td>Apply J-flag to all results between DL and LOQ.</td><td></td></tr>
</table>

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory or senior-scientist approval for manual integrations on standards and batch QC samples
7. Was the data uploaded into LIMS via direct upload (i.e. datatool) – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8260B/624/8270C/8270D/625 (Circle One)**

QA/QC Item	Yes	No	NA	Second Review	Level
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?					
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).					
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?					
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.					
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?					
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?					
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?					
8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?					

Comments on any "No" response:

_____

_____

_____

_____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 – 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC	#	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100		47-145
Acenaphthylene	100.00	0.0000	100.00	100		33-145
Anthracene	100.00	0.0000	100.00	100		27-133
Benzidine	100.00	0.0000	100.00	100		D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100		33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100		24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100		11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100		D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100		17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100		33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100		12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100		36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100		8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100		53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100		D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100		22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100		60-118
2-Chlorophenol	100.00	0.0000	100.00	100		23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100		25-158
Chrysene	100.00	0.0000	100.00	100		17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100		D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100		32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100		D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100		20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100		D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100		39-135
Diethylphthalate	100.00	0.0000	100.00	100		D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100		32-119
Dimethylphthalate	100.00	0.0000	100.00	100		D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100		1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100		D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100		D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100		39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100		50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100		4-146
Fluoranthene	100.00	0.0000	100.00	100		26-137
Fluorene	100.00	0.0000	100.00	100		59-121
Hexachlorobenzene	100.00	0.0000	100.00	100		D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100		24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100		15- 70
Hexachloroethane	100.00	0.0000	100.00	100		40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100		D-171
Isophorone	100.00	0.0000	100.00	100		21-196
Naphthalene	100.00	0.0000	100.00	100		21-133
Nitrobenzene	100.00	0.0000	100.00	100		35-180
2-Nitrophenol	100.00	0.0000	100.00	100		29-182
4-Nitrophenol	100.00	0.0000	100.00	100		D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100		29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100		23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100		D-230
Pentachlorophenol	100.00	0.0000	100.00	100		14-176
Phenanthrene	100.00	0.0000	100.00	100		54-120
Phenol	100.00	0.0000	100.00	100		5-112
Pyrene	100.00	0.0000	100.00	100		52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100		44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100		37-144

Table 6 - BNA STANDARDS USED

base/neutral mix (2000ppm)		**acids mix (2000ppm)**
bis(2-Chloroethyl)ether		2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether		2-Methylphenol
1,3-Dichlorobenzene		4-Methylphenol
1,2-Dichlorobenzene		Benzoic acid
1,4-Dichlorobenzene		4,6-Dinitro-2-methylphenol
Hexachloroethane		4-Nitrophenol
N-Nitroso-di-methylamine		2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine		2,4,6-Trichlorophenol
2,4-Dinitrotoluene		Phenol
2,6-Dinitrotoluene		Pentachlorophenol
Fluorene		2-Nitrophenol
Dimethylphthalate		4-Chloro-3-methylphenol
Hexachlorocyclopentadiene		2,4-Dichlorophenol
Anthracene		2,4-Dimethylphenol
4-Bromophenyl-phenylether		Benzoic acid
Di-n-butylphthalate		
bis(2-Chloroethoxy)methane		
1,2-Diphenylhydrazine		**semivoa misc. mix(2000ppm)**
Fluoranthene		Aniline
Hexachlorobenzene		Benzyl alcohol
N-Nitrosodiphenylamine		Carbazole
Phenanthrene		4-Chloroaniline
Hexachlorobutadiene		Dibenzofuran
Isophorone		2-Methylnaphthalene
Naphthalene		2-Nitroaniline
Nitrobenzene		3-Nitroaniline
1,2,4-Trichlorobenzene		4-Nitroaniline
Acenaphthene		Pyridine
Acenaphthylene		
2-Chloronaphthalene		
4-Chlorophenyl-phenylether		**Benzidine mix (2000ppm)**
Diethylphthalate		Benzidine
Benzo(a)anthracene		3,3'-Dichlorobenzidine
Bis(2-ethylhexyl)phthalate		
Butylbenzylphthalate		
Chrysene		**Individual or misc. mixes (2000/5000/20,000ppm)**
p-(Dimethylamino)azobenzene		Caprolactam
Pyrene		Benzaldehyde
Benzo(b)fluoranthene		Atrazine
Benzo(k)fluoranthene		1,1'-Biphenyl
Benzo(g,h,i)perylene		1,4-Dioxane
Benzo(a)pyrene		1-methylnapthalene
Dibenz(a,h)anthracene		2,6-dichlorophenol
Di-n-octylphthalate		2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene		

BNA internals (2000ppm)		Acid surrogate (7500ppm)
1,4-Dichlorobenzene-d4 (I.S)(1)		2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)		Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)		2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)		2,-Chlorophenol-d4 (S)
Chrysene-d12 (I.S) (92))		BN surrogate (5000ppm)
Perylene-d12 (I.S) (101)		Nitrobenzene-d5 (S)
		Terphenyl-d14 (S)
		2-Fluorobiphenyl (S)
		1,2-Dichlorobenzene-d4 (S)

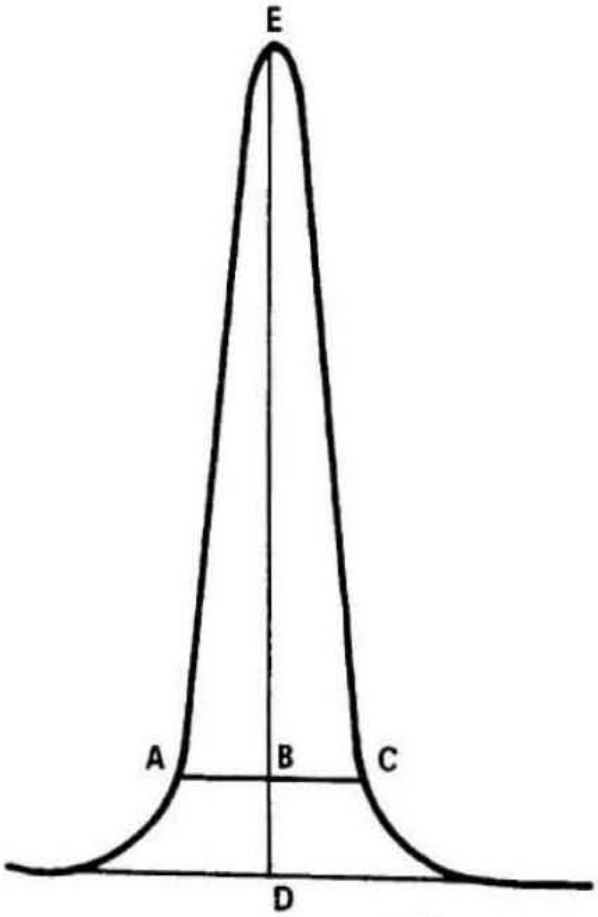
Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis					
COMPOUND	***I.S**	**Q.M**	**COMPOUND**	***I.S**	**Q.M**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 **(I.S) (79)**		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 **(I.S)(35)**		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 **(I.S) (92)**		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 **(I.S) (59)**		164	Perylene-d12 **(I.S) (101)**		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276
I.S=internal standard, Q.M=quant mass, S=surrogate					

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis (contd)					
COMPOUND	***I.S**	**Q.M**	**COMPOUND**	***I.S**	**Q.M**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
0-Toluidine	1	106		59	
	1		Phenanthrene-d10 **(I.S) (79)**		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 **(I.S)(35)**		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 **(I.S) (92)**		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 **(I.S) (59)**		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 **(I.S) (101)**	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	
I.S=internal standard, Q.M=quant mass, S=surrogate					

Table 8: LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

INTERNAL STD ASSOCIATION
Phenanthrene-d10 (IS)
Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
2-Fluorobiphenyl(SUR)
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Perylene-d12 (IS)
Terphenyl-d14(SUR)
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Table 9, DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note: While 8270D table 3 indicates different criteria, section 11.3.1.2 allows the use of alternate criteria.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 202 **REVISION #: 23** **EFFECTIVE DATE: 20100909**

GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B INCLUDING APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: _____ Date: 9/9/10

Data Quality Manager: _____ Date: 9/9/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 23, 09/09/10

- This SOP is an update from Revision 22 dated 09/30/09.
- Tables 1 and 2 have been updated with appropriate reference updates.
- Tables 5-7 have been added.

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. Detection Limit: See **Table 1** of this SOP.

4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. Definitions

6.1 Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9. Equipment & Supplies

9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.

9.3 M.S.: HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]

9.4 Purge and Trap Unit

9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.

9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

9.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.

9.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000 µL.

9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.

9.10 Disposable pasteur pipets.

9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

9.12 Spatula - stainless steel.

9.13 Glass scintillation vials - 20mL with screw caps.

9.14 Nitrile Gloves

9.15 pH paper (measures pH from 0-14).

10. Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

10.2 Organic-free reagent water - obtained from the charcoal filter system in the VOA laboratory.

10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)

10.5 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C or less from the date they are received/prepared. The freezer temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C $\pm$ 5°C or less for up to 6 months **(1 week for South Carolina samples)**. A direct injection of 1µL (or equivalent purge) is used to tune the instrument.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor,Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. (The internal standard/surrogate solution may be replaced if the –50%-200% criteria fails in the CCV when calculated against the previous CCV.)

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the LIMS for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 100µg/L (5mL purge) standard is made using 50µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE **(Cat#30265)**	20000	25	20	200
Vinyl Acetate **(#3766)**	5000	100	80	200
Ketones **(cat#30006)**	5000	100	80	200
Liquid mix **(C-349H-07)**	2000	100	100	100
Custom mix **(CCS-1037)**	5000	50	40	100
Gases **(cat#30042)**	2000	100	100	100
Acrolein/Acrylonitrile **(CC2098.10)**	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L ICV/LCS/Matrix Spike is made using 25µL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

11. Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 All water samples are stored in the "True" refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). The temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. The weekend temperature is monitored with a Min/Max thermometer and recorded upon arrival next business day.

12. Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for acceptance criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. See **Table 2** of this SOP for acceptance criteria and corrective action.

12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). See **Table 2** of this SOP for acceptance criteria and corrective action. **When analyzing samples for South Carolina the limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - A method blank is analyzed every 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action..

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. See **Table 2** of this SOP for acceptance

criteria and corrective action. MS data evaluation must include the consideration of the following factors.

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13. Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard. See **Table 5** of this SOP for acceptance criteria. Tune must be met every 12 hours sample analysis is to be performed **(every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours).** The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with backround subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. See the LIMS for preparation of standards. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate**

solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate. All manual calibration integrations must be approved by the section manager or designated peer reviewer.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** See **Table 2** of this SOP for acceptance criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual calibration integrations must be approved by the section manager or designated peer reviewer Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride

SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

13.5.2 Initial Calibration Verification (ICV) - A second source standard is prepared at or near the CCV concentration and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual ICV integrations must be approved by the section manager or designated peer reviewer.

13.5.3 Continuing Calibration Verification (CCV) - A CCV is analyzed every 12 hour tune and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual CCV integrations must be approved by the section manager or designated peer reviewer. **.**

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check

sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

14. **Procedure**

14.1 LCS - An LCS is analyzed every 12 hour tune.Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See **Table 2** of this SOP for acceptance criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample).**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a non-conformance report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample

15-Sample
16-Sample
17-Sample MS
18-Sample MSD
19-BFB (12:00pm - 12 hours since last BFB/CCV)
20-CCV
21-LCS
22-Method Blank
23-Sample
24-Sample

14.5 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. See **Table 2** of this SOP for acceptance criteria and corrective action. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas counts and retention times.

14.5.2 Surrogates – Recoveries and retention times.
Federal Register **Method 624 contains no criteria for surrogate recovery.**

Surrogate	WATER	SOIL
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration.

14.5.4 Qualitative identification based on spectrum and retention time.

15. Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calculations:

15.2.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:
A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples.
W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

16. **Method Performance**

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria.

17. **Pollution Prevention**

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. **Data Assessment and Acceptance Criteria for Quality Control Measures**

18.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. **Contingencies for Handling out-of-control or unacceptable data**

19.1 Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20. **Waste Management.**

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. **References**

21.1 40 CFR, Part 136; Appendix A
21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005

22. Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).
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Table 1 – DL/LOD/LOQ

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,1,1,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,1-Trichloroethane (1,1,1-TCA)	1.25	2.50	5.00	ug/Kg
1,1,2,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	2.50	5.00	10.0	ug/Kg
1,1,2-Trichloroethane	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethane (1,1-DCA)	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethene (1,1-DCE)	1.25	2.50	5.00	ug/Kg
1,1-Dichloropropene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichloropropane	1.25	2.50	5.00	ug/Kg
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,4-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,2-Dibromo-3-chloropropane (DBCP)	2.50	5.00	10.0	ug/Kg
1,2-Dibromoethane (EDB)	1.25	2.50	5.00	ug/Kg
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2-Dichloroethane (EDC)	1.25	2.50	5.00	ug/Kg
1,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,3,5-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
2,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/Kg
2-Chlorotoluene	1.25	2.50	5.00	ug/Kg
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/Kg
4-Chlorotoluene	1.25	2.50	5.00	ug/Kg
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/Kg
Acetone	5.00	10.0	20.0	ug/Kg
Acrolein	5.00	10.0	20.0	ug/Kg
Acrylonitrile	5.00	10.0	20.0	ug/Kg
Benzene	1.25	2.50	5.00	ug/Kg
Bromobenzene	1.25	2.50	5.00	ug/Kg
Bromochloromethane	1.25	2.50	5.00	ug/Kg
Bromodichloromethane	1.25	2.50	5.00	ug/Kg
Bromoform	1.25	2.50	5.00	ug/Kg
Bromomethane	2.50	5.00	10.0	ug/Kg
Carbon Disulfide	1.25	2.50	5.00	ug/Kg
Carbon Tetrachloride	1.25	2.50	5.00	ug/Kg
Chlorobenzene	1.25	2.50	5.00	ug/Kg
Chloroethane	2.50	5.00	10.0	ug/Kg
Chloroform	1.25	2.50	5.00	ug/Kg
Chloromethane	2.50	5.00	10.0	ug/Kg
cis-1,2-Dichloroethene (cis-1,2-DCE)	1.25	2.50	5.00	ug/Kg
cis-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Cyclohexane	1.25	2.50	5.00	ug/Kg
Dibromochloromethane	1.25	2.50	5.00	ug/Kg

Analyte	MDL/DL	LOD	MRL/LOQ	Units
Dibromomethane	1.25	2.50	5.00	ug/Kg
Dichlorodifluoromethane (CFC-12)	2.50	5.00	10.0	ug/Kg
Ethyl methacrylate	1.25	2.50	5.00	ug/Kg
Ethylbenzene	1.25	2.50	5.00	ug/Kg
Hexachlorobutadiene	1.25	2.50	5.00	ug/Kg
Iodomethane	5.00	10.0	20.0	ug/Kg
Isopropylbenzene (Cumene)	1.25	2.50	5.00	ug/Kg
Methyl Acetate	2.50	5.00	10.0	ug/Kg
Methyl methacrylate	1.25	2.50	5.00	ug/Kg
Methyl Tertiary Butyl Ether (MTBE)	1.25	2.50	5.00	ug/Kg
Methylcyclohexane	1.25	2.50	5.00	ug/Kg
Methylene Chloride, or Dichloromethane	2.50	5.00	10.0	ug/Kg
Naphthalene	1.25	2.50	5.00	ug/Kg
n-Butylbenzene	1.25	2.50	5.00	ug/Kg
n-Propylbenzene	1.25	2.50	5.00	ug/Kg
p-Isopropyltoluene	1.25	2.50	5.00	ug/Kg
sec-Butylbenzene	1.25	2.50	5.00	ug/Kg
Styrene	1.25	2.50	5.00	ug/Kg
tert-Butylbenzene	1.25	2.50	5.00	ug/Kg
Tetrachloroethene (PCE; PERC)	1.25	2.50	5.00	ug/Kg
Toluene	1.25	2.50	5.00	ug/Kg
trans-1,2-Dichloroethene (trans-1,2-DCE)	1.25	2.50	5.00	ug/Kg
trans-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Trichloroethene (TCE)	1.25	2.50	5.00	ug/Kg
Trichlorofluoromethane (CFC-11)	2.50	5.00	10.0	ug/Kg
Vinyl acetate	2.50	5.00	10.0	ug/Kg
Vinyl Chloride (VC)	2.50	5.00	10.0	ug/Kg
m,p-Xylene	2.50	5.00	10.0	ug/Kg
o-Xylene	1.25	2.50	5.00	ug/Kg
1,1,1,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,1-Trichloroethane (1,1,1-TCA)	0.25	0.50	1.00	ug/L
1,1,2,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	0.50	1.00	2.00	ug/L
1,1,2-Trichloroethane	0.25	0.50	1.00	ug/L
1,1-Dichloroethane (1,1-DCA)	0.25	0.50	1.00	ug/L
1,1-Dichloroethene (1,1-DCE)	0.25	0.50	1.00	ug/L
1,1-Dichloropropene	0.25	0.50	1.00	ug/L
1,2,3-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,3-Trichloropropane	0.50	1.00	2.00	ug/L
1,2,4-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,4-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,2-Dibromo-3-chloropropane (DBCP)	0.50	1.00	2.00	ug/L
1,2-Dibromoethane (EDB)	0.25	0.50	1.00	ug/L
1,2-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,2-Dichloroethane (EDC)	0.25	0.50	1.00	ug/L
1,2-Dichloropropane	0.25	0.50	1.00	ug/L
1,3,5-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,3-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,3-Dichloropropane	0.25	0.50	1.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,4-Dichlorobenzene	0.25	0.50	1.00	ug/L
1-Chlorohexane	0.50	1.00	2.00	ug/L
2,2-Dichloropropane	0.25	0.50	1.00	ug/L
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/L
2-Chloroethyl vinyl ether	1.25	2.50	5.00	ug/L
2-Chlorotoluene	0.25	0.50	1.00	ug/L
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/L
4-Chlorotoluene	0.25	0.50	1.00	ug/L
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/L
Acetone	2.50	5.00	10.0	ug/L
Acrolein	1.25	2.50	5.00	ug/L
Acrylonitrile	2.50	5.00	10.0	ug/L
Benzene	0.25	0.50	1.00	ug/L
Bromobenzene	0.25	0.50	1.00	ug/L
Bromochloromethane	0.25	0.50	1.00	ug/L
Bromodichloromethane	0.25	0.50	1.00	ug/L
Bromoform	0.25	0.50	1.00	ug/L
Bromomethane	0.50	1.00	2.00	ug/L
Carbon Disulfide	0.25	0.50	1.00	ug/L
Carbon Tetrachloride	0.25	0.50	1.00	ug/L
Chlorobenzene	0.25	0.50	1.00	ug/L
Chloroethane	0.50	1.00	2.00	ug/L
Chloroform	0.25	0.50	1.00	ug/L
Chloromethane	0.25	0.50	1.00	ug/L
cis-1,2-Dichloroethene (cis-1,2-DCE)	0.25	0.50	1.00	ug/L
cis-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Cyclohexane	0.25	0.50	1.00	ug/L
Dibromochloromethane	0.25	0.50	1.00	ug/L
Dibromomethane	0.25	0.50	1.00	ug/L
Dichlorodifluoromethane (CFC-12)	0.50	1.00	2.00	ug/L
Di-isopropyl ether	0.25	0.50	1.00	ug/L
ETBE	0.25	0.50	1.00	ug/L
Ethyl methacrylate	0.25	0.50	1.00	ug/L
Ethylbenzene	0.25	0.50	1.00	ug/L
Hexachlorobutadiene	0.25	0.50	1.00	ug/L
Iodomethane	0.25	0.50	1.00	ug/L
Isopropylbenzene (Cumene)	0.25	0.50	1.00	ug/L
Methyl Acetate	0.50	1.00	2.00	ug/L
Methyl methacrylate	0.25	0.50	1.00	ug/L
Methyl Tertiary Butyl Ether (MTBE)	0.25	0.50	1.00	ug/L
Methylcyclohexane	0.25	0.50	1.00	ug/L
Methylene Chloride, or Dichloromethane	0.50	1.00	2.00	ug/L
Naphthalene	0.25	0.50	1.00	ug/L
n-Butylbenzene	0.25	0.50	1.00	ug/L
n-Propylbenzene	0.25	0.50	1.00	ug/L
p-Isopropyltoluene	0.25	0.50	1.00	ug/L
sec-Butylbenzene	0.25	0.50	1.00	ug/L
Styrene	0.25	0.50	1.00	ug/L
t-Butyl alcohol	1.25	2.50	5.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
tert-Amyl methyl ether	2.50	5.00	10.0	ug/L
tert-Butylbenzene	0.25	0.50	1.00	ug/L
Tetrachloroethene (PCE; PERC)	0.25	0.50	1.00	ug/L
Tetrahydrofuran	1.25	2.50	5.00	ug/L
Toluene	0.25	0.50	1.00	ug/L
trans-1,2-Dichloroethene (trans-1,2-DCE)	0.25	0.50	1.00	ug/L
trans-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Trichloroethene (TCE)	0.25	0.50	1.00	ug/L
Trichlorofluoromethane (CFC-11)	0.50	1.00	2.00	ug/L
Vinyl acetate	1.25	2.50	5.00	ug/L
Vinyl Chloride (VC)	0.50	1.00	2.00	ug/L
m,p-Xylene	0.50	1.00	2.00	ug/L
o-Xylene	0.25	0.50	1.00	ug/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Method 8260B)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 5 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs: VOCs ≤ 30% and one option below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression r ≥ 0.995; **Option 3**: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value. [± 25% for non-DoD 8260B;]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates: VOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [± 20% for CCCs only non-DoD 8260B]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8260B, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qulaifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch.	No analytes 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 laboratory contaminants, no analytes detected >RL/LOQ	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix (see Box D-7).	Use LCS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 30% or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	<u>**Surrogate**</u> / **WATER** / **SOIL** Dibromofluoromethane: 85-120 / 80-125 1,2-Dichloroethane-d4: 85-135 / 75-140 Toluene-d8: 85-115 / 80-120 Bromofluorobenzene: 80-120 / 80-125 QC acceptance criteria specified by DoD (above) or Client. Otherwise, in-house control limits may be used. No limits specified for Method 624.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

Table 5, Tuning Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 6, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8260B/624/8270C/8270D/625 (Circle One)

QA/QC Item	Yes	No	NA	Second Review	Level
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?					
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).					
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?					
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.					
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?					
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?					
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?					
8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?					

Comments on any "No" response:

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_____

_____

_____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 7, Internal Standard Association

Analyte	Internal Standard	Analyte	Internal Standard
1,1,1-Trichloroethane	Fluorobenzene	1,1,1,2-Tetrachloroethane	d5-Chlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Fluorobenzene	1,1,2-Trichloroethane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2,3-Trichloropropane	d5-Chlorobenzene
1,1-Dichloroethene	Fluorobenzene	1,2-Dibromoethane (EDB)	d5-Chlorobenzene
1,1-Dichloropropene	Fluorobenzene	1,3-Dichloropropane	d5-Chlorobenzene
1,2-Dichloroethane	Fluorobenzene	1-Chlorohexane	d5-Chlorobenzene
1,2-Dichloroethane-d4	Fluorobenzene	2-Hexanone	d5-Chlorobenzene
1,2-Dichloroethene (total)	Fluorobenzene	Bromofluorobenzene	d5-Chlorobenzene
1,2-Dichloropropane	Fluorobenzene	Bromoform	d5-Chlorobenzene
1,4-Dioxane	Fluorobenzene	Chlorobenzene	d5-Chlorobenzene
2,2-Dichloropropane	Fluorobenzene	Chlorobenzene-d5	d5-Chlorobenzene
2-Butanone	Fluorobenzene	Dibromochloromethane	d5-Chlorobenzene
2-Chloroethyl vinyl ether	Fluorobenzene	Ethyl Methacrylate	d5-Chlorobenzene
4-Methyl-2-pentanone	Fluorobenzene	Ethylbenzene	d5-Chlorobenzene
Acetaldehyde	Fluorobenzene	m,p-Xylene	d5-Chlorobenzene
Acetone	Fluorobenzene	Methacrylonitrile	d5-Chlorobenzene
Acetonitrile	Fluorobenzene	o-Xylene	d5-Chlorobenzene
Acrolein	Fluorobenzene	Styrene	d5-Chlorobenzene
Acrylonitrile	Fluorobenzene	Tetrachloroethene	d5-Chlorobenzene
Allyl chloride	Fluorobenzene	Toluene	d5-Chlorobenzene
Benzene	Fluorobenzene	Toluene-d8	d5-Chlorobenzene
Bromochloromethane	Fluorobenzene	trans-1,3-Dichloropropene	d5-Chlorobenzene
Bromodichloromethane	Fluorobenzene	Xylenes (total)	d5-Chlorobenzene
Bromomethane	Fluorobenzene	1,1,2,2-Tetrachloroethane	1,4-dichlorobenzene-d4
Carbon disulfide	Fluorobenzene	1,2,3-Trichlorobenzene	1,4-dichlorobenzene-d4
Carbon tetrachloride	Fluorobenzene	1,2,4-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroethane	Fluorobenzene	1,2,4-Trimethylbenzene	1,4-dichlorobenzene-d4
Chloroform	Fluorobenzene	1,2-Dibromo-3-chloropropane	1,4-dichlorobenzene-d4
Chloromethane	Fluorobenzene	1,2-Dichlorobenzene	1,4-dichlorobenzene-d4
Chloroprene	Fluorobenzene	1,3,5-Trimethylbenzene	1,4-dichlorobenzene-d4
cis-1,2-Dichloroethene	Fluorobenzene	1,3-Dichlorobenzene	1,4-dichlorobenzene-d4
cis-1,3-Dichloropropene	Fluorobenzene	1,4-Dichlorobenzene	1,4-dichlorobenzene-d4
Cyclohexane	Fluorobenzene	1,4-Dichlorobenzene-d4	1,4-dichlorobenzene-d4
Dibromofluoromethane	Fluorobenzene	2-Chlorotoluene	1,4-dichlorobenzene-d4
Dibromomethane	Fluorobenzene	4-Chlorotoluene	1,4-dichlorobenzene-d4
Dichlorodifluoromethane	Fluorobenzene	Bromobenzene	1,4-dichlorobenzene-d4
Diisopropyl Ether	Fluorobenzene	cis-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Ethyl tert-Butyl Ether	Fluorobenzene	Hexachlorobutadiene	1,4-dichlorobenzene-d4
Fluorobenzene	Fluorobenzene	Naphthalene	1,4-dichlorobenzene-d4
Hexane	Fluorobenzene	n-Butylbenzene	1,4-dichlorobenzene-d4
Iodomethane	Fluorobenzene	n-Propylbenzene	1,4-dichlorobenzene-d4
Isobutyl alcohol	Fluorobenzene	p-Isopropyltoluene	1,4-dichlorobenzene-d4
Isopropylbenzene	Fluorobenzene	sec-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Acetate	Fluorobenzene	tert-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Methacrylate	Fluorobenzene	trans-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Methyl t-Butyl Ether	Fluorobenzene		
Methylcyclohexane	Fluorobenzene		
Methylene chloride	Fluorobenzene		
Propionitrile	Fluorobenzene		
t-Butyl alcohol	Fluorobenzene		
Tert-Amyl Methyl Ether	Fluorobenzene		
Tetrahydrofuran	Fluorobenzene		
trans-1,2-Dichloroethene	Fluorobenzene		
Trichloroethene	Fluorobenzene		
Trichlorofluoromethane	Fluorobenzene		
Vinyl acetate	Fluorobenzene		
Vinyl chloride	Fluorobenzene		

GC/ECD CHLORINATED ACID HERBICIDES BY EPA METHOD SW-846 8151A

SOP NUMBER: SOP-208

REVISION NUMBER: 13

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE MANAGER

EFFECTIVE DATE: 09/19/08

DATE OF LAST REVIEW: 09/19/08

GC/ECD
CHLORINATED ACID HERBICIDES
BY EPA METHOD SW-846 8151A

1.0 SCOPE AND APPLICATION

This SOP (based primarily on SW-846 Method 8000B/8151A) is used for the analysis of herbicide organic compounds in a variety of matrices (soils, sediments, waters, etc.). The analyses by these methods are clearly defined in the respective regulatory manuals. A good understanding of these methods is essential to the performance of each method. The normal laboratory list of analytes with their matrix (aqueous/non-aqueous) LCS limits are found attached in the appendix (page 10). Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8151A. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8151A. If questions still remain unanswered, check with the Organic Manager, QA/QC Officer and/or Technical Director.

2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Analytes are identified and confirmed based on the retention time of known standards then quantitated relative to known standards using the external standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories Quality Assurance Manual include details concerning sample preservation, containers and handling of samples and extracts. All water and soil samples are stored in the appropriate walk-in cooler at a temperature of 1°C – 4.4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 1°C – 4.4°C. Water samples have a holding time of 7 days from date of sampling for extraction. Soil samples have a holding time of 14 days from date of sampling for extraction (unless otherwise specified for the project). Extracts have a holding time of 40 days from extraction for analysis.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Section 3.0 of SW-846 Method 8151A details interferences and potential problems which may be encountered when dealing with herbicide analyses.

5.0. EQUIPMENT AND APPARATUS

5.1 GC's :

- 5.1.1 HP 5890/HP 5890 Series II/ Dual ECD - complete with temperature programmable gas chromatograph suitable for split/splitless injection.
- 5.1.2 Agilent 6890N/ Dual ECD - complete with temperature programmable gas chromatograph suitable for split/splitless injection.

5.2 Columns:
- 5.2.2 RTX-CLP(or equivalent): 30 m x 0.32 mm ID 0.5µm film thickness fused silica column.
- 5.2.3 RTX CLP II(or equivalent): 30 m x 0.32 mm ID 0.25 µm film thickness fused silica column.

5.3 Autosamplers:
- 5.3.1 HP 7673A/HP7673 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.
- 5.3.2 HP 7683/7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

5.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

5.5 Data Processing Software: Target DB Windows NT data system is interfaced to the HP Chemstation. The system accepts, processes, and stores acquired data.

6.0 REAGENTS

6.1 Hexane - pesticide quality or equivalent.

6.2 Ether - ultra-resi analyzed for organic residual analysis (Baker #9259-02 or equivalent)

6.3 Iso-Octane - for pesticide residual analysis (Fisher #0-297 or equivalent)

6.4 Methanol - suitable for use in gas chromatography (Omnisolv MX0484-1 or equivalent)

6.5 Diazomethane – as prepared in SOP-328.

6.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the GC standards logbook. The date they are opened is noted on the label and recorded in the GC standards logbook along with their lot number and vendor and each is given a sequential number. Each standard that is prepared is recorded in the GC standards logbook and given a sequential number. The following are noted in the logbook: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C – 4.4°C from the date they are received/prepared. The refrigerator temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook. See the GC standards log book for makeup of standards. NOTE: If standards are not purchased in the methyl ester form, they must be esterified before use **(See SOP-328)**. If purchased as methyl esters, the indicated conversion factor must be applied to convert methyl ester concentrations to acid concentrations.

6.7 The Surrogate Standard, 2,4-Dichlorophenylacetic acid, DCAA is purchased in a solution in acetone. The date that it is received is noted on the label and recorded in the GC standards logbook. The date opened is noted on the label and recorded in the GC standards logbook along with the lot number and vendor and is given a sequential number.

6.8 Calibration Standard Preparation

6.8.1 Herbicide intermediate standard mix – See the GC Standards Logbook for preparation of standards. The solution is transferred into a labeled, teflon-lined, screw-capped vial and stored in the refrigerator at a temperature of 1°C - 4.4°C for up to 6 months.

Analyte (Methyl Ester)	Conversion Factor
2, 4-D	0.9407
2, 4-DB	0.9470
2, 4, 5-TP (Silvex)	0.9506
2, 4, 5-T	0.9479
Dalapon	0.9107
Dicamba	0.9407
Dichloroprop	0.9434
Dinoseb	0.9452
MCPA	0.9346
MCPP	0.9390

6.8.2 Calibration standards are prepared from the above intermediate standard at a minimum of five concentrations. Standards are prepared fresh each time they are needed and discarded after use. Note: standards prepared from an alternate source or lot than the calibration stock standards are used for all LCS spikes and the MS/MSD for full list spikes. See the GC Standards Logbook for preparation of calibration curve standards.

6.8.3 The Initial Calibration Verification (ICV) intermediate standard is prepared from vendor stock standards in the same manner as the Calibration intermediate standard above and is stored in the refrigerator at a temperature of 1°C – 4.4°C for up to 6 months. The ICV standard is then prepared at a concentration near the midpoint in the same manner as the Calibration standards above.

7.0 PROCEDURE

The GC/ECD should be primed by injecting a herbicide standard at the highest concentration of the calibration curve. Inject this prior to beginning initial or daily calibration.

7.1 Chromatographic conditions:

7.1.1 GC	ECD1 or 3
Purge on	0.50 min.
Injector temperature	210°C
Column flow	5-6 mL/min
Initial column temperature	50°C for 7.0 minutes
Initial temperature ramp	20°C/min
Second column temperature	150°C for 1.0 minute
Second temperature ramp	5°C/min
Third column temperature	230°C for 0.5 min
Final temperature ramp	20°C/min
Final column temperature	270°C for 0.0 minutes

7.2 Calibration - (See SW-846 Method 8000B Section 7.4.2).

7.2.1 Initial Calibration - A five point calibration curve must be injected and analyzed for each analyte of interest. For most analytes a six point curve is used. Injection volume for standards and samples is equal to 2 µL using the same injection technique to introduce both standards and samples (use of auto-injectors makes this a constant). All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. The percent relative standard deviation (RSD) of the calibration factor must be < 20% over the working range for each analyte of interest. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient (***r***) is $\geq$0.995. Quadratic can be used if correlation coefficient is $\geq$0.99 Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

7.2.2 Initial Calibration Verification - A second source standard at the midpoint level concentration is used to check the validity of the curve. The standard recovery for all analytes must be between 85 and 115% (**80-120% for DOD QSM**). If the second source recovery is above 115%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 85%, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report.

7.2.3 Continuing Calibration Verification (CCV) - A mid-level standard must be analyzed every ten samples and cannot exceed 15 percent difference (%D) from the average calibration factor of the calibration

curve. The 15% criteria may still be met if the targets are <30% and the average of the %Ds is less than 15% (the client is notified). A CCV must also be analyzed at the end of the analysis sequence. If the CCV fails at any point, GC maintenance may be necessary (see SOP-222), reanalysis may be required (for samples analyzed since the last valid CCV) and a corrective action report must be completed. Alternatively, analytes may be flagged depending on their concentration and the status of the analyte in the CCV standard. No reanalysis is necessary if the analyte is undetected in the samples and recovered high in the CCV. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. Samples are then quantitated against the initial calibration curve. **For DOD QSM projects, CCVs are analyzed at the beginning and end of the sequence and after every 10 field samples. The CCVs should be within ± 20% difference.**

7.3 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program). If the established retention time window is less than ± 0.03 minutes, the window defaults to ± 0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

7.4 LCS - The LCS is analyzed 1/20 samples (1 per extraction batch - up to 20 samples - at varied concentrations). The LCS is spiked using an alternate lot or source from the calibration stock standards. See section 9.2 below for criteria and corrective action.

7.5 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch - up to 20 samples. See section 9.3 below for criteria and corrective action.

7.6 Samples - Prior to analysis, the samples are prepared for chromatography using SW-846 method 8150B/8151A.

7.7 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Chemserver 4920 data system. Quantitative measurements are performed as described in SW-846 8000B section 7.10. Rounding is performed using CLP odd/even rounding rules. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Formal data evaluation is detailed in SOP-216 (documented using the USACE Analyst Data Review Checklist for USACE projects).

7.7.1 Analyte concentration after rounding to 2 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover.

7.7.2 Surrogate standard recovery of DCAA must be checked to make sure it is within limits. See 9.1 below for corrective action. **For DOD QSM projects, DOD limits are used.**

7.8 Identification [See SW-846 method 8000B section 7.9]

7.8.1 Single peak components are identified by retention time on a primary column with confirmation by

retention time on a secondary column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. If both columns are equivalent, the highest concentration is reported. **Refer to SOP-224 for guidance with manual integrations. A before and after chromatogram with analyst's initials, date and reason must be included with the data for DOD QSM projects,**

7.8.1.1 Due to co-elution of certain compounds confirmation for all analytes can not be achieved. The percent difference (%D) for compounds, that confirmed from the primary column, on the confirmation column is 40%. The analyst must use experience and judgment to decide if the compound is there. Flag data with a "P" (**"J" for DOD projects**) if the results of the two columns differ by more than 40%. Report the higher of the two results unless overlapping peaks are causing erroneously high results.

7.8.1.2 If a compound is outside of its window on one column but in the window on the other column, the analysts will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to decide if the analyte is there.

8.0 Calculations:

8.1 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

8.2 The mean CF is calculated as follows:

$$\overline{CF} = \frac{\sum_{i-1}^{n} CFi}{n}$$

8.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i-1}^{n} (CFi - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{} X\ 100$$

CF

8.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±15% for each analyte before any sample analyses may take place.

8.5 Concentration in water samples is calculated as follows:

[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (µL).

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

CF = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

8.6 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to μg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

where:
A_x, V_t, D, and CF are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

9.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

9.1 Surrogates –Limits are determined by charting LCSs and method blanks. Surrogate standard recovery must be checked to determine if it is within limits. Corrective action must be taken when the surrogate is out of limits for a sample. Generally, the first action for herbicides is to re-esterify the sample and reanalyze. A corrective action form must be filled out and given to the organic lab manager within 24 hours. The organic lab manager will then make suggestions as to what further action should be taken, for example: sample may need to be reextracted or flagged on the report for a QC problem.

9.2 LCS Sample - The LCS is extracted 1/20 samples (every extraction batch - up to 20 samples - for USACE and GE projects). To prepare the LCS, a blank is spiked with standards prepared from alternate sources or lots than the calibration stock standards, and then extracted. The recoveries are tabulated to generate control charts and limits. See the LCS report form in the appendix for the laboratory-generated limits. If limits have not yet been generated, the limits default to those calculated from 8151A Table 4 (Mean ± 3SDs). If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the analytes should be flagged on the report. **For DOD QSM projects, DOD limits are used.**

9.3 Method Blanks - The concentration of all method target analytes should be below the MDL **(<RL, common laboratory contaminates; < ½ RL, all other compounds or client/authority specified)** for each method target analyte. If contamination exceeds the MDL, the following corrective actions must be taken. The first step is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and

any samples containing the same contaminant, would likely be reextracted/reanalyzed. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 5x that found in the method blank.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the LCS standard is used for this spike). Criteria for the MS/MSD are the same as the LCS limits with an RPD criteria of less than 25%. Samples, which do not meet these criteria due to matrix, must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **For DOD QSM projects, DOD limits are used.**

9.4.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

9.4.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

9.4.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

9.4.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

9.5 Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

10.0 HEALTH AND SAFETY

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

10.3 MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.

10.4 Diazomethane is a carcinogen and may explode under certain conditions. See Method 8151 for details in handling diazomethane.

11.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for proper disposal of waste from this sample preparation process. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method *8151*

12.2 USACE EM 200-1-3, 02-2001; Appendix I; Shell for Analytical Chemistry Requirements

12.2 DOD, Quality Systems Manual for Environmental Laboratories, Ver. 3, Jan. 2006.

13.0 EXAMPLE FORMS

Examples of the water LCS report sheet and the USACE analyst data review checklist are located in the appendix.

APPENDIX

FORM 3
WATER HERBICIDE LAB CONTROL SAMPLE

Lab Name: ELAB, Inc. Contract: QC

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.: QC.LCS

Matrix Spike - Client Sample No.: LCS

SPIKE COMPOUND	SAMPLE ADDED (µg/L)	LCS CONCENTRATION (µg/L)	LCS CONCENTRATION (µg/L)	QC. % REC #	LIMITS REC.
2,4-D	10.00	NA	10.00	100	48-214
2,4-DB	10.00	NA	10.00	100	60-126
2,4,5-TP(Silvex)	1.000	NA	1.000	100	68-166
2,4,5-T	1.000	NA	1.000	100	42-226
Dalapon	25.00	NA	25.00	100	40-110
Dicamba	1.000	NA	1.000	100	69-159
Dichloroprop	10.00	NA	10.00	100	60-110
Dinoseb	5.000	NA	5.000	100	D-110
MCPA	1000	NA	1000	100	24-117
MCPP	1000	NA	1000	100	35-131

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

Spike Recovery: 0 out of 10 outside limits

COMMENTS:_____

ANALYST REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8151A

QA/QC Item	Yes	No	NA	2nd Level Review
A. Initial Calibration				
1. Does the curve consist of five Calibration Standards?	___	___	___	_____
2. Is the low standard near, but above the MDL?	___	___	___	_____
3. Are the %RSDs within QC limits for all analytes?	___	___	___	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?	___	___	___	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples and at the end of the sequence?	___	___	___	_____
2. Are the % differences within QC limits for all analytes?	___	___	___	_____
D. Sample Analysis				
1. Are all sample holding times met?	___	___	___	_____
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	___	___	___	_____
3. Are all compounds identified on the primary column confirmed on the secondary column?	___	___	___	_____
4. Are Surrogate recoveries within QC limits?	___	___	___	_____

ANALYST REVIEW CHECKLIST

QA/QC Item	Yes	No	NA	2nd Level Review
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs?	____	____		_____
2. Is the LCS extracted at the desired frequency and are the percent recoveries within QC limits?	____	____		_____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries/RPDs within QC limits?	____	____		_____
F. Others				
1. Are all nonconformances included and noted?	____	____		_____
2. Are all calculations checked at the minimum frequency?	____	____		_____
3. Did analyst initial/date the appropriate printouts and report sheets?	____	____		_____
4. Are all sample ID and units checked for transcription errors?	____	____		_____
5. Are all manual integration checked by a second reviewer to verify why they were performed?	____	____		_____

Comments on any "No" response:

_____

_____

_____

_____

_____

_____

_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 211 **REVISION #: 22** **EFFECTIVE DATE: 070710**

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD) ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY EPA METHOD 608/608.2 or SW846 METHOD 8081A/8082 or 8081B/8082A

APPROVALS:

Lab Director: _____ Date: 7/8/10

Data Quality Manager: _____ Date: 7/7/10

Section Supervisor: _____ Date: 7/7/10

Changes Summary

Revision 22, 07/07/10

- The SOP is an update from Revision 21 dated 04/11/10.
- The SOP has been updated to move specific requirements to tables at the back of the SOP and add Mirex, PCB-1262, PCB-1268 as analytes.

Revision 21, 04/11/10

- The SOP is an update from Revision 20 dated 04/27/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

This SOP is compliant with SW-846 Methods 8000B/8081A/8082 and 8000C/8081B/8082A. *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This Standard Operating Procedure, SOP, is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits

See Table1.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Detection Limit/Method Detection Limit, Limit of Detection and Reporting Limit/Limit of Quantitation for each analyte.

4.3 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition. Analytes are quantitated relative to known standards using the external standard method.

6.0 Definitions

Laboratory Quality System SOP QS08 **"**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

Section 3.0 of SW-846 Methods 8081A/8082 and Section 4.0 of Methods 8081B/8082A details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308, 309 and 330) to evaluate possible clean-up options for any encountered interferences.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of latex gloves and lab coats is highly recommended.

8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

8.4 MSDS sheets are available for all reagents and standards that have been purchased. These are located on the bookshelves in the Quality Assurance Officer's office.

9.0 Equipment & Supplies

9.1 GC's:

9.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.

9.2 Columns:

9.2.1 Restek Siltek Guard Column (or equivalent): 10 meter x 0.32 mm ID

9.2.2 RTX-CLP or ZB-MR1 (or equivalent): 30 meter x 0.32 mm ID x 0.5 µm film thickness fused silica column.

9.2.3 RTX-CLP II or ZB-MR2 (or equivalent): 30 meters x 0.32 mm ID x 0.5 µm film thickness fused silica column.

9.3 Autosamplers:

9.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

9.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

9.5 Data Processing Software: Target DB Windows data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

10.0 Reagents and Standards

10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the reagents and standards used for the performance of the method. See **Table 5** for information on standard sources/calibration concentrations.

10.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the COA and recorded in the LIMS. The date they are opened is recorded in the LIMS along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the LIMS and given a sequential number. The following are noted in the LIMS: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, concentration, expiration date, and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C-4.4°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook.

10.3 List of Reagents:

Hexane - pesticide quality or equivalent.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling while soil samples

have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.

12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.

12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:

12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.

12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

13.1 Quality Systems **SOP QS08 "**Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

13.2 See Section 14.4 for Calibration details.

14.0 Procedure

14.1 The GC/ECD should be primed by injecting a pesticide standard at 200-500 µg/L and/or PCB standard at 2,500 µg/L, 10 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

14.2 Chromatographic conditions:

14.2.1 ZB MR1/MR2 columns:

GC	ECD3
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/340°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

14.2.2 ZB MR1/MR2 columns:

GC	ECD4
Purge on	60ml/min at 0.50 min.
Injector/Detector temperature	250/350°C
Column flow	3.0 mL/min
Initial column temperature	100°C for 1.0 minutes
Temperature ramp	35°C/min
Intermediate column temperature	220°C for 0.0 minutes
Second Temperature Ramp	15°C/min
Final Column Temperature	340°C for 2.0 minutes

Note: Current gas chromatograph conditions can be confirmed in the corresponding maintenance log.

14.3 Eval Mix – Before pesticide calibration and/or sample analysis, a degradation check standard (evaluation mix) of endrin and 4,4'-DDT must be injected. Degradation of either compound must not exceed 15 percent. See **Table 2** for criteria and corrective action.

14.4 Calibration - (See SW-846 Method 8000B Section 7.4 or Method 8000C Section 9.3).

14.4.1 Initial Calibration - An initial multi-point calibration curve must be prepared in hexane, analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources and below for makeup of the intermediates. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. For single component pesticides and surrogates, a seven point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Technical Chlordane a single low calibration point standard is analyzed unless they are expected/detected then a six-point calibration is injected and analyzed. Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other Aroclor mixtures (1221, 1232, 1242, 1248, 1254, 1262 & 1268). Full calibration is required if they are expected/detected. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

Mix A/B (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of A/B Mix and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.*

Mirex (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 100µL of Mirex and 50µL Surrogate are injected into a 10mL volumetric flask

containing approximately 9.5mL hexane and diluted to volume with same to make a 1 µg/mL standard.*

Technical Chlordane (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 100µL of Technical Chlordane and 500µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 10 µg/mL standard.

Toxaphene (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Toxaphene and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

Aroclor 1016/1260 (and Surrogate) Calibration Intermediate Solution: Using a 500µL syringe, 500µL of Aroclor 1016/1260 and 250µL Surrogate are injected into a 10mL volumetric flask containing approximately 9.5mL hexane and diluted to volume with same to make a 50 µg/mL and 5ug/ml standard.*

*After capping and inverting several times, all solutions are transferred into labeled, 12ml, teflon-lined, screw-capped vials and stored in the refrigerator at 4°C or less for up to 6 months. These standards are used to make the calibration curve standards in hexane at the concentrations found in table 5.

14.4.2 Initial Calibration Verification - A second source standard must be prepared in hexane, analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.4.3 Continuing Calibration Verification (CCV) - Every 12 hours (and at the end of the analysis sequence), a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. See **Table 5** for standard concentrations/sources. Any manual integrations are documented by inclusion of the integrated chromatograms (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.4.4 RT Windows - Retention time criteria set forth in SW-846 method 8000B Section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration using the midpoint standard RTs. If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

14.5 Samples - Prior to using Method 608, SW-846 8081A, 8081B, 8082, 8082A or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3541, 3546, 3640, 3550, 3580, EPA method 608 or CLP).

14.5.1 Example of a sequence run log:

1-Primer A/B Mix-1000 or Primer PCB-10,000
2- EVAL Mix (Pest only)
3- CCV A/B Mix
4- CCV Toxaphene (single point)
5-CCV Chlordane (single point)
6- CCV PCB 1660
7- Method Blank
8-LCS A/B Mix
9-LCS PCB
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample
19-Sample
20-Sample
21-Sample-MS
22-Sample-MSD
23-Sample
24-Sample
25-Sample
26-Sample
27-Sample
28-Sample
29- CCV A/B Mix
30-CCV PCB

14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Target DB Windows data system. Quantitative measurements are performed as described in SW-846 8081A Section 7.5.6, and SW-846 8081B Section 11.5.6.1. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Analyte concentration after rounding to 3 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

14.6.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

14.6.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your supervisor), [see SOP-308].

14.6.4 Analyte quantitation verification.

14.7 Identification/Quantitation [See SW-846 method 8081A Section 7.6 or method 8082 Sections 7.7-7.9].

14.10.1 Single peak components are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound.

14.10.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be qualified appropriately.

14.10.1.2 If a compound is outside of its window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to determine if the analyte is present.

14.10.2 Multi-peak components (PCB's, Toxaphene and Technical Chlordane) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 7.7.3 of SW-846 Method 8082. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.

A. Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components.

B. Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic lab manager’s attention.

C. Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic lab manager’s attention.

14.10.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. Calculations follow in Section 15.0.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Calculate the calibration factor (CF) for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

15.3 The mean CF is calculated as follows:

$$\text{AvgCF} = \frac{\sum(\text{CF for each standard})}{N}$$

15.4 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i-1}^{n} (CFi - \overline{CF})^2}{n-1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

15.5 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ \text{Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ \text{Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration.

15.6 Concentration in water samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.

V_t = Total volume of the concentrated extract (µL).

D= Dilution factor, if the sample was diluted prior to analysis.
If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

CF = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

15.7 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(W_s)}$$

where:

A_x, V_t, D, and CF are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

16.1 Method Detection Limit Study or Detection Limit Determination
16.2 Limit of Detection Verification
16.3 Limit of Quantitation or Reporting Limit Verification
16.4 Demonstration of Capability (DOC)
16.5 PT Studies

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Please see Waste Disposal, SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

21.0 References

21.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Method 8081A, 8081B, 8082, 8082A

21.2 USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 608, 608.2; APX-B

21.3 USEPA Contract Laboratory Program (CLP) for Organics ILM04.2; ILM04.3

21.4 DOD Quality Systems Manual, Ver. 3/4.1

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Table 5, Calibration Standards

Table1- Detection limits

Analyte (water)	MDL/DL	LOD	LOQ/RL	Units
4,4'-DDD	0.00500	0.0100	0.0200	ug/L
4,4'-DDE	0.00500	0.0100	0.0200	ug/L
4,4'-DDT	0.00500	0.0100	0.0200	ug/L
Aldrin	0.00330	0.0100	0.0200	ug/L
alpha-BHC	0.00330	0.0100	0.0200	ug/L
alpha-Chlordane	0.00330	0.0100	0.0200	ug/L
beta-BHC	0.00330	0.0100	0.0200	ug/L
Chlordane (tech)	0.0170	0.0250	0.0500	ug/L
delta-BHC	0.00330	0.0100	0.0200	ug/L
Dieldrin	0.00500	0.0100	0.0200	ug/L
Endosulfan I	0.00330	0.0100	0.0200	ug/L
Endosulfan II	0.00500	0.0100	0.0200	ug/L
Endosulfan sulfate	0.00500	0.0100	0.0200	ug/L
Endrin	0.00500	0.0100	0.0200	ug/L
Endrin aldehyde	0.00500	0.0100	0.0200	ug/L
Endrin ketone	0.00500	0.0100	0.0200	ug/L
gamma-BHC (Lindane)	0.00330	0.0100	0.0200	ug/L
gamma-Chlordane	0.00330	0.0100	0.0200	ug/L
Heptachlor	0.00330	0.0100	0.0200	ug/L
Heptachlor epoxide	0.00330	0.0100	0.0200	ug/L
Methoxychlor	0.00330	0.0100	0.0200	ug/L
Mirex	0.00330	0.0100	0.0200	ug/L
Toxaphene	0.330	0.667	1.00	ug/L
Aroclor-1016	0.125	0.250	0.500	ug/L
Aroclor-1221	0.125	0.250	0.500	ug/L
Aroclor-1232	0.125	0.250	0.500	ug/L
Aroclor-1242	0.125	0.250	0.500	ug/L
Aroclor-1248	0.125	0.250	0.500	ug/L
Aroclor-1254	0.125	0.250	0.500	ug/L
Aroclor-1260	0.125	0.250	0.500	ug/L
Aroclor-1262	0.125	0.250	0.500	ug/L
Aroclor-1268	0.125	0.250	0.500	ug/L
Analyte (Soil)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
4,4'-DDD	0.170	0.340	0.670	ug/Kg
4,4'-DDE	0.170	0.340	0.670	ug/Kg
4,4'-DDT	0.170	0.340	0.670	ug/Kg
Aldrin	0.110	0.340	0.670	ug/Kg
alpha-BHC	0.110	0.340	0.670	ug/Kg
alpha-Chlordane	0.110	0.340	0.670	ug/Kg
beta-BHC	0.110	0.340	0.670	ug/Kg
Chlordane (tech)	0.570	0.850	1.70	ug/Kg
delta-BHC	0.110	0.340	0.670	ug/Kg
Dieldrin	0.170	0.340	0.670	ug/Kg
Endosulfan I	0.110	0.340	0.670	ug/Kg
Endosulfan II	0.170	0.340	0.670	ug/Kg
Endosulfan sulfate	0.170	0.340	0.670	ug/Kg
Endrin	0.170	0.340	0.670	ug/Kg
Endrin aldehyde	0.170	0.340	0.670	ug/Kg

Analyte (Soil)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
Endrin ketone	0.170	0.340	0.670	ug/Kg
gamma-BHC (Lindane)	0.110	0.340	0.670	ug/Kg
gamma-Chlordane	0.110	0.340	0.670	ug/Kg
Heptachlor	0.110	0.340	0.670	ug/Kg
Heptachlor epoxide	0.110	0.340	0.670	ug/Kg
Methoxychlor	0.110	0.340	0.670	ug/Kg
Toxaphene	11.0	22.0	33.0	ug/Kg
Aroclor-1016	4.17	8.33	16.7	ug/Kg
Aroclor-1221	4.17	8.33	16.7	ug/Kg
Aroclor-1232	4.17	8.33	16.7	ug/Kg
Aroclor-1242	4.17	8.33	16.7	ug/Kg
Aroclor-1248	4.17	8.33	16.7	ug/Kg
Aroclor-1254	4.17	8.33	16.7	ug/Kg
Aroclor-1260	4.17	8.33	16.7	ug/Kg
Aroclor-1262	4.17	8.33	16.7	ug/Kg
Aroclor-1268	4.17	8.33	16.7	ug/Kg
Analyte (TCLP)	**MDL/DL**	**LOD**	**LOQ/RL**	**Units**
Chlordane (tech)	0.000170	0.000250	0.000500	mg/L
Endrin	0.0000500	0.000100	0.000200	mg/L
gamma-BHC (Lindane)	0.0000330	0.000100	0.000200	mg/L
Heptachlor	0.0000330	0.000100	0.000200	mg/L
Heptachlor epoxide	0.0000330	0.000100	0.000200	mg/L
Methoxychlor	0.0000330	0.000100	0.000200	mg/L
Toxaphene	0.00330	0.00670	0.0100	mg/L

Table 1. Organic Analysis by Gas Chromatography (Methods 8081, 8082)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study. Minimum ±0.030min.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD analyses. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration, if detected. Results may not be quantitated using a single point.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate for DoD analyses.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL;	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes 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.	Correct problem, then see SOP QS05. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Organic Analysis by Gas Chromatography (Methods 8081, 8082) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Qualify surrogate results on form I.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply qualifier if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 4, Data Reviewers Checklist (Prior to approving data)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8081/8082**

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards (six for quadratic curve)?				
3. Is the low standard equal to or below the MRL/LOQ?				
4. Are the %RSD or fit criteria within QC limits for all analytes?				
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				
6. Are surrogate recoveries within QC limits? (one surrogate both columns)				

ANALYST DATA REVIEW CHECKLIST, cont.

E. QC Samples

1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs? _____ _____ _____ _____
2. Is the Laboratory Control Sample and its percent recovery within QC limits? _____ _____ _____ _____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits? _____ _____ _____ _____

F. Others

1. Are all nonconformances included and noted? _____ _____ _____ _____
2. Are all calculations checked at the minimum frequency with one example worked out in the space below? _____ _____ _____ _____
3. Did analyst initial/date the appropriate printouts and report sheets? _____ _____ _____ _____
4. Are all sample IDs and units checked for transcription errors? _____ _____ _____ _____
5. Are all manual integrations checked by a second reviewer to verify they were performed correctly? _____ _____ _____ _____

Calculation – one complete calculation from raw area/height to final concentration:

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 – Standard concentrations/sources
NOTE: All standards are fully documented within the LIMS

	Level 1 (ppb)	Level 2 (ppb)	Level 3 (ppb)	Level 4 (ppb)	Level 5 (ppb)	Level 6 (ppb) MIDPOINT	Level 7 (ppb)	Primary Source (Concentration-ppm)	Secondary Source** (Concentration-ppm)
Single Component Pesticides	1	5	10	25	50	100	200	Restek (200)	Accustandard (1000)
Mirex	1	5	10	25	50	100	200	Accustandard (100)	ChemService (100)
DCB/TCMX	1	5	10	25	50	100	200	Restek (200)	NA
Technical Chlordane*	-	5	10	25	50	100	200	Restek (1000)	Ultra Scientific (5000)
Toxaphene*	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (100)
PCB-1016/PCB-1260	-	50	100	500	750	1000	2500	Restek (1000)	Accustandard (1000)
PCB-1221*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1242*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1248*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1254*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (1000)
PCB-1262*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (100)
PCB-1268*	-	*50*	*100*	*500*	*750*	*1000*	*2500*	Restek (1000)	Accustandard (500)

* - Toxaphene and Technical Chlordane single point at low standard unless detected. PCB calibration 1016/1260 unless other pattern detected.

** - Secondary Source may be from any vendor other than the primary source.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP221 REVISION #: 09 EFFECTIVE DATE: 071210

TOTAL ORGANIC CARBON (TOC)
BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN

METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

APPROVALS:

Lab Director: _____ Date: 7/12/10

Data Quality Manager: _____ Date: 7/12/10

Section Supervisor: Betty DeVille Date: 7/12/10

Changes Summary

Revision 09, 07/12/10

- The SOP is an update from Revision 08 dated 04/28/09
- The SOP has been reviewed and confirmed to be accurate.
- The soil TOC method calibration concentrations were updated.
- The LCS reference was changed to BS.

TOTAL ORGANIC CARBON (TOC)
BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN
METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

I. SCOPE AND APPLICATION

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060/9060A and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the L1oyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices.SW846 Method 9060/9060A and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

II. SUMMARY OF METHOD

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO_2 formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

III. SAMPLING HANDLING AND PRESERVATION

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of

the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH </= 2) with HCl or H_2SO_4. Both water and soil samples are stored at 4°C.

IV. INTERFERENCES

4.1 WATER METHOD

4.1.1 Removal or carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

4.2 SOIL METHOD

4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.

4.2.2 The infrared detector is sensitized to CO_2 and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

V. DEFINITIONS

5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.

5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of

field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

5.7 LABORATORY CONTROL SAMPLE (BS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).

5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C (± 2°C).

6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL
25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL
50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL
100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL
0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.

2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

VII. INSTRUMENTATION

7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

7.2 There is a Shimadzu autosampler which will hold 68 samples.

7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

VIII. AQUEOUS SAMPLE PROCEDURE

8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.

8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.

1. 100 ppm
2. 50 ppm
3. 25 ppm
4. 10 ppm
5. 5.0 ppm
6. 2.5 ppm
7. 1.0 ppm
8. Method blank
9. BS + 9 samples (including any sample QC
10. 25 ppm
11. 10 samples (including any sample QC)
12. 50 ppm

8.8 Insrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

TC Mode Instrument Settings:

Analysis Temp:900°C
Analysis Time: 6.5 minutes
Nitrogen Gas Flow: 60-100 psi (external regulator regulator)
Oxygen Gas Flow: 40-60 psi (external regulator)

This is a step by step description of a routine soil TOC analysis.

9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!

9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.

9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).

9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.

9.5 If doing an initial calibration curve use an appropriate µL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

µL 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
25	7.5
50	15

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100µL of the 30% Sucrose standard with 500µL of water.

9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
2. Method Blank(MB + date analyzed for ID) – single analyses
3. BS, 15 mg dextrose (BS + date analyzed for ID) – single analyses
4. Sample – 4 replicates
5. Sample – 4 replicates
6. Sample – 4 replicates
7. Sample – 4 replicates
8. Sample – 4 replicates
9. CCV(CCV1+ date analyzed for ID)2 – single analyses
10. Sample – 4 replicates
11. Sample – 4 replicates
12. Sample – 4 replicates
13. Sample – 4 replicates
14. Sample – 4 replicates
15. CCV (CCV2+ date analyzed for ID) – single analyses

16. Sample – 4 replicates
17. Sample – 4 replicates
18. Sample – 4 replicates
19. Sample – 4 replicates
20. Sample – 4 replicates
21. CCV(CCV3+ date analyzed for ID) – single analyses
22. Sample – 4 replicates
23. Sample – 4 replicates
24. Sample – 4 replicates
25. Sample – 4 replicates
26. Sample – 4 replicates
27. CCV(CCV4+ date analyzed for ID) – single analyses
28. SampleMS – 4 replicates
29. SampleDUP – 4 replicates
30. FCV(CCV4+ date analyzed for ID) – single analyses
31. FCB(FCB4+ date analyzed for ID) – single analyses

9.7 Samples should be stored away from light and at 4°C (± 2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.

9.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.** After samples are dried crush samples using a clean mortar and pestle.

9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.

9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

X. QC REQUIREMENTS

10.1 Analyze a laboratory control sample (BS) for each batch of samples **(maximum of 10 samples per day).** If the BS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.

10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.

10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.

10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are $\leq$15% for aqueous samples and $\leq$20% for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or $\leq$30% (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.

10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than $\pm$ the MDL.

10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

. Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within $\pm$10%. A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060/9060A requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required numbered of sample replications.

10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

XI. CORRECTIVE ACTIONS

11.1 INSTRUMENT RELATED

1. ICV not within ± 20% (Soil) or ± 10% (SM 5310C0
   a. If the problem is with the solution.
      i. Re-prepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within ± 30% (Soil) or ±15% (SM 5310C)
   a. If the problem is with the solution.
      i. Re-prepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
         a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
         b. If the CCV was biased low, the previous ten samples must be reanalyzed.

*** Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

11.2 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% aqueous or ±50% soil/sediment
   i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within ±25% aqueous or ±50% soil/sediment
   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

XII. HEALTH AND SAFETY

A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.

D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the

disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

XIV. METHOD PERFORMANCE

14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

***KHP = potassium acid phthalate.**

14.2 There was no method performance data available for the soil procedure.

XV. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).

3. EPA SW-846, Method 9060/9060A.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

APPENDIX I.

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.

5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eigth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1"beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they ar loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightening bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into corret position will take almost 3 hours to finish. Can view data as its coming off by selecting "view" "sample window". After calibration is done review.
27. Select "File" then "New" then "sample run" "ok"
28. General information screen: No change select "ok"
29. Save as screen: Select today's date for file name example 00month/00day/00year
30. Select "save"
31. Sample Table Screen: Select "insert" then select " auto generate" enter
32. **Page 1** sample group wizard sample source: select "calibration curve" then double click on box with 3 dots …
33. Open latest curve from calibration curves file
34. Highlight latest curve and select "open"
35. Should send you back to page 1 with calibration curve info submitted. Select "next"
36. **Page 2** Sample Parameter: Enter final sample count for "number of samples" select "next"
37. **Page 3** Calibration Curves: No changes Select "Next"
38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, BSW, Sample #, client,etc.
41. Once everything is typed in double check that it matches the way samples and QC are loaded..
42. Click or select the lightening bolt symbol then select "use settings on PC". Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.

43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.

44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.

45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 225 **REVISION #: 09** **EFFECTIVE DATE: 20100907**

GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035/5035A FOR 8260B ANALYSIS

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

Revision 09, 09/07/10

- The SOP is an update from Revision 08 dated 09/24/08
- The SOP has been updated to include reference to 5035A and preservation by freezing for unpreserved Terracores and Encores.

GC/MS - VOLATILE
NON - AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 5035/A

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035 and 5035A. Soil samples should be sampled in the field using the EnCore™ sampler or prepared VOA vials (sometimes referred to as Terracore samplers) then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

2.0 SUMMARY

Samples are collected in EnCores or prepared VOA vials and submitted to the laboratory for preparation/analysis.. EnCore samplers have to be frozen or prepared within 48 hrs of collection. Prepared VOA vials (sometimes referred to as Terracores) are shipped already prepared in water, methanol or preservative solution. If prepared in water, freezing is required within 48 hours. If preservative is used, refrigeration is the only requirement.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

EnCores are prepped within 48 hrs of collection or frozen until preparation can be completed. Preparation can be in sodium bisulfate with refrigeration at 4°C or in reagent water with freezing. Prepared VOA vials are received already prepared in water, methanol or sodium bisulfate solution. If prepared in water, freezing is required within 48 hours of collection. If preservative is used, refrigeration at 4°C is the only requirement. Holding Time is 14 days from collection once preserved.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

5.0 EQUIPMENT AND MATERIALS

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH_3OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of $NaHSO_4$ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the $NaHSO_4$ in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

7.1 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

7.2 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the "low-level" sample. (Some projects may not require the "low-level" detection limits, in this case only the high level sample preparation would be required.). A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. **Additional bulk samples should be collected for screening and dry weight determination without the preservative.** Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

8.0 PROCEDURE

8.1 Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.

8.2 The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.

8.3 Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:

8.3.1 Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.

8.3.2 Get the samples from the Hobart assigned for volatile sample storage and log them out.

8.3.3 Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).

8.3.4 Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial

to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation.

8.3.5 For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate an NCR to document the problem.

8.3.6 Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.

8.3.7 Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

9.0 ANALYSIS

9.1 Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.

9.2 Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

9.2 If an extra bulk jar was not received for percent moisture determination, an alternate procedure using the methanol vial can be used with advance notice:

- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids (recommend a tare weight on the vial to the same degree of accuracy).
- Preserve the vial as normal.
- After we know the methanol extract is not needed or has been analyzed successfully, allow the methanol to evaporate and dry as necessary for % solids determination.
- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids.

10. HEALTH,SAFETY,WASTE MANAGEMENTAND POLLUTION PREVENTION

10.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

10.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the Quality Assurance Officers office.

10.4 Please see Waste Disposal SOP QS14 for proper disposal of waste coming from this area within our laboratory. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

REFERENCES

1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035, December 1996.
1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Other Methods; Method 5035A, July 2002.

DEFINITIONS

Refer to SOP QS08 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 300 **REVISION #: 18** **EFFECTIVE DATE: 042610**

GC/MS SEMI-VOLATILE BNA-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 3510C FOR 8270/625 ANALYSIS

APPROVALS:

Lab Director: _____ Date: 4/27/10

Data Quality Manager: _____ Date: 4/27/10

Section Supervisor: _____ Date: 4/27/10

Changes Summary

Revision Date: 042610

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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2. Applicable Matrix or Matrices
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1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 625.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples.

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated to an appropriate final volume.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.

7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.

7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

9.1 Separatory Funnel – 2L with Teflon stopcock

9.2 Beaker – 250mL or 400mL

9.3 Drying/Chromatographic column – 20mm I.D. x 300mm

9.4 Filter funnel

9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent

9.6 Metal rack – capable of holding six glass evaporation tubes

9.7 Turbo-Vap Evaporator – heated and capable of temperature control (+5˚C); the bath should be vented into a hood
9.8 Vials, 2.0 mL glass with Teflon-lined screw cap
9.9 pH indicator paper – wide range (1.0-12.0)
9.10 Syringe – 1mL
9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
9.12 Pasteur pipette – length 9”
9.13 Pasteur pipette bulb
9.14 Labels – DYMO
9.15 Teflon Bottles – 500mL
9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
9.17 Ring Stand – 3-prong
9.18 Burette clamp – double
9.19 Aluminum foil – heavy duty
9.20 Nitrogen tank – equipped with pressure regulator
9.21 Boiling chips – Teflon
9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

10.1 Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab as needed.
10.2 Sodium Hydroxide Solution - (10N). Weigh 800g NaOH (purchased in a fiber drum from Tennessee Reagents # 2-31825-25lb or equivalent) into a 2000mL volumetric flask and add approximately 1000mL of reagent water. Swirl until pellets are mostly dissolved. Add a stir bar and place on stir plate. This mixture will get very hot. Continue to add reagent water while mixture is being stirred until a final volume of 2000mL is attained. Let stand until cool. Transfer to 1000mL Teflon containers.
10.3 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh (purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent). For low level tests, place an aliquot in a 1500mL heavy duty Pyrex beaker and bake in muffle furnace at 400°C for a minimum of 4 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature
10.4 Glass Wool – Roving , 9989 Glass (purchased from Fisher #11-388 or equivalent).
10.5 Sulfuric Acid Solution - (1:1), slowly add 500mL of H2SO4 (Fisher, suitable for trace metal analysis #A300C-212 or equivalent) to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Allow to cool before use.
10.6 Extraction Solvent - Methylene Chloride (purchased from Fisher #D151-4 or equivalent) Please read SOP-336 before handling this solvent in our laboratory.
10.7 The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.
10.7.1 BNA Surrogate – The base neutral and acid surrogate are mixed together in one solution (purchased from NSI #WL-371-C at concentrations of 100-200ug/mL). The expiration for this standard is 6 months from the date opened. Use 0.5mL of this solution per 1000mL of aqueous sample.

10.7.2 BNA Spiking Solution – The base neutral and acid spiking solutions are mixed together in one solution called BNA LCS#1 (This spiking solution contains all the compounds that are normally calibrated by GC/MS). This solution, with a final concentration of 100ug/mL, is prepared in Methanol by making a dilution of stock purchased from reputable vendors (BNA LCS #1 spike kit #K-943 and 1-methylnapthalene #1288-01-08 are purchased from NSI, 2,6 Dichlorophenol #95591 is purchased from Absolute Standards and 1,4 Dioxane #30287 is purchased from Restek). Use 0.5mL of this solution per 1000mL of aqueous sample. Another spiking solution is also used, called BNA LCS#2. This solution contains short or matrix spike list base extractable compounds. This solution, with a final concentration of 100ug/mL,is prepared in Methanol by making a dilution of stock purchased from NSI #Q-6104-0. Use 0.5mL of this solution in combination with BNA LCS#1 for all full list BNA requirements. BNA LCS #2 may be omitted from samples requiring PAH analysis. (For low level PAHs, use 1.0mL of a 1.0ug/mL solution made from BNA LCS #1, called "LLPAH spiking solution.) All standards expire 6 months from the date they are made.

10.7.3 BNA TCLP Spike – 0.5mL of BNA LCS#1 and BNA LCS#2 is added per 100mL volume. This volume is provided by Wet Chemistry in a 1L glass amber bottle. 100mL is removed from this container and measured using a graduated cylinder.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not Applicable to this SOP

14.0 Procedure

14.1 All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):

14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS.

14.1.2 This backlog is used to determine extraction priorities based on hold times and due dates.

14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.

14.1.4 Samples are placed in LIMS "batches" based on parameter and extracted accordingly.

14.2 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

14.2.1 SLUDGE - use only 100mL and dilute to 1000mL with reagent water.

14.2.2 TCLP EXTRACT - use only 100mL and dilute to 1000mL with reagent water. A separate matrix spike of 100mLs should be set up at the same time. Dilute to 1000mL with reagent water.

14.2.3 1BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. SPLP extract-use 1 liter.

14.2.4 NPDES client - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1mL is added to the LCS and the matrix spike.

14.2.5 ACID EXTRACT WITH BAD MATRIX - a cleanup step is added. Samples are taken to a high pH, extracted with 60mL methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

14.3 LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs) – Samples require a BNA extraction. Use the surrogate and spiking solution specified.

14.4 Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the LIMS bench sheet and, later, in LIMS.

14.5 Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100mL and dilute to 1000mL with reagent water. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed. Rinse separatory funnels with methanol. Place label from sample bottle onto separatory funnel as samples are poured into funnels to ensure proper identification. Use Avery labels to properly identify method blank, LCS, and LCSD.

14.6 Using the 1000mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.

14.7 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

14.8 Generally 0.5mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate**.** Someone must verify that the surrogate has been added by initialing LIMS bench sheet.

14.8.1 NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

14.9 For the sample in each analytical batch selected for spiking, use the 0.5mL glass syringe designated for BNA spike, to add 0.5mL of BNA spiking solution. **For low level PAHs use 1.0mL of the 1.0ppm LLPAHs spiking solution**. Someone must verify that the spike has been added by initialing the LIMS bench sheet. For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.

14.10 Enter the ID# of the surrogate/spike used on the LIMS bench sheet and, later, in LIMS.

14.11 ACID EXTRACTION: Adjust the pH to between 1.0 and 2.0, using 2mL of 1:1 H2SO4. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H2SO4 solution in small increments, as required to attain the proper pH.

14.12 Add 40mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.

14.13 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.

14.13.1 NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

14.14 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40mL into a 250mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 250mL glass beaker.

14.15 Following Steps 14.12 through 14.14, extract two more times with 40mL of methylene chloride. Combine the three solvent extracts into the same 250mL beaker.

14.16 BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater, using 10N NaOH. Start by adding 5.0mL to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in

small increments, as required to attain the proper pH. BNA extraction is necessary when doing low level PAHs.

14.16.1 NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.

14.17 FOR 8270 extraction: Extract one more time with 40mL of methylene chloride following Step 14.16. Do not combine BN and Acid extracts in a same 250mL beaker. However, you may filter BN and Acid extracts through the same sodium sulfate filter and combine into the same turbo in order to concentrate BN and acid extracts for one final extract.

14.17.1 NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.

14.18 For 625 extractions: extract 3 more times with 40 mL methylene chloride following steps 14.12 through 14.14. Combine BN extracts in the empty 250mL sample beaker as the acid portion concentrates in the turbo vap. Following step 14.24, concentrate the acid extract to ~5mL and then filter the BN extract into the same turbo.

14.19 Prepare to dry the sample by either of the following methods:

14.19.1 Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR

14.19.2 Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

14.20 If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of Na2SO4 by sprinkling the top layer with Na2SO4 until it hardens, separates, and drops to the bottom.

14.22 Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume on the LIMS bench sheet to the nearest 10-mL and record, later, in LIMS.

14.23 Prepare sample vial tray using labels printed off from LIMS that identify sample numbers, initial/final volumes, client, parameter, and date extracted.

14.24 TURBO-VAP CONCENTRATION

14.24.1 Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.

14.24.2 Record the numbers of the Turbo-Vap tube on the LIMS bench sheet and place the tube in a metal holder.

14.24.3 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.

14.24.4 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

14.24.5 When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.

14.24.6 Draw ~0.25 mL of methylene chloride into a 9'' Pasteur pipette and add this aliquot to the turbo-vap. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 2-mL vial. Add methylene chloride from the designated clean pipette and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.

14.25 The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

14.26 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations

Not Applicable to this SOP

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not Applicable to this SOP

19.0 Contingencies for Handling out of control or unacceptable data

Not Applicable to this SOP

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition

21.2 40 CFR, Method 625.

22.0 Tables, Diagrams, Flowcharts, and Validation Data

Not Applicable to this SOP

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 302 **REVISION #: 17** **EFFECTIVE DATE: 042610**

PESTICIDE/PCBs
AQUEOUS MATRIX EXTRACTION
FOR EPA METHOD 608/608.2 AND
SW846 METHOD 8081/8082
USING SW846 METHOD 3510C

APPROVALS:

Lab Director: _____ Date: 4/27/10

Data Quality Manager: _____ Date: 4/27/10

Section Supervisor: _____ Date: 4/27/10

Changes Summary

Revision Date: 042610

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 608/608.2

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated and exchanged to hexane.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

6.2 Additional definitions specific to this SOP are listed below:

6.2.1 PCBs- polychlorinated biphenyls

6.2.2 Pest- pesticides

6.2.3 TCMX- tetrachloro-m-xylene

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.

7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.

7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

9.1 Separatory Funnel – 2L with Teflon stopcock
9.2 Beaker – 250mL or 400mL
9.3 Drying/Chromatographic column – 20mm I.D. x 300mm
9.4 Filter funnel
9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent
9.6 Metal rack – capable of holding six glass evaporation tubes
9.7 Turbo-Vap Evaporator – heated and capable of temperature control (±5˚C); the bath should be vented into a hood
9.8 Vials, 10mL glass with Teflon-lined screw cap
9.9 pH indicator paper – wide range (1.0-12.0)
9.10 Syringe – 1mL
9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
9.12 Pasteur pipette – length 9”
9.13 Pasteur pipette bulb
9.14 Labels – Avery
9.15 Teflon Bottles – 500mL
9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
9.17 Ring Stand – 3-prong
9.18 Burette clamp – double
9.19 Aluminum foil – heavy duty
9.20 Nitrogen tank – equipped with pressure regulator
9.21 Boiling chips – Teflon
9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

10.1 Reagents

10.1.1 Reagent water – Reagent water is gathered in a carboy from source in the instrument lab daily.

10.1.2 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent. Place an aliquot in a 1500mL heavy-duty Pyrex beaker and bake in muffle furnace at 400˚C for a minimum of 4 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature.

10.1.3 Sulfuric Acid Solution (1:1) – Slowly add 500mL concentrated Sulfuric Acid, purchased from Fisher #A300C-212 or equivalent, to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Let stand until cool.

10.1.4 Sodium Hydroxide Solution (10N) – Weigh 800g NaOH, purchased in a fiber drum from Tennessee Reagents #2-31825-25lb or equivalent, into a 2000mL volumetric flask and add approximately 1000mL of reagent water. Swirl until pellets are mostly dissolved. Add a stir bar and place on stir plate. This mixture will get very hot. Continue to add reagent water while mixture is being stirred until a final volume of 2000mL is attained. Let stand until cool. Transfer to 1000mL Teflon containers.

10.1.5 Methylene Chloride - purchased from Fisher #D151-4 or equivalent. **Please see SOP 336 before handling this solvent in our laboratory.**

10.1.6 Hexane – suitable for gas chromatography, purchased from Fisher #H303-4

10.2 Standards – The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

10.2.1 TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) – Surrogate solution is prepared, with a final concentration of 0.5ug/mL, by diluting a stock solution (purchased from Restek #32000) in acetone. This solution is named "Pesticide Surrogate for Extractions 500ppb" and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample.

10.2.2 PCB Spiking Solution – For all standard extractions, a mixture of 1016/1260 is prepared and used. The stock standards (purchased by Accustandard 1016 #APP-9-158-10X and 1260 #C260S-H-10X) are diluted in acetone to a final concentration of 5ug?mL. This solution is named "PCB 1660 LCS for Extractions 5ppm" and expires 6 months after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. The Laboratory Director and/or Organic Manager will determine if another PCB mixture is necessary, such as 1242, 1258, or 1254.

10.2.3 Pesticide Spiking Solution – A spiking solution, with a final concentration of 1ug/mL, is prepared by making a dilution of the Pesticide AB ICV Intermediate (this is made in-house by GC operators) in acetone. This solution is named "Pesticide AB LCS for Extractions 1.0ppm" and expires 2 weeks after the date it is made. Use 1.0mL of this solution per 1000mL of aqueous sample. For 608 samples, 1 out of every 10 samples must be spiked

10.2.4 TCLP- When necessary to set up a TCLP, in addition to setting up the sample, two matrix spikes must be set up and should include the following:

A. TCLP Spike 1 – This matrix spike must include a solution containing Chlordane at a concentration of 100ug/mL and Toxaphene at a concentration 10ug/mL. Both compounds are diluted in acetone from stock standards purchased from reputable vendors (Chlordane from Ultra Scientific #EPA-1086, Toxaphene from AccuStandard #P-0935-H). This solution is named "Tox/Chlor LCS for Extractions 10-100ppm" and expires 6 months from the date it is made. Add 1.0mL of leachate.

B. TCLP Spike 2 – This matrix spike must include the Pesticide Spiking Solution known as "Pesticide AB LCS for Extractions 10ppm." Add 1.0mL of this solution per 100mL of leachate.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

12.1 Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not applicable to this SOP

14.0 Procedure

14.1 All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary to extract from the following (Note: never extract samples of unknown origin without discussion with supervisor):

- 14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS
- 14.1.2 This backlog is used to determine extraction priorities based n hold times and due dates.
- 14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.
- 14.1.4 Samples are placed in LIMS "batches" based on parameter and extracted accordingly.

14.2 Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass amber jars and have a Teflon lid.

14.3 Find out if any special dilutions need to be made for client. Routine procedures for difficult matrices are listed below:

- 14.3.1 Sludge – use only 100mL and dilute to 1000mL with reagent water
- 14.3.2 TCLP Extract – use only 100mL for the sample and dilute to 1000mL with reagent water. There must be two matrix spikes of 100mL as well that are also diluted to 1000mL with reagent water.
- 14.3.3 Bad Matrix – e.g. a liquid that is partially sediment. See Organics Supervisor to find out what dilution, if any, should be made.
- 14.3.4 NPDES client – Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0-9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9 using NaOH solution or Sulfuric Acid, as necessary) or to extract the sample within 72 hours of sampling. If a pH adjustment is made, the details of the adjustment must be recorded on the sample COC and in LIMS. Set up one full list matrix spike for every ten samples.

14.4 Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume.

14.5 Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the bench sheet and later, in LIMS.

14.6 Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and a LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Follow instructions for TCLP in section 14.3.2 of this SOP. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed.

14.7 Rinse separatory funnels with methanol and discard of waste according to SOP QS14.

14.8 Pour samples into separatory funnel, placing the label from the sample bottle on the designated separatory to ensure proper identification. Use Avery labels to properly identify method blank, LCS, LCSD, any TCLPs, and TCLP spikes. If a sample requires both Pesticide and PCB analysis, a Pesticide LCS/MS/MSD (if client specified) or LCS/LCSD and a PCS LCS/MS/MSD (if client specified) or LCS/LCSD must be processed to satisfy QC requirements for the batch.

14.8.1 Due to limited volume received, it is usually necessary to use 500mL of sample to do a matrix spike so that a matrix spike duplicate can also be extracted. If only one sample container is provided for spiking purposes, use a 500mL glass cylinder to measure out half of the sample for extraction. Add half of the normal amount of spiking solution and half of the normal amount of surrogate.

14.9 Add 50mL of methylene chloride to the empty sample container, swirl, and pour into the designated separatory funnel.

14.10 Using the 1L glass graduated cylinder marked “DIH20 WATER ONLY” measure 1L of reagent water from the carboy and transfer it to the designated separatory funnels for method blank, LCS, and LCSD.

14.11 Add 50mL of methylene chloride to the method blank, LCS, and LCSD.

14.12 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set the surrogate/spike out at least ten minutes before use to allow it to warm to room temperature.

14.13 Using the 1.0mL glass syringe marked “TCMX/DCB” surrogate, add 1.0mL of TCMX/DCB surrogate to each sample, method blank, and spike. A second analyst must verify that the surrogate has been added. Enter the ID# of the standard, amount, and the initials of the analysts on the LIMS generated bench sheet and later in LIMS.

14.14 Determine if the sample will require a Pesticide spike, PCB spike, or both and proceed as follows:

14.14.1 Pesticide and PCB – Refer to 14.8 for instructions on how to determine QC requirements. To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike. To all PCB QC, add 1.0mL of PCB 1660 LCS using a glass syringe dedicated for that particular spike.

14.14.2 Pesticide only – To all Pesticide QC, add 1.0mL of Pesticide AB LCS with a glass syringe dedicated for that particular spike.

14.14.3 PCB only – To all PCB QC, add 1.0mL of PCB 1660 LCS with a glass syringe dedicated for that particular spike. 1660 is the standard PCB that we analyze for, if client specifies another PCB the extraction analyst will need to prepare another spike mix accordingly.

14.14.4 Enter the LIMS generated spike mix ID#, amount added, and the initials of the extraction and verifying analysts on the bench sheet and, later, in LIMS.

14.15 If the pH is not within 5.0-9.0 range, it must be adjusted using either the NaOH solution or Sulfuric Acid solution. If a pH adjustment is made, the details of the adjustment must be recorded in LIMS.

14.16 Seal and shake the separatory funnel vigorously for 3 minutes in the shaker apparatus with the stopcock open.

14.16.1 Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

14.17 Allow the sample to set for a few minutes, if needed, after it has been shaken. It will separate into two layers with the solvent layer on the bottom.

14.17.1 If it forms an emulsion (thick, cloudy, viscous mixture that you cannot see through), drain what you believe to be 50mL into a 250mL centrifuge bottle.

14.17.2 Save and drain into this centrifuge bottle until the extraction is complete.

14.17.3 The emulsion must be centrifuged at 2500rpm for a good separation of the water from solvent.

14.18 Drain solvent layer into an appropriately labeled 250mL beaker.

14.19 Following steps 14.16 through 14.18, extract two more times with 40mL of methylene chloride combining all solvent extracts into the same appropriately labeled 250mL beaker.

14.20 Prepare a sample vial tray with 12mL vials and vial labels printed from LIMS. These labels contain the sample number, client name, initial/final volume, parameter, and date extracted.

14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by either or both of the following two methods.

14.21.1 Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not solvent. Discard this layer.

14.21.2 Use the smallest amount possible of Sodium Sulfate by sprinkling the top layer until it hardens, separates, and drops to the bottom.

14.22 Turbo-Vap Concentration

14.22.1 Rinse a Turbo-Vap tube and arrange it underneath a methylene chloride rinsed sodium sulfate filled filter funnel.

14.22.2 Using a sharpie, label the Turbo-Vap with the sample IDs

14.22.3 Pour the extract through the filter funnel into the appropriately labeled Turbo-Vap tube.

14.22.4 Rinse the beaker three times with methylene chloride and pour through funnel.

14.22.5 Rinse the filter funnel with methylene chloride once more and allow the funnels to sit until there is no more solvent dripping.

14.22.6 For solvent exchange purposes, add 50mL of hexane to each tube. Total volume in the Turbo-Vap tube should not exceed 200mL to avoid splattering

on the lid of the Turbo-Vap. If there is a large volume of methylene chloride extract, allow the sample to condense in Turbo-Vap until 75mL-100mL are left in the turbo tube.

14.22.7 Adjust pressure of nitrogen gas tank to >30psi, making sure that the tank has 200psi or more on the main valve.

14.22.8 Record the water bath temperature in the logbook located beside the TurboVap, making sure that it is 40˚C-50˚C.

14.22.9 Place turbo-vap tube in the Turbo-Vap. Be sure to push the tube down so the tip slides into the sensor well.

14.22.10 Close the lid and push corresponding well light to start concentration.

14.23 For PCBs Only – Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows:

14.23.1 Add just enough methylene chloride to make the gel go back into solution

14.23.2 Acid clean the extract and reconcentrate.

14.23.3 Exchange with hexane again

14.23.4 If gel forms again, add enough methylene chloride to get gel back into solution

14.23.5 Transfer to a suitable container and record the final volume on the label and on bench sheet. Make sure to note the percentage of methylene chloride in sample.

14.24 When the samples reach a volume of 3mL-5mL, remove the tube from the batch

14.25 Hold the sample vial and tube in one hand at ~45˚ angle and 9" Pasteur pipette equipped with a latex bulb in the other.

14.26 Draw up sample and transfer into appropriately labeled 12mL sample vial. Be careful not to spill a drop during transfer.

14.27 Add 2-3mL of hexane to the tube and rinse several times using the pipette. Transfer this rinsate to sample vial and bring sample up to 10mL with hexane and cover the extract with a Teflon-sealed screw cap.

14.28 Take sample batch to GC Hobart sample refrigerator and log the sample numbers, analyst initials, and the date and time the samples were placed into the Hobart in the sample logbook located beside the refrigerator.

14.29 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations

Not applicable to this SOP

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

17.1 Quantity of chemicals purchased should be based on expected usage during its shelf life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not applicable to this SOP

19.0 Contingencies for Handling out-of-control or unacceptable data

Not applicable to this SOP

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition

21.2 40 CFR, Method 608

22.0 Tables, Diagrams, Flowcharts, and Validation Data

Not applicable to this SOP.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 343 **REVISION #: 01** **EFFECTIVE DATE: 20100909**

BNA & Pesticide/PCBs & TPH NON-AQUEOUS MATRIX (MICROWAVE EXTRACTION) USING SW-846 METHOD 3546

APPROVALS:

Lab Director: _____ Date: 9/9/10

Data Quality Manager: _____ Date: 9/9/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 01, 09/09/2010

- SOP has been updated to reflect the correct QS SOPs and include missing solvent/spike information.

Revision 00, 08/01/09

- Review of SOP indicated no changes were necessary
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX
(Microwave Extraction)
Using SW846 METHOD 3546

1. SCOPE AND APPLICATION

a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

2. SUMMARY

a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

3. INTERFERENCES

a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

4. APPARATUS AND MATERIALS

d. Stainless Steel spatula
e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
f. Microwave extraction Teflon tubes, capacity approximately 75mL
g. Suitable Teflon cap and screw-top lid
h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
i. Vial – 2mL clear with Teflon-lined screw cap
j. Vial – 12mL clear with Teflon-lined screw cap
k. Syringe – 1mL, 500uL
l. Pasteur pipet – 9” length
m. Pasteur pipet bulb
n. Labels – Dymo
o. Aluminum foil – heavy duty
p. Nitrogen tank – equipped with pressure regulator
q. TurboVap Concentrator with 200mL concentrator tubes
r. Teflon funnels for pouring off
s. Balance – capable of weighing to 0.1grams
t. Aluminum pie pans for mixing samples
u. Filter paper – 185mm

5. REAGENTS

a. Sodium Sulfate (Na2SO4) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
c. Hexane – suitable for spectrophotmetry and gas chromatography (Fisher #H303-4)
d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
    i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0ug/mL BN Surrogate spiking solution.)**
    ii. **BNA Spiking Solution #1 & #2 (100 ug/mL) –** The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL PAH spiking solution.) The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
    iii. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) Surrogate solution is** prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
    iv. **PCB Spiking Solution –** Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
    v. **Pesticide Spiking Solution –** A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
    vi. **TPH Surrogate –** Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
    vii. **TPH Spike –** A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

6. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
b. Samples are preserved by cooling to 4°C.
c. Holding time is 14 days from collection date to extraction.

7. PROCEDURE

a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information. (DO NOT extract samples for which you have no information.):
    i. Each day a backlog is generated in the LIMS providing all relevant sample information, including samples numbers and respective analysis required.
    ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
    iii. Check the backlog throughout the day to re-evaluate priority if needed.
b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*

- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container
Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

f. Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
g. Quantitatively transfer samples to microwave tubes. Make sure samples are loaded in the rack in the order of the bench sheet.
h. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.
i. Surrogate: **BNA** - using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **Pest/PCBs -** using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH – use the appropriate 1.0-mL glass syringe to add 1.0 mL of the appropriate surrogate to each sample, blank and spike.
j. Spiking: For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
For Pest/PCB samples, determine if the sample will require a Pesticide

Spike and/or a PCB Spike. Proceed as follows:
Pesticide and PCB - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.
Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.
PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB.

For TPH - To the sample in each analytical batch selected for spiking, add 1mL of the appropriate spiking solution (i.e. DRO or TNEPH or MAEPH) using a 1.0 mL glass syringe dedicated to that spike.

k. **Solvent:** Add 30mL methylene chloride for BNA/PAH/TPH extractions or 30ml hexane for Pest/PCB extractions.
l. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
m. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave.
n. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
o. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications.

For 1-15 samples:
Max power: 800W 100%
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

For 16-40 samples:
Max power: 1600W 100%
Ramp time: 15:00
Control temperature (in Celsius): 110
Hold time: 10:00
Cool down: 5:00

p. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
q. Transfer the extract to a pre-rinsed turbo vap tube by first passing through

a funnel with P4filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with the extraction solvent and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of the extraction solvent using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.

r. Now concentrate the extract to 1.0mL using the turbovap concentrator.
    i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45°C. The pressure target range should be about 20-25 psi.
    ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
    iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
s. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

8. DOCUMENTATION OF CAPABILITY (DOC)

a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS08 for guidance.

9. WASTE MANAGEMENT AND POLLUTION PREVENTION

a. Please see Waste Disposal SOP QS14 for the proper disposal of waste generated from this area.
b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

10. METHOD PERFORMANCE

a. Refer to SOP-201, SOP-211 and SOP-219 for method performance.

11. REFERENCES

a. EPA Methods SW-846, Method 3546

12. DEFINITIONS

a. Refer to SOP QS08 for definitions.

13. HEALTH AND SAFETY

a. Wear appropriate personal protection equipment when working with chemicals or samples.
b. Use the lab hoods when working with solvents.
c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER: SOP-405

REVISION NUMBER: 5

APPROVED BY:

LAB DIRECTOR

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: 06/23/09

DATE OF LAST REVIEW: 06/23/09

Analytical Laboratory Waste Disposal
Standard Operating Procedure

I. SCOPE AND APPLICATION:

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients.

   a. Unused aliquots of completed water samples.

   b. Unused aliquots of completed non-aqueous samples.

2. Soils from quarantined areas

3. All other soils, sediments, building debris, wipes etc.

4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client.

2. Throw the sample away after confirming that it is non-hazardous.

3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.

4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

For example: Solvent waste must be stored in the vented flammable cabinet.

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).

B. USDOT approved drums for storing and shipping hazardous waste.

C. Fume hood vented outside the building.

D. Flammable storage cabinet which is vented to the outside

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.

a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.

b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.

c. **be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**

d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.

e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.

f. All samples disposed of in this manner must be documented in the bound disposal log.

2. If water samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.

c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

B. Disposal of completed non-aqueous samples:

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for two hours.
b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this manner must be documented in the bound disposal logbook with the following information:**

a. Client
b. Sample #s

c. Date(s) treated
d. Treatment method used

C. Disposal of laboratory generated waste:

Generated waste is stored outside the building, inside the caged fence until a waste pick up occurs. This area must be maintained properly.

1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, **(>2% by volume) and** dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**.
- Dilute acid waste **(<2% by volume or less)** are neutralized using concentrated amounts of sodium hydroxide and poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated along with the amount of sodium hydroxide used to neutralize the acid waste, is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed

below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards **(>2% by volume)** are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ....etc...) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type and quantity of solvent is entered into the *'Organic Solvent Waste Logbook'* located on the shelf next to the drums.**

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located in the GC/HPLC Laboratory.

PCB Box – all samples/standards
Pesticide Box – all samples/standards
Herbicide Box – all samples/standards
8330 Box – all samples/standards
Methylene Chloride Box- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial.

Acid cleaned extracts are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

Unused stock and working standards are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

Waste from the instrument - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain(pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and dispose of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

Standards - Unused stock and working standards are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company**.

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.

- Hazardous or product samples are returned to the client.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Officer, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence (c. *Organic Extraction Laboratory Area*, * **Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.

2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.

3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.

4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

DISTRIBUTION/TRAINING LOG

ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER: **SOP-405**

REVISION NUMBER: **5**

RECEIVED BY/DATE:

W. Schwab	
R. Townsend	
F. Rivers	
J. Holliman	
B. DeVille	
A. Monteiro	
B. Richard	

Signature above signifies acknowledgement of responsibility to know and follow the contents of this document. It also signifies receipt of training covering all new aspects of the SOP.

TRAINED BY:

EFFECTIVE DATE: **06/23/09**

PLEASE COLLECT OLD SOPs AND RETURN WITH SIGNED FORM TO QAO

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS10 **REVISION #: 14** **EFFECTIVE DATE: 20100907**

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

Revision 14, 09/07/10

- The SOP combines SOPs 404, 406, 410, 415 and 432 into one SOP with updated naming.

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1.0 Sample Acceptance Criteria

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. DoD certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Project Manager or your section manager immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

1.1 A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1.1.1 Sample must be properly preserved and in the proper container for the requested analysis.

1.1.2 Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.

1.1.3 Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).

1.1.4 The sample ID on the bottle label must match the sample ID listed on the chain of custody.

1.1.5 The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.

1.1.6 Sample temperature must be less than 6°C or received on ice.

1.1.7 Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

2.0 Sample Receiving

2.1 Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.

2.1.1 The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2.1.2 Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment II*] is used

the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

2.2 Visually inspect all coolers for tampering, custody seals, (intact if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express air bills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

2.3 Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. Your signature and the date and time the samples were received must be placed onto the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined (for leakage, breakage etc.), the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

2.3.1 Attach any shipping receipts, work orders, documentation, etc. to the chain of custody.

2.3.2 If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.

2.3.3 Project specific paperwork may be required. For all projects, a *Cooler Receipt Form [Attachment IV]* must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.

2.4 The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer quarterly and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.

2.4.1 To measure the temperature, point the IR thermometer at the cooler temperature blank (if supplied) or a direct sample and wait a few seconds for the temperature to stabilize. The IR gun should be held 6 inches away (from the temperature blank or sample) for an accurate reading. Read the temperature to the nearest 0.1 °C.

The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that does not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.

2.4.2 If the temperature exceeds 6°C for any sample, the project manager must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Project Manager and/or Laboratory Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision.

The only exceptions to the 6°C rule are:

2.4.2.1 Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO_3 to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*

2.4.2.2 Samples for Fluoride, Chloride and Bromide.

2.4.2.3 Waste/Product samples for all parameters.

2.4.2.4 Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.

2.4.2.5 Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are “Received on Ice” (ROI).

2.5 If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

2.5.1 Any analyses which have a 24-72 hour holding time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.

2.5.2 Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in sample receiving.

2.5.2.1 If a sample is received already out of holding time, this must be documented and the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.

2.5.2.2 If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc.

2.5.3 Samples requiring rush turnaround.

2.5.3.1 If sample(s) require 24 or 48-hour turnaround they will take first priority. Other rush requests also have high priority.

2.5.3.2 The project manager and/or section manager must be contacted for approval concerning any unscheduled rush requests.

2.6 Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

2.6.1 Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.

2.6.2 Check for leakage and sample container breakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. The Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.

2.7 Check the chain of custody information against the information recorded on the containers. If these do not agree, this must be documented and the Project Manager must be notified.

2.7.1 If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2.7.2 Any error found on the chain of custody must be marked through with one line, initialed, dated and the correction written in.

2.8 Note any unusual requests, methodology, hazards (known or suspected) to the project manager and/or laboratory section manager or analysts before the samples are actually logged in. Make notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.).

3.0 Sample Log In

3.1 After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. Contact the project manager if there are any questions, problems, etc.

3.2 Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody

3.2.1 All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.).

3.2.2 Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3.2.3 Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).

3.3 Check the following items and record this information on the cooler receipt form to further ensure sample integrity. If any of the following requirements are not met it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

3.3.1 Determine if the samples were received at the proper temperature.

3.3.2 The sample descriptions on the bottle should match those on the chain of custody.

3.3.3 Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that does not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound

notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO3 used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.

3.3.3.1 The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in. This information is then documented on the project cooler receipt form.

3.3.3.2 The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper rather than a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

3.3.3.3 When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH, Electrometric.]

3.3.4 The following guidelines must be followed to check pH preservation:

3.3.4.1 Water samples for Cyanide analyses must be preserved to a pH of >12.0 with NaOH upon collection. If the pH of these samples is <12.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to >12.0.

3.3.4.2 Water samples for Metals analyses must be preserved to a pH of <2.0 with HNO_3 upon collection. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.

3.3.4.3 Samples requiring analyses which are preserved with H_2SO_4 (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) should be preserved to have a pH of <2.0. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0. Samples for sulfide analysis must have a pH >9.

3.3.4.4 If a sample is not properly preserved, log-in personnel must either do the following:

3.3.4.4.1 To meet project specific requirements, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required.

3.3.4.4.2 For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required.

3.3.4.4.3 All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. The client must be notified to see if the lab is to proceed with analysis.

3.3.4.5 In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. Make sure you note on the cooler receipt form and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

3.3.4.6 Samples may be generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required.

3.3.5 Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

3.3.6 If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted. Large bubbles or head space is not acceptable and this information must be documented on the cooler receipt form. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with $Na_2S_2O_3$ (0.2g) when chlorine is known to be, or suspected to be present.

3.3.7 All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add ascorbic acid approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included on the cooler receipt form. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.

3.3.8 Be aware of holding time requirements.

3.4 Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested.

4.0 Sample Storage

4.1 After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***

4.1.1 The refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

4.1.2 Walk in Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.

4.1.3 Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

4.1.4 VOC Soil Freezer: All soil samples requiring VOC analysis with short hold prep times (Encores, Organic Free Water Terracores, etc.) must be stored in this freezer.

4.1.5 VOC Dry Storage Rack: All water VOCs that have exceeded double holding time can be stored on this rack. These samples are stored here segregated alone to ensure no cross contamination occurs between VOC samples and other non-VOC aqueous samples.

4.2 Quarantined soils are those quarantined by the US Department of Agriculture. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing. Quarantined soils are defined as:

4.2.1 Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately*. A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.

4.2.2 All soils taken from foreign sources, US. Territories and Hawaii.

NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.

4.3 All samples must be stored in one of the four refrigerators detailed above with the following exceptions:

4.3.1 Matrices that may be adversely affected by the cold temperature. (E.g. surfactant samples, multi-phase samples).

4.3.2 Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the project manager for other options.

4.4 The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year **(digital thermometers quarterly)** and this information recorded in a bound notebook. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. If the temperature exceeds this range, corrective action measures must be put in place immediately. The Wet Chemistry Manager, Organic Manager, and Laboratory Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

4.5 All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IX]*** which is attached to the door of each refrigerator. These completed forms are kept on file. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

4.6 The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week.

4.7 As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.

4.8 A temperature maintenance record book is kept for each refrigerator.

4.9 Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 45 days after the final report unless otherwise specified. See SOP QS14 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

5.0 Laboratory Information Management System (LIMS)

5.1 Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

5.1.1 Client: Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

5.1.2 Project: Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should populate. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

5.1.2.1 Internal – Empirical Laboratories projects;

5.1.2.2 External – direct laboratory clients.

5.1.3 Comments: This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning

rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, preservation or matrix problems, etc.

5.1.4 Received By: Enter the name of the person who received the samples.

5.1.5 Logged In By: Enter the name of the person who logged in the samples.

5.1.6 Received: Enter the date and time received separated by a space and using military time. Example: *08/02/2008 08:30*.

5.1.7 Project/Package Date Due: After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

5.1.8 Shipping Containers: Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

5.1.9 COC Number: If an identifiable COC number is listed, record that ID here.

5.1.10 Shipped By: Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped off by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

5.1.11 Sample Name:

5.1.11.1 Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

5.1.11.2 If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

5.1.11.3 When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # _____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks.

5.1.12 Collection Date: Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:00 can be used temporarily until client

verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, this must be documented on the cooler receipt form and the project manager must be notified. All attempts should be made to get all our clients to supply this information.

5.1.13 Lab/Report Matrix: Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.

5.1.14 Sample Type: This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

5.1.15 Container: Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

5.1.16 pH (Container Preservative): Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

5.1.17 Comments: Enter any information that is applicable at the sample level.

5.1.18 Field Analysis: Click on field analysis tab and enter field information when provided.

5.1.19 Work Analyses: Select all parameters requested for the sample from this list.

5.1.19.1 If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

5.1.19.2 If any analyses are selected from the All Analyses list, the project manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.

5.1.19.3 All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in

the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

5.1.20 Analyses Comments: These comments should be used for any notes that only apply to that particular test code.

5.1.21 RTAT: If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

5.1.22 Save: Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

5.1.22.1 A 2-digit numeral of the year. Example (**08**11248-06).

5.1.22.2 A 2-digit numeral of the month. Example (08**11**248-06).

5.1.22.3 A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06).

5.1.22.4 A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order.

5.1.23 Add/Edit/Copy: Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

5.2 After log-in of a work order is complete, the COC can then be scanned into the system and attached to the work order on the Work Order screen. The work order then must be updated to Available status so as to be seen by the analysts.

6.0 Daily Follow Up for Sample Receiving/Log In

6.1 Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room.

6.2 At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Managers:

6.2.1 The original chains of custody and yellow original or copy of each. The Cooler Receipt Forms will accompany the COC for the project.

6.2.2 Any information (letters, regulatory limits, etc.) from a client which was received with any samples.

6.3 All the above information from the day will be reviewed as soon as possible.

6.3.1 If any corrections or changes are required, all laboratory personnel will be notified by distributing a ***Sample Log Change Form [Attachment XIII]*** through email distribution. A ***Sample Log Change Form*** by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

6.4 Sample Receiving will distribute the following to the appropriate laboratory personnel:

6.4.1 Copies of the LIMS receiving reports to necessary laboratory personnel.

6.4.2 Original (white copy) chains of custody are given to the project manager.

6.4.3 Copies of any project/sample specific information to the Section Manager and analysts.

6.5 Information will be filed as follows:

6.5.1 Chains of custody:

6.5.1.1 Original (white copy) is returned to the customer with the final report along with the CRF.

6.5.1.2 Pink copies should be retained by the sampler.

6.5.2 Sample Log Change Forms

6.5.2.1 Sample Log Change Forms are distributed through email to all laboratory personnel.

7.0 Miscellaneous

7.1 All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

7.2 If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

7.3 Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL manager. The samples are then logged in by ATL personnel.

7.4 All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Sample Receiving Group Leader will review the log books each week to check for completeness.

Log Book ID	Log Book Description
LI014	Trip Blank Prep Log Book
LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI015	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

8.0 Sample Storage, Secure Areas and Sample Custody

8.1 Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

8.2 Log-in is also responsible for maintaining a Sample Receiving Custody and Disposal Form for samples received. This form is to be filled out before the actual disposing of any sample in house. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- 8.2.1 Client and Log #s
- 8.2.2 Date/Time Unpacked
- 8.2.3 Logged In/Numbered By (Initials)
- 8.2.4 2nd Checked By (Initials)
- 8.2.5 Date/Time Placed in Cold Storage
- 8.2.6 Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- 8.2.7 Disposed of By/Date
- 8.2.8 Method of Disposal

8.3 Sample extracts and digestates are stored in the following areas:

- 8.3.1 All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.
- 8.3.2 Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.
- 8.3.3 Extracts from medium level VOC analyses are also stored in the Soil Walk – in or VOC sample freezer in the VOC Lab.
- 8.3.4 All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

8.4 The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure (SOP QS14).

The following personnel as of September 1st, 2010 have access to all sample storage areas:

Amy Barnett	Antonio Monteiro
Betty Deville	Brian Richard
Chase Block	Christy Thompson
Crystal Brand	Dahae Kim
Christine Gramada	Delia Weber
Eric Coburn	Franklin Rivers

Gary Quick	Gwen Hallquist
Herbie Johnson	Jade Holliman
Jessica Sales	Karu Huka
Kendra Gentry	Marcia McGinnity
Margarett Pitt	Mark Cobb
Melanie Sams	Melynda Nelson
Penny Cormier	Rachael Mahan
Randy Ward	Renee Vogel
Rick Davis	Roger Burr
Russell Townsend	Sonya Gordon
Veronica Mullen	William Lancaster
William Schwab	Sabina Kemp

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

9.0 Sample Custodian's Duties and Responsibilities

9.1 The Sample Custodian is responsible for the receiving; log in, tracking and disposal of all samples. The duties of this position are performed by the persons in the sample receiving section of the laboratory. These individuals are the primary custodian, secondary custodian and section supervisor. Although other laboratory personnel may assist with the duties, this is done under supervision and direction of one of the three individuals listed above. The sample custodians are responsible for the following:

9.1.1 Receive all samples for the analytical laboratory and maintain chain of custody. This includes documenting the validated time/date of receipt.

9.1.2 Maintain the flow of samples through the log in process and make them available to the analysts on a timely manner. This includes prioritizing samples/projects based on turnaround requests and holding times.

9.1.3 Assign the correct laboratory ID sample numbers and validate that this information is properly labeled on the containers and entered into the Laboratory Information Management System (LIMS).

9.1.4 Validate that every sample proceed through all steps of the log in process. This includes checking the following to determine that the sample integrity has been upheld from the time the sample is collected until it is received in the laboratory: proper containers with ample sample volume, correct preservation, sample dates/times to ensure that holding times can be met, condition of the sample containers, headspace of vials for VOC analysis, sample ID discrepancies and completeness of the chain of custody.

9.1.5 Communicate any information or specific requests by the client that are listed on the chain of custody, i.e., method information, detection limits, specific analytes, reporting information, turnaround information, potential hazards etc. They are also responsible for forwarding any additional information that may be received

along with the samples, i.e. permit or regulatory information, letters, etc. to the laboratory managers.

9.1.6 The sample custodian is personally responsible for continuing to uphold the sample integrity throughout the log in procedure and until the time when the samples are properly stored and disposed.

9.1.7 Ensure that samples are transferred into the proper storage area and that these secure areas are locked after hours.

9.1.8 Maintain all log books used in the section. These must be kept up to date, complete, neat and orderly.

9.1.9 Maintain the sample receiving and sample disposal areas in a clean, orderly and safe manner.

9.1.10 Follow good laboratory practices and safety procedures.

9.1.11 Communicate all problems, discrepancies, etc. to the section supervisor and laboratory Director.

9.1.12 In situations where the client cannot be contacted, the sample custodian along with the section supervisor must apply the best judgment on how to handle the samples or situation.

9.1.13 Complete all the necessary paperwork and section forms including Cooler Receipt Forms, LIMS daily print outs, Sample Receiving Custody and Disposal Form, etc. in a timely manner.

9.1.14 Dispose of all samples in a manner that is safe, cost efficient, timely, meets project requirements and is in accordance with hazardous waste regulations.

9.1.15 The sample custodian(s) are responsible for compliance of all procedures outlined in this SOP and the following SOPs. They must maintain personal copies of each SOP:

9.1.15.1	SOP QS10	Laboratory Sample Receiving, Login and Storage
9.1.15.2	SOP QS14	Analytical Laboratory Waste Disposal
9.1.15.3	SOP QS11	Field Sampling & Bottle Kit Preparation

10.0 Procedure for Treatment of Soil Samples from Quarantined Areas

10.1 This summary is to explain the handling and treatment of soil samples that come from USDA quarantined areas of the United States, territories of the United States and foreign sources. This treatment is done to prevent the spread of pests to other areas.

10.1.1 When soil samples are ready for disposal, separate out soils that are from quarantined areas that need to be treated. Quarantined areas are from the southern United States (see attached maps), from United States territories such as Puerto Rico, and from foreign countries.

10.1.2 Only quarantined non-hazardous soil samples with containers that are less than three feet in depth will be treated by this procedure. Hazardous samples will have to be treated differently. A list of samples to be treated will be determined by the login supervisor.

10.1.3 Log the samples to be treated in the Soil Treatment Logbook as to location, date, and quantity.

10.1.4 Turn the oven on. Place soil samples in their containers uncovered in the oven. After oven reaches 121°, heat samples for 2 hours. Treat container liners too. When time is up, remove soil samples with gloves or tongs and cool.

10.1.5 After samples have cooled, put them in the non-hazardous soil barrel for disposal.

11.0 Subcontracting Laboratory Samples

11.1 Sample receiving is responsible for handling all aspects of shipment of subcontracted samples. Once samples have been confirmed as sub-outs, login then notifies the project manager that subout samples are in house. The project manager then generates a purchase order number for the specific subout samples. Once the purchase order is generated by the project manager, then login prints out a subcontracted chain of custody from LIMS that will accompany the subout samples during transit. Then login packs up the samples into a cooler, ices them down (if necessary) to keep the samples chilled during transit, and then the cooler is shipped to the subcontracted laboratory.

11.2 Chain of Custody/Shipping Requirements

11.2.1 When the samples are sent out, a completed chain of custody must be sent with the samples. Make sure to include the following information:

11.2.1.1 Be specific in your analyses request. List the method number if applicable and/or any specific analytes required. This should already have been discussed with the laboratory.

11.2.1.2 List the name of sub contract laboratory and the date shipped or delivered.

11.2.1.3 List the Empirical Laboratories; LLC LIMS log # as the sample description on the chain of custody. Do not list the actual client name or actual project information.

11.2.1.4 Record the date and time that the samples were sampled on the chain of custody.

11.2.1.5 Results and invoice should be sent to the project manager.

11.2.2 Two copies of the sub contract chain of custody should be retained. One copy should be stapled to the original chain of custody received from the client and the other should be stapled to the copy in log in.

11.2.3 Make sure samples are packed well so they will not break or spill in shipment. Ice must be packed in the cooler to keep the samples cold if chilling is required.

11.2.4 A P.O. must be completed and approved by the project manager prior to sample shipment. Sample receiving should then keep a copy of this P.O. for their records.

Attachments to QS10

I	Chain of Custody Record
IV	Cooler Receipt Form
V	List of Short Holding Time Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the U.S.
IX	Laboratory Sample Custody Form for Walk in Refrigerator
X	Container Codes for the LIMS
XIII	Sample Log Change Form (Green Sheet)

[Attachments II, III, VI, XI, XII, and XIV were removed during the editing process and not added to the QS.]

Attachment I

EMPIRICAL LABORATORIES, LLC - CHAIN OF CUSTODY RECORD

SHIP TO: 227 French Landing Drive, Suite 550 ♦ Nashville, TN 37228 ♦ 615-345-1115 ♦ (fax) 615-846-5426

Send Results to:	**Send Invoice to:**	**Analysis Requirements:**	**Lab Use Only:**			
Name _____	Name _____		VOA Headspace	Y	N	NA
Company _____	Company _____		Field Filtered	Y	N	NA
Address _____	Address _____		Correct Containers	Y	N	NA
City _____	City _____		Discrepancies	Y	N	NA
State, Zip _____	State, Zip _____		Cust. Seals Intact	Y	N	NA
Phone _____	Phone _____		Containers Intact	Y	N	NA
Fax _____	Fax _____					
E-mail _____	E-mail _____		Airbill #: _____			
Project No./Name:	**Sampler's (Signature):**		CAR #: _____			

Lab Use Only Lab #	Date/Time Sampled	Sample Description	Sample Matrix	Comments	No. of Bottles	*Lab Use Only* Containers/Pres.

Sample Kit Prep'd by: (Signature)	Date/Time	Received By: (Signature)	REMARKS:	Details:
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Page _____ of _____
Relinquished by: (Signature)	Date/Time	Received By: (Signature)		Cooler No. ____ of _____
Received for Laboratory by: (Signature)	Date/Time	*Temperature*		Date Shipped _____
				Shipped By _____
				Turnaround _____

Distribution: Original and yellow copies accompany sample shipment to laboratory; Pink retained by samplers

Attachment IV

EMPIRICAL LABORATORIES
COOLER RECEIPT FORM

LIMS Number: _____ Number of Coolers: _____ of _____

Client: _____ Project: _____

Date/Time Received: Date cooler(s) opened:

Opened By (print): (signature): _____

Circle response below as appropriate

1. How did the samples arrive? FedEx UPS DHL Hand Delivered
EL Courier Other: _____

If applicable, enter airbill number here: _____

2. Were custody seals on outside of cooler(s)? ..... Yes No

How many: _____ Seal date: _____ Seal Initials: _____

3. Were custody seals unbroken and intact at the date and time of arrival?	Yes	No	N/A
4. Were custody papers sealed in a plastic bag included in the sample cooler?	Yes	No	N/A
5. Were custody papers filled out properly (ink, signed, etc.)?	Yes	No	N/A
6. Did you sign custody papers in the appropriate place for acceptance?	Yes	No	N/A
7. Was project identifiable from custody papers?	Yes	No	N/A
8. If required, was enough ice present in the cooler(s)?	Yes	No	N/A

Type of Coolant: WET DRY BLUE NONE Temperature of Samples upon Receipt: _____ °C

Dates samples were logged-in:

9. Initial this form to acknowledge login of sample(s): (Name): _____ (Initial): _____

10. Were all bottle lids intact and sealed tightly?	Yes	No	N/A
11. Did all bottles arrive unbroken?	Yes	No	N/A
12. Was all required bottle label information complete?	Yes	No	N/A
13. Did all bottle labels agree with custody papers?	Yes	No	N/A
14. Were correct containers used for the analyses indicated?	Yes	No	N/A
15. Were preservative levels correct in all applicable sample containers?	Yes	No	N/A
16. Was residual chlorine present in any applicable sample containers?	Yes	No	N/A
17. Was sufficient amount of sample sent for the analyses required?	Yes	No	N/A
18. Was headspace present in any included VOA vials?	Yes	No	N/A

If Non-Conformance issues were present, list by sample ID: _____

_____ CAR#: _____

ATTACHMENT V

Short Holding Time Parameters

(Immediate-72 hours)

Parameter	Holding Time
pH	Immediate [a]
Sulfite	Immediate [a]
Temperature	Immediate [a]
Residual Chlorine	Immediate [a]
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) *Drinking Water only*	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours [b]

[a] Immediate generally means within 15 minutes of sample collection.

[b] This is an internal holding time. The method does not specify a holding time.

Attachment VII

Empirical Laboratories, LLC

SAMPLE RECEIVING CUSTODY AND DISPOSAL FORM

Date Samples Received _____

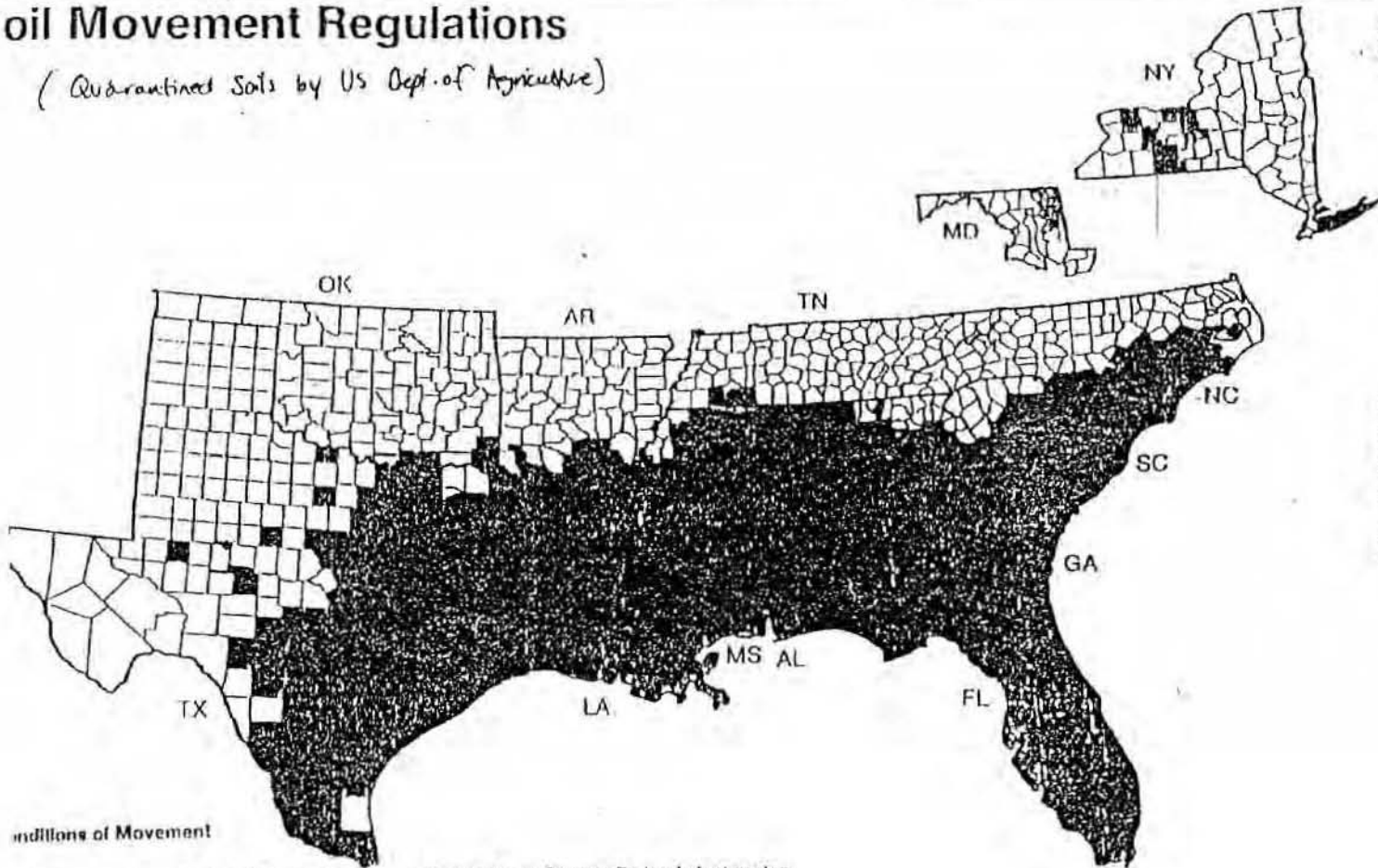
Client	Log #'s	Date Unpacked	Logged In/ Numbered By	2nd Checked By	Placed in Storage By	Date Placed in Storage	Storage Area (1)	Disposed of By/Date	Method of Disposal

(1) W=Walk in Refrigerator
HV= Hobart Refrigerator, VOC samples only
Q= Refrigerator for Quarantined Soil Samples
QV= Refrigerator for Quarantined Soil, VOC samples only
O=Other (Must Specify)

ATTACHMENT VIII

oil Movement Regulations

(Quarantined Soils by US Dept. of Agriculture)



nditions of Movement

strictions are imposed on the move-
nt of regulated articles from a regulated
a as follows:
om red areas into or through white
eas. Movement within red areas may be
gulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

Regulated Area

Attachment IX

Empirical Laboratories

EMPIRICAL LABORATORIES, LLC
LABORATORY SAMPLE CUSTODY FORM
WALK-IN REFRIGERATOR

Sample Log # (s)	Time/Date/Initials Removed	Time/Date/Initials Returned (Note if all Sample Used)	Notes/ Comments	Task Performed

Attachment X

Preservatives		Types of Container	
NI	*HNO3*	A	*1 LITER - PLASTIC*
NF	*HNO3 (Filtered)*	B	*500 mL - PLASTIC*
SU	*H2SO4*	C	*250 mL - PLASTIC*
SH	*NaOH*	D	*120 mL - PLASTIC*
ZN	*ZnAC / NaOH*	EN	*ENCORE PAK*
HY	*HCl*	F	*1 LITER - GLASS CLEAR WIDE MOUTH*
		G	*1 LITER - GLASS CLEAR BOSTON ROUND*
		H	*1 LITER - GLASS AMBER*
		I	*250 ml. - AMBER*
		J	*VOA VIALS - (40 ml.)*
		K	*500 ml. - (16 oz)*
		L	*250 ml. - (8 oz)*
		M	*125 ml. - (4 oz)*
		N	*60 ml. - (2 oz)*
		O	*OTHER*
		P	*PLASTIC BAG -1 Gallon*

Attachment XIII

SAMPLE LOG CHANGE FORM

DATE:

Workorder:

CLIENT:

PARAMETERS:

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

QUALITY SYSTEMS: QS14 REVISION #: 06 EFFECTIVE DATE: 20100831

ANALYTICAL LABORATORY WASTE DISPOSAL

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

9/9/10

Changes to this Revision – R06 08/31/2010

- Revision to SOP405 R05 dated 6/23/2009.
- Changed the document control and named this as QS14 R06.
- Minor cosmetic/grammatical changes made.

Analytical Laboratory Waste Disposal
Standard Operating Procedure

I. SCOPE AND APPLICATION:

Laboratory waste includes excess client sample waste and waste that is generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients
   a. Unused aliquots of completed water samples
   b. Unused aliquots of completed non-aqueous samples
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes, oils, etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client
2. Disposal of samples after confirming that they are non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until transported off site.
2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

A. Proper safety equipment should be in good working condition. This includes gloves, lab coats, safety glasses/goggles, and face shields. Voluntary use of cartridge respirators is allowed (see area manager or QAO).

B. USDOT approved drums for storing and shipping hazardous waste.

C. Fume hoods.

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Safety Coordinator.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for a minimum of 45 days from receipt and sample extracts are held for 90 days minimum from receipt. Engineering support projects involving CLP work, litigation cases, etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point, the area manager and/or analyst will communicate information about samples deemed as hazardous**.

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.
   a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.
   b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat, and gloves.
   C. **Be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor, etc. Check with the area manager or Safety Coordinator before disposing of any questionable samples.**
   d. Tap water must be running when samples are poured out for approximately 10 minutes in order for sufficient flushing and dilution to take place.
   e. All containers must be rinsed out and thrown into the trash.
   f. All samples disposed of in this manner must be documented in the bound sample disposal logbook.

2. If water samples are hazardous (known or suspected), one of the following steps must be taken:
   a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vendor.**

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
c. Store the sample properly until either a sealed drum or lab pack is sent out.
d. All samples disposed of in this manner must be documented in the bound sample disposal logbook.

B. Disposal of completed non-aqueous samples:

The majority of non-aqueous samples are soils and sediments. Although there may also be building debris, wipes, oils**,** and occasionally product type samples.

1. If samples are non-hazardous, they are placed in a sealed drum and destroyed. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.
2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.
    a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations**. If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**
    b. Store the sample properly until a lab pack is sent out.
3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:
    a. The sample is heated to 180°C (356°F) in a vented oven for two hours.
    b. After heating the samples they are removed from the oven to cool and then placed in a sealed drum and destroyed.
4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples**. All samples disposed of in this manner must be documented in both the bound sample receiving disposal logbook, and soil quarantine logbook with the following information:**
    a. Client
    b. Work Order/Sample #s (from LIMS)
    c. Date(s) treated
    d. How much sample volume (in ounces) was treated

C. Disposal of laboratory generated waste:

Generated waste is stored outside the building until a waste pick up occurs. This area must be maintained properly.

1. Waste handling and disposal within each laboratory section:

NOTE: Each laboratory analyst and section manager is responsible in assuring that **handling** operations (within their area) are being followed according to the laboratory requirements.

a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety coordinator.

- Concentrated acid waste, **(>2% by volume) and** dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**.
- Dilute acid waste **(<2% by volume or less)** are neutralized by using concentrated amounts of sodium hydroxide. Once the pH of the acid waste is neutralized, the acid waste is then poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated, the amount of sodium hydroxide used to neutralize the acid waste, final pH of the acid waste, date performed, and date disposed of is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents, and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety coordinator.

- Concentrated acid waste, aqueous sample waste digestates, and old unused calibration standards **(>2% by volume)** are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and**

amount of waste in the acid waste logbook, then initial and date the entry

- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**
- **Throw the soil/solids in the trash once the acid has been rinsed free**.
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum. Document the type and amount of waste in the acid waste logbook, then initial and date the entry**

****Note: The laboratory '*Acid Waste Logbook*' is located in Extractions.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the coordinator.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ….etc…) is poured into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) is poured into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are**

located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type, and quantity of solvent is entered into the '*Organic Solvent Waste Logbook*' located in Extractions.

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until evaporated. Then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory:

- Autosampler vials are discarded into the appropriate buckets located in the GC/HPLC Laboratory.
- Sample and spike extract vials are separated according to the contents in the vial. **Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory and then transferred to the appropriate satellite drum when deemed necessary. **Document the type and amount of waste into the appropriate logbook, then initial and date the entry.**
- **Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- **Volatile sample, standard, and reagent waste:**

Instrument Waste - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain (pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and disposed of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area (see A and B listed above). Lachat instrument waste and COD waste is collected and poured into the acid satellite drum when deemed necessary. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**

Standards - Unused stock and working standards are discarded into the chlorinated solvent waste bottle in the hood located in extractions. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste, unused aqueous and unused solid samples (see procedures A and B listed above).

- **Semi-volatile sample and standard waste disposal:**

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in extractions. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

Auto sampler vials are collected in buckets, and then either consolidated in lab packs or the contents are transferred into the appropriate waste drums.

Lab packs for disposal are done by a licensed waste disposal company.

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.
- Hazardous or product samples are returned to the client.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Coordinator, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence. A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.
2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.
4. The Safety Coordinator will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

   It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

American Association for Laboratory Accreditation

World Class Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

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ENVIRONMENTAL

Valid To: May 31, 2012 Certificate Number: 3014.01

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 DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1)) 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

High Resolution Gas Chromatography / Mass Spectrometry

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,3,7,8-Tetrachlorodibenzo-p-dioxin	EPA 1613B	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
2,3,7,8-Tetrachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8-Pentachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
2,3,4,7,8-Pentachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8-Hexachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,6,7,8-Hexachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
2,3,4,6,7,8-Hexachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,7,8,9-Hexachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,6,7,8-Heptachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,7,8,9-Heptachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
1,2,3,4,5,6,7,8-Octachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Tetrachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Pentachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Hexachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Heptachlorodibenzo-p-dioxin	----------	EPA 1613B/8290A	EPA 1613B/8290A

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Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
Total Tetrachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Pentachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Hexachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
Total Heptachlorodibenzofuran	----------	EPA 1613B/8290A	EPA 1613B/8290A
2-Chlorobiphenyl (1)	----------	EPA 1668A	EPA 1668A
3-Chlorobiphenyl (2)	----------	EPA 1668A	EPA 1668A
4-Chlorobiphenyl (3)	----------	EPA 1668A	EPA 1668A
2,2'-Dichlorobiphenyl (4)	----------	EPA 1668A	EPA 1668A
2,3-Dichlorobiphenyl (5)	----------	EPA 1668A	EPA 1668A
2,3'-Dichlorobiphenyl (6)	----------	EPA 1668A	EPA 1668A
2,4-Dichlorobiphenyl (7)	----------	EPA 1668A	EPA 1668A
2,4'-Dichlorobiphenyl (8)	----------	EPA 1668A	EPA 1668A
2,5-Dichlorobiphenyl (9)	----------	EPA 1668A	EPA 1668A
2,6-Dichlorobiphenyl (10)	----------	EPA 1668A	EPA 1668A
3,3'-Dichlorobiphenyl (11)	----------	EPA 1668A	EPA 1668A
3,4-Dichlorobiphenyl (12)	----------	EPA 1668A	EPA 1668A
3,4'-Dichlorobiphenyl (13)	----------	EPA 1668A	EPA 1668A
3,5-Dichlorobiphenyl (14)	----------	EPA 1668A	EPA 1668A
4,4'-Dichlorobiphenyl (15)	----------	EPA 1668A	EPA 1668A
2,2',3-Trichlorobiphenyl (16)	----------	EPA 1668A	EPA 1668A
2,2',4-Trichlorobiphenyl (17)	----------	EPA 1668A	EPA 1668A
2,2',5-Trichlorobiphenyl (18)	----------	EPA 1668A	EPA 1668A
2,2',6-Trichlorobiphenyl (19)	----------	EPA 1668A	EPA 1668A
2,3,3'-Trichlorobiphenyl (20)	----------	EPA 1668A	EPA 1668A
2,3,4-Trichlorobiphenyl (21)	----------	EPA 1668A	EPA 1668A
2,3,4'-Trichlorobiphenyl (22)	----------	EPA 1668A	EPA 1668A
2,3,5-Trichlorobiphenyl (23)	----------	EPA 1668A	EPA 1668A
2,3,6-Trichlorobiphenyl (24)	----------	EPA 1668A	EPA 1668A
2,3',4-Trichlorobiphenyl (25)	----------	EPA 1668A	EPA 1668A
2,3',5-Trichlorobiphenyl (26)	----------	EPA 1668A	EPA 1668A
2,3',6-Trichlorobiphenyl (27)	----------	EPA 1668A	EPA 1668A
2,4,4'-Trichlorobiphenyl (28)	----------	EPA 1668A	EPA 1668A
2,4,5-Trichlorobiphenyl (29)	----------	EPA 1668A	EPA 1668A
2,4,6-Trichlorobiphenyl (30)	----------	EPA 1668A	EPA 1668A
2,4',5-Trichlorobiphenyl (31)	----------	EPA 1668A	EPA 1668A
2,4',6-Trichlorobiphenyl (32)	----------	EPA 1668A	EPA 1668A
2',3,4-Trichlorobiphenyl (33)	----------	EPA 1668A	EPA 1668A
2',3,5-Trichlorobiphenyl (34)	----------	EPA 1668A	EPA 1668A
3,3',4-Trichlorobiphenyl (35)	----------	EPA 1668A	EPA 1668A
3,3',5-Trichlorobiphenyl (36)	----------	EPA 1668A	EPA 1668A
3,4,4'-Trichlorobiphenyl (37)	----------	EPA 1668A	EPA 1668A
3,4,5-Trichlorobiphenyl (38)	----------	EPA 1668A	EPA 1668A
3,4',5-Trichlorobiphenyl (39)	----------	EPA 1668A	EPA 1668A
2,2',3,3'-Tetrachorobiphenyl (40)	----------	EPA 1668A	EPA 1668A
2,2',3,4-Tetrachlorobiphenyl (41)	----------	EPA 1668A	EPA 1668A
2,2',3,4'-Tetrachlorobiphenyl (42)	----------	EPA 1668A	EPA 1668A
2,2',3,5-Tetrachlorobiphenyl (43)	----------	EPA 1668A	EPA 1668A
2,2',3,5'-Tetrachlorobiphenyl (44)	----------	EPA 1668A	EPA 1668A
2,2',3,6-Tetrachlorobiphenyl (45)	----------	EPA 1668A	EPA 1668A
2,2',3,6'-Tetrachlorobiphenyl (46)	----------	EPA 1668A	EPA 1668A
2,2'4,4'-Tetrachlorobiphenyl (47)	----------	EPA 1668A	EPA 1668A

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',4,5-Tetrachlorobiphenyl (48)	----------	EPA 1668A	EPA 1668A
2,2',4,5'-Tetrachlorobiphenyl (49)	----------	EPA 1668A	EPA 1668A
2,2',4,6-Tetrachlorobiphenyl (50)	----------	EPA 1668A	EPA 1668A
2,2',4,6'-Tetrachlorobiphenyl (51)	----------	EPA 1668A	EPA 1668A
2,2',5,5'-Tetrachlorobiphenyl (52)	----------	EPA 1668A	EPA 1668A
2,2',5,6'-Tetrachlorobiphenyl (53)	----------	EPA 1668A	EPA 1668A
2,2',6,6'-Tetrachlorobiphenyl (54)	----------	EPA 1668A	EPA 1668A
2,3,3',4-Tetrachlorobiphenyl (55)	----------	EPA 1668A	EPA 1668A
2,3,3',4'-Tetrachlorobiphenyl (56)	----------	EPA 1668A	EPA 1668A
2,3,3',5-Tetrachlorobiphenyl (57)	----------	EPA 1668A	EPA 1668A
2,3,3',5'-Tetrachlorobiphenyl (58)	----------	EPA 1668A	EPA 1668A
2,3,3',6-Tetrachlorobiphenyl (59)	----------	EPA 1668A	EPA 1668A
2,3,4,4'-Tetrachlorobiphenyl (60)	----------	EPA 1668A	EPA 1668A
2,3,4,5-Tetrachlorobiphenyl (61)	----------	EPA 1668A	EPA 1668A
2,3,4,6-Tetrachlorobiphenyl (62)	----------	EPA 1668A	EPA 1668A
2,3,4',5-Tetrachlorobiphenyl (63)	----------	EPA 1668A	EPA 1668A
2,3,4',6-Tetrachlorobiphenyl (64)	----------	EPA 1668A	EPA 1668A
2,3,5,6-Tetrachlorobiphenyl (65)	----------	EPA 1668A	EPA 1668A
2,3',4,4'-Tetrachlorobiphenyl (66)	----------	EPA 1668A	EPA 1668A
2,3',4,5-Tetrachlorobiphenyl (67)	----------	EPA 1668A	EPA 1668A
2,3',4,5'-Tetrachlorobiphenyl (68)	----------	EPA 1668A	EPA 1668A
2,3',4,6-Tetrachlorobiphenyl (69)	----------	EPA 1668A	EPA 1668A
2,3',4',5-Tetrachlorobiphenyl (70)	----------	EPA 1668A	EPA 1668A
2,3',4',6-Tetrachlorobiphenyl (71)	----------	EPA 1668A	EPA 1668A
2,3',5,5'-Tetrachlorobiphenyl (72)	----------	EPA 1668A	EPA 1668A
2,3',5',6-Tetrachlorobiphenyl (73)	----------	EPA 1668A	EPA 1668A
2,4,4',5-Tetrachlorobiphenyl (74)	----------	EPA 1668A	EPA 1668A
2,4,4',6-Tetrachlorobiphenyl (75)	----------	EPA 1668A	EPA 1668A
2',3,4,5-Tetrachlorobiphenyl (76)	----------	EPA 1668A	EPA 1668A
3,3',4,4'-Tetrachlorobiphenyl (77)	----------	EPA 1668A	EPA 1668A
3,3',4,5-Tetrachlorobiphenyl (78)	----------	EPA 1668A	EPA 1668A
3,3',4,5'-Tetrachlorobiphenyl (79)	----------	EPA 1668A	EPA 1668A
3,3',5,5'-Tetrachlorobiphenyl (80)	----------	EPA 1668A	EPA 1668A
3,4,4',5-Tetrachlorobiphenyl (81)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4-Pentachlorobiphenyl (82)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5-Pentachlorobiphenyl (83)	----------	EPA 1668A	EPA 1668A
2,2',3,3',6-Pentachlorobiphenyl (84)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4'-Pentachlorobiphenyl (85)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5-Pentachlorobiphenyl (86)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5'-Pentachlorobiphenyl (87)	----------	EPA 1668A	EPA 1668A
2,2',3,4,6-Pentachlorobiphenyl (88)	----------	EPA 1668A	EPA 1668A
2,2',3,4,6'-Pentachlorobiphenyl (89)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5-Pentachlorobiphenyl (90)	----------	EPA 1668A	EPA 1668A
2,2',3,4',6-Pentachlorobiphenyl (91)	----------	EPA 1668A	EPA 1668A
2,2',3,5,5'-Pentachlorobiphenyl (92)	----------	EPA 1668A	EPA 1668A
2,2',3,5,6-Pentachlorobiphenyl (93)	----------	EPA 1668A	EPA 1668A
2,2',3,5,6'-Pentachlorobiphenyl (94)	----------	EPA 1668A	EPA 1668A
2,2',3,5',6-Pentachlorobiphenyl (95)	----------	EPA 1668A	EPA 1668A
2,2',3,6,6'-Pentachlorobiphenyl (96)	----------	EPA 1668A	EPA 1668A
2,2',3',4,5-Pentachlorobiphenyl (97)	----------	EPA 1668A	EPA 1668A
2,2',3',4,6-Pentachlorobiphenyl (98)	----------	EPA 1668A	EPA 1668A

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',4,4',5-Pentachlorobiphenyl (99)	----------	EPA 1668A	EPA 1668A
2,2',4,4',6-Pentachlorobiphenyl (100)	----------	EPA 1668A	EPA 1668A
2,2',4,5,5'-Pentachlorobiphenyl (101)	----------	EPA 1668A	EPA 1668A
2,2',4,5,6'-Pentachlorobiphenyl (102)	----------	EPA 1668A	EPA 1668A
2,2',4,5',6-Pentachlorobiphenyl (103)	----------	EPA 1668A	EPA 1668A
2,2',4,6,6'-Pentachlorobiphenyl (104)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4'-Pentachlorobiphenyl (105)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5-Pentachlorobiphenyl (106)	----------	EPA 1668A	EPA 1668A
2,3,3',4',5-Pentachlorobiphenyl (107)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5'-Pentachlorobiphenyl (108)	----------	EPA 1668A	EPA 1668A
2,3,3',4,6-Pentachlorobiphenyl (109)	----------	EPA 1668A	EPA 1668A
2,3,3',4',6-Pentachlorobiphenyl (110)	----------	EPA 1668A	EPA 1668A
2,3,3',5,5'-Pentachlorobiphenyl (111)	----------	EPA 1668A	EPA 1668A
2,3,3',5,6-Pentachlorobiphenyl (112)	----------	EPA 1668A	EPA 1668A
2,3,3',5',6-Pentachlorobiphenyl (113)	----------	EPA 1668A	EPA 1668A
2,3,4,4',5-Pentachlorobiphenyl (114)	----------	EPA 1668A	EPA 1668A
2,3,4,4',6-Pentachlorobiphenyl (115)	----------	EPA 1668A	EPA 1668A
2,3,4,5,6-Pentachlorobiphenyl (116)	----------	EPA 1668A	EPA 1668A
2,3,4',5,6-Pentachlorobiphenyl (117)	----------	EPA 1668A	EPA 1668A
2,3',4,4',5-Pentachlorobiphenyl (118)	----------	EPA 1668A	EPA 1668A
2,3',4,4',6-Pentachlorobiphenyl (119)	----------	EPA 1668A	EPA 1668A
2,3',4,5,5'-Pentachlorobiphenyl (120)	----------	EPA 1668A	EPA 1668A
2,3',4,5',6-Pentachlorobiphenyl (121)	----------	EPA 1668A	EPA 1668A
2',3,3',4,5-Pentachlorobiphenyl (122)	----------	EPA 1668A	EPA 1668A
2',3,4,4',5-Pentachlorobiphenyl (123)	----------	EPA 1668A	EPA 1668A
2',3,4,5,5'-Pentachlorobiphenyl (124)	----------	EPA 1668A	EPA 1668A
2',3,4,5,6'-Pentachlorobiphenyl (125)	----------	EPA 1668A	EPA 1668A
3,3',4,4',5-Pentachlorobiphenyl (126)	----------	EPA 1668A	EPA 1668A
3,3',4,5,5'-Pentachlorobiphenyl (127)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4'-Hexachlorobiphenyl (128)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5-Hexachlorobiphenyl (129)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5'-Hexachlorobiphenyl (130)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,6-Hexachlorobiphenyl (131)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,6'-Hexachlorobiphenyl (132)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,5'-Hexachlorobiphenyl (133)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,6-Hexachlorobiphenyl (134)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,6'-Hexachlorobiphenyl (135)	----------	EPA 1668A	EPA 1668A
2,2',3,3',6,6'-Hexachlorobiphenyl (136)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5-Hexachlorobiphenyl (137)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5'-Hexachlorobiphenyl (138)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',6-Hexachlorobiphenyl (139)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',6'-Hexachlorobiphenyl (140)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5,5'-Hexachlorobiphenyl (141)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5,6-Hexachlorobiphenyl (142)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5,6'-Hexachlorobiphenyl (143)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5',6-Hexachlorobiphenyl (144)	----------	EPA 1668A	EPA 1668A
2,2',3,4,6,6'-Hexachlorobiphenyl (145)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5,5'-Hexachlorobiphenyl (146)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5,6-Hexachlorobiphenyl (147)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5,6'-Hexachlorobiphenyl (148)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5',6-Hexachlorobiphenyl (149)	----------	EPA 1668A	EPA 1668A

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',3,4',6,6'-Hexachlorobiphenyl (150)	----------	EPA 1668A	EPA 1668A
2,2',3,5,5',6-Hexachlorobiphenyl (151)	----------	EPA 1668A	EPA 1668A
2,2',3,5,6,6'-Hexachlorobiphenyl (152)	----------	EPA 1668A	EPA 1668A
2,2',4,4',5,5'-Hexachlorobiphenyl (153)	----------	EPA 1668A	EPA 1668A
2,2',4,4',5',6-Hexachlorobiphenyl (154)	----------	EPA 1668A	EPA 1668A
2,2',4,4',6,6'-Hexachlorobiphenyl (155)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5-Hexachlorobiphenyl (156)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5'-Hexachlorobiphenyl (157)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',6-Hexachlorobiphenyl (158)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5,5'-Hexachlorobiphenyl (159)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5,6-Hexachlorobiphenyl (160)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5',6-Hexachlorobiphenyl (161)	----------	EPA 1668A	EPA 1668A
2,3,3',4',5,5'-Hexachlorobiphenyl (162)	----------	EPA 1668A	EPA 1668A
2,3,3',4',5,6-Hexachlorobiphenyl (163)	----------	EPA 1668A	EPA 1668A
2,3,3',4',5',6-Hexachlorobiphenyl (164)	----------	EPA 1668A	EPA 1668A
2,3,3',5,5',6-Hexachlorobiphenyl (165)	----------	EPA 1668A	EPA 1668A
2,3,4,4',5,6-Hexachlorobiphenyl (166)	----------	EPA 1668A	EPA 1668A
2,3',4,4',5,5'-Hexachlorobiphenyl (167)	----------	EPA 1668A	EPA 1668A
2,3',4,4',5',6-Hexachlorobiphenyl (168)	----------	EPA 1668A	EPA 1668A
3,3',4,4',5,5'-Hexachlorobiphenyl (169)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5-Heptachlorobiphenyl (170)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',6-Heptachlorobiphenyl (171)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5'-Heptachlorobiphenyl (172)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6-Heptachlorobiphenyl (173)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6'-Heptachlorobiphenyl (174)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5',6-Heptachlorobiphenyl (175)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,6,6'-Heptachlorobiphenyl (176)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4',5,6-Heptachlorobiphenyl (177)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6-Heptachlorobiphenyl (178)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,6,6'-Heptachlorobiphenyl (179)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5,5'-Heptachlorobiphenyl (180)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6-Heptachlorobiphenyl (181)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6'-Heptachlorobiphenyl (182)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5',6-Heptachlorobiphenyl (183)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',6,6'-Heptachlorobiphenyl (184)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5,5',6-Heptachlorobiphenyl (185)	----------	EPA 1668A	EPA 1668A
2,2',3,4,5,6,6'-Heptachlorobiphenyl (186)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5,5',6-Heptachlorobiphenyl (187)	----------	EPA 1668A	EPA 1668A
2,2',3,4',5,6,6'-Heptachlorobiphenyl (188)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5'-Heptachlorobiphenyl (189)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5,6-Heptachlorobiphenyl (190)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5',6-Heptachlorobiphenyl (191)	----------	EPA 1668A	EPA 1668A
2,3,3',4,5,5',6-Heptachlorobiphenyl (192)	----------	EPA 1668A	EPA 1668A
2,3,3',4',5,5',6-Heptachlorobiphenyl (193)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5'-Octachlorobiphenyl (194)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6-Octachlorobiphenyl (195)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6'-Octachlorobiphenyl (196)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',6,6'-Octachlorobiphenyl (197)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6-Octachlorobiphenyl (198)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6'-Octachlorobiphenyl (199)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,6,6'-Octachlorobiphenyl (200)	----------	EPA 1668A	EPA 1668A

Parameter/Analyte	Potable Water	Nonpotable Water	Solid Hazardous Waste
2,2',3,3',4,5',6,6'-Octachlorobiphenyl (201)	----------	EPA 1668A	EPA 1668A
2,2',3,3',5,5',6,6'-Octachlorobiphenyl (202)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5,5',6-Octachlorobiphenyl (203)	----------	EPA 1668A	EPA 1668A
2,2',3,4,4',5,6,6'-Octachlorobiphenyl (204)	----------	EPA 1668A	EPA 1668A
2,3,3',4,4',5,5',6-Octachlorobiphenyl (205)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (206)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl (207)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl (208)	----------	EPA 1668A	EPA 1668A
2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl (209)	----------	EPA 1668A	EPA 1668A
Soxhlet/Dean-Stark Extraction	----------	----------	EPA 3540C
Continuous Liquid-Liquid Extraction	EPA 3520C	----------	----------

The American Association for Laboratory Accreditation

World Class Accreditation

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A2LA has accredited

CAPE FEAR ANALYTICAL, LLC

Wilmington, NC

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 DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); 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*).

Presented this 5th day of April 2010.

President & CEO
For the Accreditation Council
Certificate Number 3014.01
Valid to May 31, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

DIOXIN/FURAN/PCB CONGENER SAMPLE PROCESSING

(CF-OA-E-001 REVISION 2)

APPLICABLE TO METHODS:
EPA SW-846 Method 8290A, EPA Method 1613B and EPA Method 1668B

PROPRIETARY INFORMATION

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CFA

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W. M. Larkins

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1.0 STANDARD OPERATING PROCEDURE FOR DIOXIN/FURAN/PCB CONGENER SAMPLE PROCESSING

2.0 METHOD OBJECTIVE, PURPOSE, CODES, AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions for the preparation, extraction and cleanup of environmental samples for analysis by high-resolution GC/MS methods listed below, for the determination of polychlorinated dibenzo-dioxins/furans(PCDDs/PCDFs) and polychlorinated biphenyls (PCBs).

2.2 Solid and tissue samples are homogenized as needed and extracted by soxhlet/Dean-Stark (SDS). Aqueous samples are extracted by continuous liquid-liquid extraction (CLLE). Sample extracts are fractionated and prepared for analysis using a variety of cleanup procedures, including silica gel and florisil column chromatography.

2.3 Analytical methods

2.3.1 EPA SW-846 Method 8290A

2.3.2 EPA Method 1613B

2.3.3 EPA Method 1668B

2.4 Extraction techniques

2.4.1 Method 3520C (CLLE)

2.4.2 Method 3540C (SDS)

2.5 Cleanup techniques

2.5.1 Method 3620C (Florisil)

2.5.2 Method 3630C (Silica)

3.0 APPLICABLE MATRICES

This SOP is applicable to solid (soil, sediment, sludge), aqueous (groundwater, surface water, leachate, drinking water) and tissue matrices.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

4.1 Calibration ranges and PQLs may be found in the appropriate analytical SOPs.

4.2 Analysts must demonstrate proficiency prior to work under this SOP. Demonstrated proficiency may be in the form of an IDOC or PT study. Records are maintained in the Quality Department.

5.0 METHOD VARIATIONS

5.1 Aqueous samples containing >1% solids are filtered prior to extraction. The aqueous portion is extracted by CLLE, and the filter and solids are extracted by SDS. The fractions are re-combined prior to cleanup.

5.2 Aqueous samples are adjusted to a pH of </= 7 using 50:50 sulfuric acid. Samples with an initial pH >10 are re-checked after acidifying to ensure a pH of </= 7.

6.0 DEFINITIONS

6.1 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.

6.2 Blank: An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the

laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.

6.3 Cleanup Standards: Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1613B uses one compound (37Cl4-2378-TCDD) as the Cleanup Standard. Method 1668B uses three compounds as cleanup standards. Method 8290A does not address the use of cleanup standards; CFA chooses to use five labeled compounds for in-house monitoring of fractionation efficiency when performing this method.

6.4 Extraction Standards: Isotopes added prior to extraction that serve as internal standards. In addition, to measure the overall extraction and fractionation efficiencies.

6.5 Laboratory Control Standard/Duplicate (LCS/LCSD): Aliquots of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS/LCSD are analyzed exactly like a sample, and their purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.6 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The duplicate aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.7 Matrix Spike and Matrix Spike Duplicate (MS and MSD): Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.

7.0 INTERFERENCES/LIMITATIONS

7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.

7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.

7.3 Reagents and solvents should be purified and tested before use, or alternatively, they should be purchased pre-cleaned by the manufacturer.

7.4 PCB extracts must not be allowed to concentrate to dryness due to the potential for loss of low molecular weight chlorinated biphenyls.

8.0 SAFETY PRECAUTIONS AND WARNINGS

WARNING

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

NO OCCUPATIONAL EXPOSURE LIMIT FOR DIOXIN HAS BEEN ESTABLISHED. IT IS A KNOWN AND PROBABLE HUMAN CARCINOGEN.

PCBs HAVE BEEN TENTATIVELY CLASSIFIED AS KNOWN OR SUSPECTED HUMAN OR MAMMALIAN CARCINOGENS.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS FROM SOLIDS.

8.1 Eye protection should be worn when handling samples, reagents, or standards. NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.

8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.

8.3 Personal Protective Equipment (PPE)

8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied.

8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.

8.8 For further safety instructions, consult the Safety, Health and Chemical Hygiene Plan, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

9.1 Equipment associated with this SOP includes:

- Soxhlet/Continuous Liquid-Liquid Extraction Rack
- Soxhlet/Dean-Stark (SDS) glassware apparatus
- Continuous Liquid-Liquid (CLLE) glassware apparatus
- Balances
- Fume hoods
- Turbovap
- Dry weight oven
- Buchner funnels
- Vacuum pump

- 2 L vacuum flask

9.2 Materials and supplies:

- Boiling chips
- Cellulose thimbles
- Spatulas
- Aluminum dry weight pans
- pH strips
- 5 ¾" disposable pipets and rubber pipet bulps
- Filter paper
- Nitrogen
- 60 mL vials with PTFE caps
- 60 mL vial racks
- 25 mL drying columns
- 10 mL drying columns
- Glass wool
- 10-100 µL air displacement pipet and disposable tips
- 100-1000 µL air displacement pipet and disposable tips
- 1000 mL graduated cylinder

10.0 REAGENTS AND STANDARDS

10.1 Reagents, chemicals, and standards:

- Methylene chloride
- Toluene
- Hexane
- Carbon—Carbopack C or equivalent
- Celite
- Tridecane: Plug a 25 mL drying column with glass wool; add 1 g sodium sulfate; add 2 g carbon-coated celite. Percolate the tridecane from the supplier's bottle through the carbon column. Collect into a clean and properly labeled container; document preparation in the Supply Prep logbook.
- Nonane
- Deionized water
- Sodium sulfate
- Concentrated sulfuric acid
- 1 N Sodium hydroxide
- High purity potassium hydroxide
- Neutral silica

- Acid-coated silica: 44.0 g concentrated sulfuric acid to 100 g neutral silica. Add 1 to 2 mL acid to the silica and shake vigorously; continue until all acid has been added. Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- NaOH-coated silica: 30.0 g 1 N NaOH to 100 g neutral silica. Add 1 to 2 mL base to the silica and shake vigorously; continue until all base has been added. Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- KOH-coated silica: Dissolve 56 g high purity KOH in 300 mL methanol. Add 100 grams neutral silica; stir on hot plate at 60° to 70°C for 1 to 2 hours. Decant liquid and rinse with two 100 mL aliquots methanol. Rinse once with 100 mL methylene chloride. Spread potassium silicate on foil and dry for 1 to 2 hours in a fume hood. Activate at 200 °C for 18 hours. Stir in 37 g neutral silica (This is a deviation from the methods, intended to reduce the strength of the KOH-coated silica to prevent recovery losses in the samples). Document preparation in the Supply Prep logbook; affix appropriate label to jar.
- Florisil
- Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These include extraction standard, matrix spiking standard, cleanup standard, injection standard. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference CF-LB-E-007 for further information regarding standards and their preparation.

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 For method 8290A, samples must be extracted within 30 days of collection. For methods 1613B and 1668B, samples must be extracted within 1 year of collection. Sample extracts have a 45-day holding time from the date of extraction by method 8290A and a 365 day holding time from the date of extraction by 1613B and 1668B. Note that per method 8290A, tissue extracts must be completely analyzed by 45 days from collection.

11.2 Samples should be collected in amber glass containers with PTFE-lined caps.

11.3 Aqueous samples should be checked in the field for the presence of residual chlorine, and if present, preserved with 80 mg sodium thiosulfate. If sample labels do not indicate whether they have been checked/preserved, the samples should be checked at the time of receipt, and preserved if necessary.

11.4 Samples should be maintained at $0 \leq 6$ °C after collection until such time as they may be disposed by the laboratory.

11.5 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to extract into their custody.

11.6 All samples and sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

12.1 Sample preparation and conditioning

12.1.1 Homogenization of tissue

- Vegetation samples can require special procedures to prevent cross contamination from other matrices. Some vegetation can be difficult to reduce in particle size and homogenize. Vegetation sample processing is handled on a case-by-case basis with project management to client communication.

12.1.2 Homogenization of tissue

- Tissue samples should be received and maintained at -10 to -20°C. If a sample is received as a whole specimen (fish, rodent, etc.), a project manager should verify with the client which portion of the specimen is to be analyzed.
- If specific portions must be dissected from the whole specimen, the remaining tissue can be refrozen. When the entire sample, usually fish, requires analysis, reduce the sample down to manageable means using a butcher knife.

NOTE: Great care must be exercised when reducing a frozen fish with a knife. Cut-resistant gloves must be worn to protect the hands as well as to hold the fish in place.

- When analyzing specific portions, such as a fillet, dissect the required portion under stringent safety guidelines to prevent injury as well as to ensure the integrity of the sample.
- Once reduced in size or dissected to the appropriate portion, process the tissue through a meat grinder, if required, and collect into an amber glass jar.
- To ensure a homogeneous mixture, grind the sample two more times.
- Record any observations on the bench sheet.

12.1.3 Compositing and aliquotting

Specific procedures for compositing and aliquotting may be found in the following SOPs: CF-LB-E-031 and CF-LB-E-032.

12.1.4 Percent solids determination

Specific procedures for determination of percent solids (moisture content) may be found in SOP CF-OA-E-020.

12.1.5 Percent lipids determination

- Extract the sample by soxhlet using methylene chloride. Do not add tridecane.
- Concentrate the extract to approximately 20 mL and transfer to a tared 60 mL vial.
- Concentrate the extract to dryness in a Turbovap.
- Allow to cool; take an initial weight using an analytical balance.
- Store the extract vial in a dessicator for 12 hours and re-weigh. Continue to perform re-weighs every hour until constant weight is achieved (<1% change).
- Calculate percent lipids as follows:

$$\%L = \frac{R}{W} \times 100$$

Where:
R = weight of dry residue in grams
W = initial weight of sample in grams

12.1.6 Sub-Sampling

Sample matrices and mechanisms of contamination are infinitely variable and require judgments to be made. If the client provides no instructions, it is advisable that experienced analysts decide which sub-sampling techniques are employed. For solid samples, mixing is the preferred process. For liquid samples without obvious layers, the entire container is used and rinsed. Documentation of these two forms of sub-sampling is not required due to the frequency of their use.

All other selected sub-sampling techniques should be documented. If the sample integrity or composition does not match that anticipated by the laboratory, the client must be contacted to confirm or clarify any sub-sampling instructions. An example of a time when client direction is required can be demonstrated with the receipt of a coring sleeve. If upon opening the container, the analyst notices obvious heterogeneous composition of the sample, (clay in one end of the sleeve, and sand at the other end), appropriate instructions from the client must be obtained prior to beginning analysis.

Anytime the analytical result will obviously be biased, it must be documented, and the client should be notified prior to beginning any sub-sampling technique. An example of a time when this occurs is when interferences or target contamination require the use of a reduced extraction volume for a liquid sample preventing a container rinse.

12.2 Sample Extraction

12.2.1 Soxhlet/Dean-Stark (SDS) Extraction

12.2.1.1 Pre-extraction

- Fill 500 mL round-bottom flask with 350 mL extraction solvent (methylene chloride for all tissues and PCB solids; toluene for dioxin/furan solids).
- Add one scoop of boiling chips to the round-bottom and attach soxhlet and Dean-Stark glassware. If pre-extracting for PCB samples, also include the thimble. If samples to be extracted are not extremely wet (i.e. >30% moisture), the Dean-Stark may not be needed.
- Clamp the apparatus securely to the extraction rack and attach the condenser.
- Pull out emergency stops to energize heater controllers.
- Set the heating mantle controller to ‘65’ for toluene or ‘50’ for methylene chloride, turn power switch to ‘on’, and allow to reflux for 3 to 4 hours.
- Turn power switch to ‘off’ and allow apparatus to cool.
- Hit emergency stops to turn off controllers.

- Disassemble condenser and SDS and dump solvent into appropriate waste container.
- Glassware is now prepared for extraction.

12.2.1.2 Extraction of Solid Samples

- Refill the round-bottom flask with 350 mL extraction solvent.
- Add one scoop fresh boiling chips.
- For dioxin/furan extractions, add 500 uL tridecane as a keeper solvent. Do NOT add tridecane for PCB extractions.
- Reassemble the glassware apparatus, clamp securely to the extraction rack.
- Weigh a 10 g dry weight equivalent soil aliquot into a cellulose thimble.
- Add 10 to 15 g sodium sulfate to the thimble and mix thoroughly.
- Place the thimble into the appropriately labeled soxhlet.
- Add the appropriate type and amount of extraction standard (spike) to the thimble (see Appendix 1).
- Attach a Dean-Stark adaptor (if needed) and attach the condenser.
- Allow the sample 1 – 2 hours for equilibration
- Pull out emergency stops.
- Set the heating mantle controller to '65' for toluene or '50' for methylene chloride, turn power switch to 'on', wrap the round-bottom flask with aluminum foil and allow to reflux for 18 to 24 hours.
- At the end of the extraction period, open the stopcock on the soxhlet and allow the solvent to drain to waste.
- When the solvent level reaches 30 to 40 mL, turn off the heating mantle controller and allow the apparatus to cool.
- Hit emergency stops.
- Remove the condenser, Dean-Stark and soxhlet from the round-bottom flask, and place the cellulose thimble into the appropriate waste can.
- Quantitatively transfer the extract from the round-bottom flask to an appropriately labeled 60 mL vial.
- Concentrate the extract under nitrogen in a Turbovap. Dioxin/furan extracts should be concentrated to 500 µL (tridecane) and exchanged with hexane. PCB extracts should be concentrated to approximately 2 to 3 mL to avoid analyte loss. DO NOT TAKE PCB EXTRACTS TO DRYNESS.
- Add the appropriate type and amount of cleanup standard to the extract (see Appendix 1).
- The extract is now ready for cleanup.

12.2.2 Aqueous Extraction

12.2.2.1 Continuous Liquid-Liquid Pre-extraction

- Verify water level in circulation tank.

- Turn the two-way valves to 'extract' and turn on the pump switch to 'extract'. Verify water is flowing.
- Turn on the water heater to 160 °F.
- Add 1 to 2 boiling chips to the concentrator, fill the drying adapter halfway with sodium sulfate, and assemble the glassware apparatus.
- Securely attach the apparatus to the CLLE extraction rack. Attach the water hoses to the concentrator.
- Add 200 mL methylene chloride to the extractor body. Then add 1 L deionized water, being careful not to allow any water into the sidearm.
- Attach the condenser to the apparatus.
- Verify chiller temperature and flow.
- Open the water valves to allow hot water to flow through the concentrator.
- Open the sidearm stopcock to allow methylene chloride to flow over to the drying adapter.
- Allow pre-extraction to run for 3 to 4 hours.
- Close stopcock and allow methylene chloride to concentrate to 15-20 mL.
- Turn off heater, close water valves, turn two-way valves to 'drain', and turn pump switch to 'drain'.
- Once the water has drained, turn the pump switch to 'off".
- Remove the concentrator and drying adapter and pour remaining methylene chloride into the appropriate waste container, leaving the boiling chips inside.
- Rinse twice more with methylene chloride and dump to waste.
- Re-attach the drying adapter.
- Remove the extractor body and dump the deionized water and methylene chloride in the appropriate waste container.
- Re-attach the entire apparatus to the extraction rack.
- Glassware is now ready for sample extraction.

12.2.2.2 High- solids (>1%) Procedure

- Remove water sample from the cooler and allow to come to room temperature.
- Pipet 1-2 mL of sample onto a pH strip and record pH on bench sheet.
- Spike the sample with the appropriate type and amount of extraction standard (see Appendix 1)
- Allow sample to equilibrate for 1 – 2 hours
- Place sample on balance and tare.
- Attach large Buchner funnel to 2L vacuum flask.
- Attach vacuum pump hose to vacuum flask.

- Place sheet of glass fiber filter paper inside funnel and rinse with toluene.
- Pull off toluene with vacuum and discard.
- Pour sample into funnel and apply vacuum.
- Reweigh the sample bottle on the tared scale and record the resulting sample volume on the bench sheet.
- Rinse the sample bottle with 50 mL deionized water and add to funnel.
- Continue to pull vacuum until all water has been pulled through the filter.
- Turn off vacuum pump.
- Remove filter paper and place in soxhlet extractor. Extract using procedure in Section 12.2.1.
- Pour water into extractor body and extract using procedure in Section 12.2.2.3. NOTE: SAMPLE HAS ALREADY BEEN SPIKED. DO NOT RESPIKE.

12.2.2.3 Low-solids (<1%) Extraction by Continuous Liquid-Liquid (CLLE)

- Fill extractor body with 250 mL methylene chloride.
- Remove water sample from the cooler and allow to come to room temperature.
- Pipet 1 to 2 mL of sample onto a pH strip and record pH on bench sheet.
- Place sample on balance and tare.
- Shake to thoroughly homogenize the sample, and pour into the appropriately labeled extractor body, being sure the sample does not get into the sidearm.
- Reweigh the sample bottle on the tared scale and record the resulting sample volume on the bench sheet.
- Rinse the sample bottle with 50 mL methylene chloride and pour into the extractor body.
- Spike the sample with the appropriate type and amount of extraction standard (see Appendix 1).
- Allow the sample to equilibrate for 1 – 2 hours.
- Add 2 mL 50:50 sulfuric acid to the extractor body. If initial pH was >10, re-check pH; if still > 7, continue adding acid to achieve a pH </= 7.
- Attach the condenser.
- Turn the two-way valves to ‘extract’ and turn on the pump switch to ‘extract’.
- Turn on the water heater.
- Open the water valves.
- Open the stopcock.
- Allow the sample to extract for 18 to 24 hours.

- Close the stopcock and allow the extract to concentrate down to the nipple of the concentrator. If samples are for PCB analysis, only concentrate to 5 to 10 mL to avoid analyte loss.
- Turn heater off, close water valves, switch two-way valves to 'drain' and turn pump to 'drain'.
- When water has drained, turn pump to 'off'.
- Remove concentrator and drying adapter.
- Pour contents of extractor body into the appropriate waste container.
- Remove drying adapter from concentrator and dump sodium sulfate into appropriate waste container.
- Quantitatively transfer the extract to an appropriately labeled 60 mL vial.
- Add the appropriate type and amount of cleanup standard to the vial. For dioxin/furan extracts, add 100 µL tridecane to the vial.
- Concentrate the extracts down to the 100 µL tridecane in the Turbovap at 45 °C. For PCB extracts, only concentrate down to 2 to 3 mL.
- Remove the vial and cap.
- Extract is now ready for cleanup.

12.2.3 Cleanup Procedures

12.2.3.1 Acid/Base Silica Column (for dioxin/furan samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column from bottom to top as follows: 1 g neutral silica gel, 4 g basic silica gel, 1 g neutral silica gel, 8 g acidic silica gel, 2 g neutral silica gel, 4 g sodium sulfate.
- Rinse the column with 50 mL hexane and catch in a waste container.
- Position a 60 mL vial under the column.
- Quantitatively transfer the extract onto the column.
- Elute the sample with 50 mL hexane.
- Remove the 60 mL vial and cap; position a new 60 mL vial under the column.
- Elute with an additional 50 mL hexane.
- Concentrate the two vials to approximately 5 ml in the Turbovap at 45 °C and combine into one vial.
- If the extract requires additional cleanup, concentrate to approximately 1 to 2 mL and proceed to another cleanup step.
- If the extract is ready for analysis, concentrate to dryness and add 0.5 mL methylene chloride.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.2 Acid/Base Silica Column (for PCB samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column from bottom to top as follows: 1 g neutral silica gel, 4 g basic silica gel, 1 g neutral silica gel, 8 g acidic silica gel, 2 g neutral silica gel, 4 g sodium sulfate.
- Rinse the column with 50 mL hexane and catch in a waste container.
- Position a 60 mL vial under the column.
- Quantitatively transfer the extract onto the column.
- Elute the sample with 25 mL hexane.
- If the extract requires additional cleanup, concentrate to approximately 1 to 2 mL in the Turbovap at 45 °C and proceed to another cleanup step.
- If the extract is ready for analysis, concentrate to 0.5 mL. DO NOT let PCB extracts go to dryness.
- Quantitatively transfer the extract to a labeled GC vial containing 18 µL nonane and deliver to the GC/MS analyst.

12.2.3.3 Florisil Column (for dioxin/furan samples)

- Insert a glass wool plug into a 10 mL disposable pipet, pack with 1.5 g florisil, and top with 1 g sodium sulfate.
- Rinse the column with 10 mL methylene chloride, 10 mL 2% methylene chloride/hexane, and 10 mL hexane, and discard to waste.
- Quantitatively transfer the extract to the column with hexane.
- Rinse the column with 10 mL hexane.
- Elute with 20 mL 2% methylene chloride/hexane and discard to waste.
- Position a 60 mL vial under the column and elute the sample with 35 mL methylene chloride.
- Concentrate the extract in the Turbovap at 45 °C to approximately 0.5 mL.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.4 Anthropogenic Isolation Column (for all tissue samples)

- Insert a glass wool plug into a 25 mL drying column and pack the column as follows: 2 g neutral silica gel, 2 g potassium silicate, 2 g sodium sulfate, 10 g acid silica gel, 2 g sodium sulfate.
- Elute the column with 100 mL hexane and collect to a waste container.
- Quantitatively transfer the extract to the column.
- Elute with 200 mL hexane and collect into an appropriate sample container.

- If the sample requires further cleanup, concentrate the extract to approximately 1 to 2 mL hexane in the Turbovap at 45 °C and proceed with another cleanup.
- If no further cleanup is required, concentrate the extract to dryness (for dioxin/furan samples) and add 0.5 mL methylene chloride. For PCB samples, concentrate only to approximately 0.5 mL. DO NOT take PCB samples to dryness.
- Quantitatively transfer the extract to a labeled GC vial and deliver to the GC/MS analyst.

12.2.3.5 Acid/Base Back-Extraction (for PCB samples)

- Partition the extract against 50 mL potassium hydroxide solution.
- Shake for 2 minutes, venting periodically.
- Remove and discard the aqueous layer.
- Repeat until no color is visible in the aqueous layer, to a maximum of four washings.
- Partition the extract against 50 mL sodium chloride solution.
- Discard the aqueous layer.
- Partition the extract against 50 mL sulfuric acid.
- Discard the aqueous layer.
- Repeat until no color is visible in the aqueous layer, to a maximum of four washings.
- Partition the extract against 50 mL sodium chloride solution.
- Discard the aqueous layer.
- Transfer the extract to a drying column containing 7-10 grams sodium sulfate.
- Concentrate the extract to 1 to 2 mL hexane in the Turbovap at 45°C and proceed with additional cleanup steps.

13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

13.1 Method Blank (MB)

A method blank is extracted with each extraction batch of 20 samples or less. The method blank should be a reagent-free matrix similar to that of the batch, such as deionized water, glass beads or purified corn oil. The method blank is spiked in the same manner as the samples. The method blank is subjected to the same extraction and cleanup procedures as the samples, and is used as a measure of laboratory contamination. Method blank acceptance criteria may be found in the appropriate analytical SOP.

13.2 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

A laboratory control sample is extracted with each extraction batch of 20 samples or less. The LCS should be a reagent-free matrix (as listed above), and is subjected to the same extraction and cleanup procedures. The LCS is spiked with native analytes in addition to the extraction standards added to all samples. The LCS is subjected to the same extraction and cleanup procedures as the samples, and is used as a measure of system performance. A LCSD may also be performed as required by the

analytical method. LCS/LCSD acceptance criteria may be found in the appropriate analytical SOP.

13.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A MS/MSD pair may be performed as required by the analytical method, or as requested by a specific client. A matrix spike is a second aliquot of sample which is spiked with native analytes prior to extraction, and is used as another measure of system performance. MS/MSD acceptance criteria (if available) may be found in the appropriate analytical SOP.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

Samples are analyzed using a Waters Autospec Premier high-resolution GC/MS system. Instrument calibration, standardization and performance information may be found in the appropriate analytical SOP.

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

Procedures for analysis and instrument operation may be found in the appropriate analytical SOP.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Chiller water levels should be checked monthly.

16.2 Chromalox heater water level should be checked each day of use.

16.3 Balance

Procedures for balance calibration and maintenance may be found in SOP CF-LB-E-002 for Balances.

16.4 Fume Hood

Fume hood monitoring and maintenance may be found in SOP CF-FC-E-003.

16.5 Dry weight oven temperature should be verified each day of use.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data such as sample weights/volumes, dry weights, pH, spike amounts and lot numbers are input to the LIMS.

17.2 Calculations such as percent moisture and percent lipids are calculated in the LIMS.

18.0 POLLUTION/CONTAMINATION

18.1 Work areas should be maintained free of dust and dirt accumulations.

18.2 Whenever possible, work should be performed in a fume hood to reduce the spread of solvent fumes and airborne contaminants.

18.3 Glassware should be washed and prepared in a designated area.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Bench data are input manually to AlphaLIMS. The data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-extraction, the use of a new spike mix, or a more complex set of actions. When troubleshooting measures (Section 21) fail to bring an analytical process or data into control, a nonconformance report (NCR) and/or corrective action should be initiated in accordance with CF-QS-E-004 for the

Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when a sample or QC fails to meet defined acceptance criteria. Troubleshooting may involve one or more of the following actions:

21.1 When a method blank fails the defined criteria, the analyst must find and eliminate the source of contamination before proceeding with the analysis. This may involve the further testing of reagents, solvents, equipment or glassware. If the contamination is believed to be from a highly contaminated sample in the batch, the sample may be removed from the batch and the remaining batch re-processed.

21.2 If an extraction standard recovery fails the specified criteria, the sample must be evaluated as to why the failure may have occurred. If matrix interferences are apparent, the extract may need further cleanup steps, or the sample may need to be re-extracted at a reduced weight/volume.

21.3 If any native analyte recoveries in the LCS are outside specified limits, the process should be examined. Corrective action may include repeating the extraction or analysis, utilizing a new spike mix, or more complex actions.

21.4 If normal equipment and software operating procedures do not resolve trouble-shooting efforts, the manuals for software, hardware and other equipment discussed in this SOP are available for consultation and resolution. On-line support may be available from software and instrument manufacturers, as well. Any revisions, repairs or corrective actions required must be documented in accordance with the laboratory's Quality System as described in CF-QS-B-001.

22.0 RECORDS MANAGEMENT

22.1 Bench data logbooks are stored in the lab in storage boxes as long as there is space available. When space runs out, the boxes are sent to off-site storage.

22.2 Records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 METHOD VARIATIONS

EPA Method 3520C, Continuous Liquid-Liquid Extraction and EPA Method 3540C, Soxhlet Extraction include the use and control of laboratory equipment not in use at Cape Fear Analytical, LLC. The laboratory SOP correctly reflects the equipment used to perform the preparatory methods of reference.

24.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

25.0 REFERENCES

25.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 8290A, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/ High Resolution Mass

Spectrometry (HRGC/HRMS)," Rev. 1, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

25.2 Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Rev. B, Oct. 1994. USEPA, Office of Water, Engineering and Analysis Division, 401 M Street SW, Washington, DC 20460.

25.3 Method 1668B, "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS", November 2008. USEPA, Office of Water, Office of Science and Technology, Engineering and Analysis Division, 1200 Pennsylvania Avenue NW, Washington, DC 20460.

26.0 HISTORY

Revision 0: New document.
Revision 1: Updated solid reference matrix in Section 13. Added Appendix 1, Spike Profiles. Added vegetation sample processing comments in section 12.1.1.
Revision 2: Clarified pH requirements, revised extraction time to match reference method, re-ordered some bulleted steps to match actual practices, changed to nonane as PCB solvent, added method variation section, added discussion of software and equipment troubleshooting support.

Appendix 1: Spike Profiles

1613	Volume (µL)	[Conc] (ng/µL)	Amt. (ng)	Final Vol. (µL)	[Inst] (pg/µL)	[Sx] (pg/g)
1613ES	40	0.05	2	20	100	200
1613MX (LCS)	40	0.005	0.2		10	20
1613CS	20	0.01	0.2		10	20
8290JS*	20	0.1	2		100	200

8290	Volume (µL)	[Conc] (ng/µL)	Amt. (ng)	Final Vol. (µL)	[Inst] (pg/µL)	[Sx] (pg/g)
8290ES	40	0.05	2	20	100	200
1613MX* (LCS)	40	0.005	0.2		10	20
8290CS	20	0.01	0.2		10	20
8290JS	20	0.1	2		100	200

1668	Volume (µL)	[Conc] (ng/µL)	Amt. (ng)	Final Vol. (µL)	[Inst] (pg/µL)	[Sx] (pg/g)
1668ES	40	0.05	2	20	100	200
1668MX (LCS)	40	0.0125	0.5		25	50
1668CS	40	0.05	2		100	200
1668JS	2	1	2		100	200

* Standard is interchangeable between methods 8290 and 1613

Complete compound lists for each standard may be found in the standards logbook.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE ANALYSIS OF POLYCHLORINATED DIBENZO-p-DIOXINS AND POLYCHLORINATED DIBENZOFURANS (PCDDs/PCDFs)

BY

HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

(CF-OA-E-002 REVISION 4)

APPLICABLE TO METHODS:
EPA SW-846 Method 8290A, EPA Method 1613B, EPA SW-846 Method 0023A

PROPRIETARY INFORMATION

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CFA's Document Control Officer certifies this document to be a true copy of the fully executed original.

W. M. Larkins

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1.0 STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXINS AND POLYCHLORINATED DIBENZOFURANS (PCDD/PCDF) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

This standard operating procedure (SOP) covers the analytical determination of PCDD/PCDFs according to the following methods:

2.1 SW-846 Method 8290A

2.2 EPA Method 1613B

2.3 SW-846 Method 0023A

3.0 APPLICABLE MATRICES

Applicable matrices for methods 8290A and 1613B include groundwater, wastewater, surface water, leachate, soil, sediment, sludge, oil, and tissue. The applicable matrix for method 0023A is an air sampling train, which may contain XAD resin, impinger water and solvent rinses.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

4.1 Methods 8290A, 1613B and 0023A may be used to quantify PCDD/PCDFs that are soluble in methylene chloride and/or toluene. The compounds are separated using a gas chromatograph (GC) and detected using a high-resolution double focusing mass spectrometer (HRMS). Appendix 1 lists the analytes currently analyzed using these methods and their practical quantitation limits.

4.2 The practical quantitation limit (PQL) is the lowest level in the calibration curve. The PQL is the lowest level at which compounds may be accurately quantitated and is compound dependent. The calibration curve typically ranges from 1.0 ng/mL to 1000 ng/mL for methods 8290A and 0023A, and from 0.5 ng/mL to 2000 ng/mL for method 1613B. These ranges reflect instrument readings, which are in ng/mL (ppb). It should be noted that the calibration range may vary between calibrations and instruments.

4.3 Method detection limit studies (MDLs) are performed and/or verified on an annual basis. MDLs are done for aqueous, solid, tissue and XAD matrices. For more information regarding MDLs, refer to The Determination of Method Detection Limits, CF-LB-E-001.

4.4 Qualified analysts must demonstrate proficiency initially and annually thereafter with an IDOC, CDOC, or PT study. Acceptability criteria may be found in the applicable analytical method.

4.4.1 To establish the ability to generate acceptable accuracy and precision, the analyst should perform an "analyst validation study" or Initial Demonstration of Capability. Four LCS standards are extracted and analyzed. Calculate the average recovery and the standard deviation of the recovery for each analyte of interest using the four results. Then compare the average and the standard deviation with the corresponding criteria found in Table 6 of method 1613B, or with the determined limits for methods 8290A and 0023A. If the average and the standard deviation for all analytes of interest meet the acceptance criteria, then the analyst may begin work on actual samples. If the validation

study fails for one or more of the compounds, then the study must be repeated for those compounds which failed.

5.0 METHOD VARIATIONS

5.1 Cape Fear Analytical analyzes a calibration point at 0.25 ng/mL, which is below the method required low point.

5.2 Standards and sample extracts are stored at room temperature to avoid analyte loss. Many of the target analytes in these methods form a strong cohesive bond with solids such as glass in cold temperatures; this type of analyte loss is not addressed in the method. (This is a variance from the following method recommendations: ≤6° per method 8290A; < -10°C per 1613B; -10° to -20° C per DoD QSM.)

5.3 Cape Fear Analytical utilizes the DB-5MS GC column, which is capable of better resolution of the TCDF isomers. This column exhibits a different elution pattern than the DB-5 column referenced in the analytical methods. Relative retention time limits have been determined for this column for use with method 1613B, and are listed in Table 8.

5.4 Method 1613B does not address the reporting of EDL and EMPC. These values are reported for this method only when requested by the client.

6.0 DEFINITIONS

6.1 Accuracy: The degree of agreement between an observed value and an accepted reference value.

6.2 AlphaLIMS: The Laboratory Information Management System used at CFA, LLC.

6.3 Blank: An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and standard additions that are used with other samples. The LMB (Lab Method Blank) is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Contamination may be derived during sampling, transportation, storage or analysis. The blank may be used to establish a background value.

6.4 Calibration Standard (CAL): An aliquot of a primary standard solution or stock standard solution. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.5 Calibration Verification Standard (CVS, CCAL, CS3WT): A solution of target analytes with a concentration near the mid-point of the calibration range. It should be obtained from a second source vendor and is used to verify the initial calibration on a basis described in the determinative method. This solution may also contain the window defining analytes and the column performance mix.

6.6 Cleanup Standards: Isotopes added prior to cleanup that are used to measure the efficiency of the fractionation step alone. Method 1613B uses one compound (37Cl4-2378-TCDD) as the Cleanup Standard. Method 8290A does not address the use of cleanup standards; CFA chooses to use five labeled compounds for in-house monitoring of fractionation efficiency in Method 8290.

6.7 Duplicate Analysis: The analysis or measurement of the variable of interest performed identically on two field subsamples of the same sample. The results from

duplicate analyses are used to evaluate analytical or measurement precision of sample, preservation, or storage internal to the laboratory.

6.8 Estimated Detection Limit (EDL): A calculation of 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. The EDL is calculated for each 2378-substituted congener that is not identified.

6.9 Estimated Maximum Possible Concentration (EMPC): A calculation for a peak characterized by a response with a signal-to-noise ratio of at least 2.5 for both the quantitation ions, and meeting all identification criteria except ion ratio. EMPC is a worst-case estimate of the concentration.

6.10 Extraction Standards: Isotopes added prior to extraction that serve as internal standards for many 2,3,7,8 substituted congeners. In addition, to measure the overall extraction and fractionation efficiencies. Method 8290A names them Internal Standards while Method 1613B uses the Labeled Compounds terminology.

6.11 Injection Standards: Isotopes added prior to injection to determine the recoveries of the Extraction and Cleanup Standards. Method 8290A names them Recovery Standards while Method 1613B calls them Internal Standards.

6.12 Internal Standard (ISTD): A known amount of standard added to a test portion of a sample as a reference for evaluating the retention time and concentration of dependent analytes and controlling the precision and bias of the applied analytical method.

6.13 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.14 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.15 Matrix Spike and Matrix Spike Duplicate (MS and MSD): Two separate aliquots of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD adjusted. Percent recovery is calculated for both aliquots, and RPD is calculated between the two.

6.16 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.17 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.

6.18 Quantitation Limits (also PQL, RL): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the linear calibration range.

6.19 Sampling Standards: Isotopes added prior to field sampling for Method 0023A that are used to measure the efficiency of the sampling step alone.

7.0 INTERFERENCES/LIMITATIONS

7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.

7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.

7.3 Contamination may also occur in the GC/MS system. High boiling materials tend to build up in the injection port and the front end of the column. The analyst should maintain a thorough working knowledge of keeping the injection port free of contamination, including changing out the septum, injection port liner, O-ring, ferrule, and gold seal.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. If carryover is suspected, potentially impacted samples must be re-analyzed after any needed maintenance, solvent replacement, and/or cleaning has been done.

8.0 SAFETY PRECAUTIONS AND WARNINGS

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

NO OCCUPATIONAL EXPOSURE LIMIT FOR DIOXIN HAS BEEN ESTABLISHED. IT IS A KNOWN AND PROBABLE HUMAN CARCINOGEN.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN USING METHYLENE CHLORIDE.

8.1 Eye protection should be worn when handling samples, reagents, or standards.
NOTE: Contact lenses pose a special problem; soft lenses may absorb irritants and all lenses concentrate them. DO NOT wear contact lenses in the laboratory.

8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. CFA maintains a current reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.

8.3 Personal Protective Equipment (PPE)

8.3.1 Gloves and eye protection should be worn when handling reagents, solvents, standards and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied.

8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, management should be notified of all accidents.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers and fire blankets are located in the lab. Training is available on the proper operation of this equipment.

8.8 The analyst must use care when assembling and operating instrumentation. Check to see that the gas chromatograph equipment is properly assembled and hooked up to the proper gas cylinder and power, referencing the appropriate manual. Analytical equipment must only be operated by qualified personnel.

8.9 For further safety instructions, consult the Safety Manual, CF-LB-N-001.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

9.1 Equipment associated with this method includes:

- 9.1.1 Gas tight syringes
- 9.1.2 2 mL high recovery (conical) autosampler vials and storage racks
- 9.1.3 Teflon crimp tops
- 9.1.4 Crimper/De-crimper
- 9.1.5 GC Columns
  - 9.1.5.1 Agilent DB5-MS or equivalent; 60 m, 0.25 mm, 0.25 um
  - 9.1.5.2 Agilent DB-225 or equivalent; 30 m, 0.25 mm, 0.25 um
- 9.1.6 Quartz/Glass injection port liners
- 9.1.7 Injection port liner O-ring seals
- 9.1.8 Gold seals
- 9.1.9 Ferrules
- 9.1.10 Column cleaving tool
- 9.1.11 Septa (thermogreen)
- 9.1.12 10-100 uL adjustable air displacement pipette with disposable tips

9.2 Instrumentation

- 9.2.1 Waters Autospec Premier high resolution mass spectrometer
- 9.2.2 Agilent 7890 Gas Chromatograph
  - 9.2.2.1 A suggested temperature program for primary analysis follows:

Initial Temp.	140° C
Hold Time	1.0 min.
Rate 1	20° C/min.
Temperature 2	180° C
Time 2	2°/min
Temperature 3	235° C
Rate 3	30° C/min.
Final Temp.	290° C

Hold Time	13 min.
Run Time:	45 minutes (may vary due to column length or flow rate)
Solvent Delay:	18.0 min.
Splitless Valve Time:	1.5 min.
Flow:	1.8 mL/min.
Mass Range:	See descriptor definitions (Table 2)

NOTE: These instrument conditions and rates are guidelines which may change.

9.2.3 LEAP Technologies GC PAL Autosampler

9.2.3.1 Suggested parameters:

Sample volume – 1 µL
Air volume – 0.5 µL
Solvent push volume – 1 µL
Number of sample washes - 0
Solvent washes - 30
Sample viscosity wait – 1 second
Number of sample pumps - 0
Injection mode - Fast

10.0 REAGENTS AND STANDARDS

10.1 Reagents and standards

10.1.1 Nonane

10.1.2 Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. These may include extraction standard, matrix spiking standard, cleanup standard, injection standard, as well as others. Source standards expire per the vendor expiration date or after five years from the date opened, whichever is shorter. Please reference GL-LB-E-007 and GL-OA-E-002 for further information regarding standards and their preparation.

10.1.3 Initial Calibration (ICAL) Standards: Certified calibration standards are purchased from commercial vendors at a minimum of five concentration levels. One of the calibration standards is at a concentration near, but above, the method detection limit; the others should correspond to the expected range of compounds found in samples. Calibration standards expire after a maximum of five years and should be monitored frequently for signs of degradation.

10.1.4 Calibration Verification Standards (CVS, CCAL, CS3WT): A certified CVS is purchased from a second source commercial vendor at a concentration that is near to the midpoint of the calibration curve.

10.1.5 Window Defining Mix and Column Performance Mix (WDM and CPM): A standard containing the first and last eluters for each homolog group, as well as

the dioxin and furan isomers used to demonstrate isomer specificity on the GC column in use. These may be contained in the same standard as the calibration verification (known as CS3WT).

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 Sample extracts have a 45-day holding time from the date of extraction by methods 8290A and 0023A, and a 365 day holding time from the date of extraction by 1613B. Note that per method 8290A, tissue extracts must be completely analyzed by 45 days from collection.

11.2 Sample extracts are delivered from the prep lab to the instrument lab and are stored in a darkened hood at room temperature. The extracts are usually grouped according to preparation batches and are accompanied by the batch pull sheet and other pertinent paperwork.

11.3 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples planned to run into their custody prior to analysis.

11.4 All sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION

12.1 Before extracts can be analyzed on the instrument, they must first be evaporated to dryness under nitrogen and then spiked with injection standard to set the final volume nominally at 20 µL. A determination must also be made as to whether the extract should be diluted. The decision to dilute a sample extract is based on a number of factors: sample screening, historical data about the sample or sample site, the appearance of the extract (color, viscosity, incidental odor, turbidity, etc.), or regulatory considerations. The experience of the analyst is invaluable in making this determination.

NOTE: Sample extracts may contain multiple layers or sediment. Samples that contain sediment are returned to cleanup. Multiple layers are treated on a case-by-case basis. If the extract can be homogenized, then a uniform sample is achieved. If the extract remains bi-phasic, the PM and client are contacted for further guidance.

12.2 If a sample is to be analyzed without dilution ('neat'), 2 nanograms of injection standard solution is added to the extract using a pipette (20 µL of a 0.1 ng/µL = 100 pg/µL extract concentration). A cap is then placed on the vial and secured by crimping before vortexing the sample to ensure complete mixing and vial wall washing.

12.3 If samples require dilution, the dilution is made using nonane or appropriate solvent. If not previously added, 2 nanograms of JS is added to the autosampler vial. Dilution prep may involve the addition of supplemental extraction standard (ES) and is documented in the injection prep logbook.

12.4 Once samples are prepped, they are ready to be injected onto the instrument. An autosampler is used to inject standards and sample extracts on the instrument.

12.5 The need for dilution may also be determined after analysis is performed, and may still be performed as above. Under normal circumstances, a sample would be diluted if any chromatographic peaks saturate the detector.

13.0 QUALITY CONTROL REQUIREMENTS

Typically a blank (LMB), laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) are extracted and analyzed with each prep batch. Other client requirements may include a matrix spike (MS) and matrix spike duplicate (MSD) or sample duplicate (DUP).

13.1 Blanks

13.1.1 A blank is extracted with each batch of 20 or fewer samples to demonstrate that interferences from glassware, reagents and the analytical system are under control. Blanks are carried through all stages of sample preparation and analysis. For Method 1613B, an acceptable blank must be below the minimum levels listed in Table 2 of the method for all analytes. For Methods 8290A and 0023A, all analytes must be below the Lower Method Calibration Limits.

13.1.2 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 6 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. CFA has established an internal guideline of 70-130% for Method 8290 cleanup standards.

13.2 Laboratory Control Samples and Matrix Spikes

13.2.1 The spiking standard for LCS/LCSDs and MS/MSDs contains all analytes listed in Table 4. For each LCS, LCSD, MS and MSD, the concentration of each analyte and its percent recovery are calculated as shown in Sec.17.4.1 and 17.4.5. For methods 8290A and 0023A, percent recoveries should be within 70-130%. For method 1613B, recovered concentrations should be within the limits in Table 5.

13.2.2 If recovery is not within these limits, the data may need to be re-checked for errors, or the samples and QC may need to be re-analyzed. In addition, the instrumentation may need to be checked for performance problems. If the LCS fails to meet acceptance criteria due to low recovery, the associated samples may have to be re-extracted and re-analyzed when possible. If one or more recoveries are high in the LCS and these analytes are not detected in the samples, the event should be documented and data may be reported. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.

NOTE: Many clients have contract specific criteria that must be considered when evaluating recovery of the Quality Control samples.

13.2.3 The percent recovery of each labeled standard (extraction and cleanup) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 5 for method 1613B. For methods 8290A and 0023A, extraction standard recoveries must be within 40-135%. Sampling standards for Method 0023A must be within 70-130%. CFA has established an internal guideline of 70-130% for Method 8290 cleanup standards.

13.3 Samples

13.3.1 The percent recovery of each labeled standard (as listed in SOP CF-OA-E-001) is calculated as shown in Sec. 17.4.5. Recoveries must be within the limits in Table 5 for method 1613B or 40-135% for method 8290A.

13.3.2 Calculated EDLs should be below the PQLs in Table 1. Any reported EDLs above the PQLs should be noted in the case narrative.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

14.1 Mass spectrometer performance

14.1.1 The mass spectrometer is operated in 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. Static resolving power checks must be performed at the beginning and at the end of each 12-hr period of operation. Corrective action must be implemented whenever the resolving power does not meet the requirement.

14.1.1.1 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. A lock-mass ion from the reference compound PFK is 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. Lock mass ions may be found in the descriptor table, Table 2. 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 these 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 an increase in downtime for source cleaning.

14.1.2 Documentation of the instrument resolving power must be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760) obtained during the above peak matching experiment by using the low mass PFK ion at m/z 304.9824 as a reference. 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 2) 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).

14.2 System Performance

System performance criteria are presented below. The laboratory may use the recommended GC column described in Sec. 9.1. The laboratory must document that all applicable system performance criteria are met before sample analysis begins. Sec. 9.2.2 provides recommended GC conditions that may be used to satisfy the required criteria. Mass spectrometer resolving power checks must be performed at the beginning and the end of each 12-hr period of operation. A GC column performance check is required at the beginning of each 12-hr period during which samples are analyzed. For Method 1613B, a continuing calibration must be performed at the beginning of the sequence, while for Methods 0023A and 8290A, continuing calibrations must be performed at both the beginning and the end of a sequence. An ending continuing calibration may also serve as the beginning check for the next sequence.

14.2.1 GC Column performance check

14.2.1.1 Inject 1 µL of an aliquot of the column performance check solution (Sec. 10.1.5) and acquire selected ion monitoring (SIM) data within a total cycle time of $\leq$ 1 second. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of $\leq$ 25 percent (Figure 1), where:

Valley percent = (x/y) × 100

x = measured as in Figure 1 from the 2,3,7,8-closest TCDD eluting isomer

y = the peak height of 2,3,7,8-TCDD

For 2378-TCDF confirmatory analysis, the chromatographic separation between 2378-TCDF and its closest eluters must be resolved with a valley of $\leq$ 25 percent.

14.2.1.2 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 described in this SOP. Their retention times are used to determine the five homologue retention time windows that are used for qualitative (Sec. 15.3.1.1) and quantitative purposes. All peaks (including 13C12-2,3,7,8-TCDD) should be labeled and identified on the chromatograms. All first eluters of a homologous series should be labeled with the letter "F," and all last eluters of a homologous series should be labeled with the letter "L". Any individual selected ion current profile (SICP) or the reconstructed homologue ion current constitutes an acceptable form of data presentation. A SICP for the labeled compounds is also required.

14.2.1.3 Particular caution should be exercised for the switching time between the last tetra-chlorinated congener (1,2,8,9-TCDF) and the first penta-chlorinated congener (1,3,4,6,8-PeCDF), as these two compounds elute within 15 sec of each other on the 60-m DB-5

column, and overlap on the 60-m DB-5ms column. Both congeners must be acquired within one analysis.

14.2.1.4 The absolute retention time of $^{13}C_{12}$-1,2,3,4-TCDD must exceed 25.0 minutes on the primary GC column in use, and 15.0 minutes on the confirmatory GC column.

14.3 Initial Calibration

14.3.1 Prior to running a multi-level calibration, take precautions to ensure that the instrument meets system performance criteria. The analyst must document that all system performance criteria are met before analyzing an initial calibration.

14.3.2 Initial calibration is required before any samples are analyzed for PCDDs and PCDFs and must meet the acceptance criteria listed below. Initial calibration is also required if any routine calibration does not meet the required criteria listed in Sec. 15.2, and at a minimum, annually.

14.3.3 At a minimum, all five high-resolution concentration calibration solutions listed in Table 4 must be used for the initial calibration.

14.3.4 Tune the instrument with PFK to meet the above-specified system performance criteria.

14.3.5 Inject the GC column performance check solution and acquire SIM mass spectral data. The total cycle time for each descriptor must be < 1 second. The laboratory must not perform any further analysis until it is demonstrated and documented that the criteria listed in Sec. 15.1.1.1 are met.

14.3.6 By using the same conditions (GC and MS) that produced acceptable results with the column performance check solution, analyze each of the five concentration calibration solutions. Each injection must meet the following ion ratio and signal-to-noise (S/N) requirements:

14.3.6.1 The ratio of the areas of the integrated ion current for the ions appearing in Table 2 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) in Table 3. These ion ratio requirements must be within the specified control limits simultaneously in one run. It is the analyst's responsibility to take corrective action if the ion abundance ratios are outside the limits.

14.3.6.2 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the S/N ratio must be better than or equal to 10. Manual measurement of S/N is required for any GC peak that has an apparent S/N of less than 15:1. The result of the calculation must appear on the SICP above the GC peak in question.

14.3.7 Calculate the 17 relative response factors (RF) for unlabeled target analytes relative to their appropriate internal standards. Also calculate the RFs for the ESs and CSs relative to the appropriate injection standards according to the following formula:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Sum of the Areas of the two characteristic ions for the compound being measured.

Ais = Sum of the Areas of the two characteristic ions for the specific internal standard.

Cis = Concentration of the specific internal standard.

Cx = Concentration of the compound being measured.

The RF is a dimensionless quantity; the units used to express Cis and Cx must be the same.

14.3.8 The RF for other isomers within a homolog group shall be determined from the average RF of the 2,3,7,8-substituted isomers. For example, the RF for non-2,3,7,8-substituted HxCDD isomers (totals peaks) is the average of the three 2,3,7,8-substituted isomers. NOTE: If only one 2,3,7,8-substituted isomer is present in the calibration then use that isomer's RF for all isomers within its homolog group.

14.3.9 Because more than five calibration levels may be analyzed, the analyst may choose to deactivate one or more levels globally. If a level is not used, it will be deactivated in the method for all analytes in that calibration mixture. In some cases the upper level(s) of the calibration may be deactivated in order to meet method criteria for single compounds. This practice results in a narrower calibration range. The low standard representing the PQL cannot dropped. Please note that this practice does not represent "cherry picking," which is acknowledged as an unacceptable laboratory practice.

14.3.10 The average RF must be calculated for each compound as follows:

$$RF_{avg} = \frac{\sum_{i=1}^{n} X}{n}$$

Where:

N = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

14.3.11 Criteria for acceptable initial calibration

The criteria listed below for acceptable calibration must be met before sample analyses are performed.

14.3.11.1 Per method 8290A, the percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the nine labeled reference compounds must not exceed ± 20 percent. These limits also apply to Method 0023A. Per method 1613B, the percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for

the fifteen labeled reference compounds must not exceed ± 35 percent.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = relative standard deviation

$\bar{x}$ = mean of 5 or more initial RFs for a compound

SD = standard deviation of average RFs for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (X - A)^2}{n-1}}$$

where:

n = number of calibration levels

X_i; i=1 to n, are the compounds RF values for each calibration point

A = average of the RFs from above

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

15.1 Resolution check

15.1.1 At the beginning and end of each 12-hour window, mass resolution must be tuned and/or verified. A static resolving power of at least 10,000 must be demonstrated at appropriate masses before analysis is performed.

15.1.2 Using a PFK molecular leak, tune the instrument to the minimum required resolving power of 10,000 at m/z 330.9792 (for day to day operations, the instrument may be tuned to approximately 11,000). Verify that the exact mass of m/z 380.9760 is within 5 ppm of the required value.

15.2 Column Performance/Window Defining/Continuing Calibration Check (CS3WT)

15.2.1 Inject 1 uL of the CS3WT or CPM. Verify that all column performance and window defining criteria in Section 14.2.1 have been met.

15.2.2 The CS3WT also contains the analytes for continuing calibration. The initial calibration curve for each compound of interest must be verified once every 12 hours.

Calculate the percent difference using:

$$\%\ \text{Difference} = \frac{\left|\overline{RF}_i - RF_c\right|}{\overline{RF}_i} \times 100$$

Where:

$\overline{RF}_i$ = average response factor from initial calibration

RF_c = response factor from current CS3WT

Calculate analyte concentrations using:

$$PCDD/PCDF = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{RF}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard in pg/uL
RF = Average RF from the ICAL for the compound

15.2.2.1 For methods 0023A and 8290A, if the percent difference for each native analyte in the CS3WT is $\leq$ 20%, and for each labeled analyte is $\leq$ 30%, the initial calibration is assumed to be valid. For method 1613B, analyte concentrations must fall within the limits in Table 7. If the criteria are not met, corrective action should be taken. If no source of the problem can be determined after corrective action has been taken, a new calibration may need to be generated.

15.2.2.2 All ion ratios must be within the limits in Table 3.

15.2.2.3 For methods 0023A and 8290A, if no more than two unrelated compounds in the continuing calibration check performed at the end of a 12-hour period fail by no more than $\pm$25% for the 17 unlabeled compounds and $\pm$35% for the 9 labeled compounds, the average RF values from the beginning and ending continuing calibration checks should be used to compute the analyte concentrations, instead of the RF values obtained from the initial calibration. No further sample analyses should be performed until an acceptable calibration is achieved.

15.3 Sample Analysis

15.3.1 Data Interpretation

15.3.1.1 Qualitative Determination

For a peak to be identified as a PCDD or PCDF, it must meet all of the criteria listed below.

15.3.1.1.1 The signals for the two m/z's being monitored must be present and maximize within ±2 seconds of each other.

15.3.1.1.2 The signal-to-noise ratio between the two m/z's must be $\geq$ 2.5 for native compounds and $\geq$ 10 for labeled compounds.

15.3.1.1.3 Ion ratios must be within the limits in Table 3.

15.3.1.1.4 Relative Retention Times

15.3.1.1.4.1 For Methods 0023A and 8290A, congeners which have an isotopically labeled compound must fall within -1 to +3 seconds of the labeled compound. Congeners with no labeled compound must be within 0.005 retention time units

of the RRT measured in the continuing calibration.

15.3.1.1.4.2 For Method 1613B, relative retention times must be within the RRT limits found in Table 8.

15.3.1.1.4.3 For non-2378 peaks, retention times must be within the retention time windows established by the analysis of the window defining mixture (Sec. 14.2.1.2).

15.3.1.1.5 For PCDFs, no peak may be present in the associated PCDPE channel at the same retention time. If a PCDPE peak is present, the PCDF peak should be reported with a flag denoting the interference.

15.3.1.1.6 Any sample in which 2378-TCDF has been identified at or above the method reporting limit must be confirmed on a second column (DB-225 or equivalent).

15.3.1.2 Calibration Limit Exceedance

15.3.1.2.1 If a compound in a sample exceeds the upper calibration limit, all subsequent samples must be checked for carryover contamination.

15.3.1.2.2 When a subsequent sample is non-detect for the compound in question, the sequence is again considered acceptable for reporting.

15.3.1.2.3 All affected samples between the exceeding sample and the non-detect sample must be re-analyzed.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1 Preventive maintenance on a HRGC/HRMS system involves the following basic areas:

16.1.1 Vacuum pumps for the inlets, source, and analyzer need a change of oil about every year or when system performance indicates it is needed.

16.1.2 The GC injection port is cleaned as needed, approximately once a week. It is recommended that the septum and injection port liner be replaced at the time of cleaning. Additionally, the gold plated seal should be cleaned or replaced.

16.1.3 Ion source maintenance is usage dependent. The type and quantity of samples that have been injected determine the frequency of ion source cleaning and filament replacement.

16.1.4 Autosampler maintenance is primarily that of cleanliness. Most autosamplers need their moving parts to be clean and lightly lubricated. The most frequent corrective maintenance is that of changing the syringe, usually about once per month.

16.1.5 Instrument maintenance logs are kept with each instrument and serve as a record of all the maintenance that has been done on the instrument.

16.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

16.2.1 Service is provided to the instrument via the analyst, the in-house instrument service engineer, or a technical support specialist from the manufacturer. When instrument failure occurs, different parts of the instrument are isolated to determine the root cause. For example, the injection port may be capped off if a leak is suspected to prove the leak is/is not coming from that source. Instrument maintenance logbooks are kept for each instrument detailing the type of maintenance performed on the instrument and when it was performed. Preventive maintenance visits are scheduled annually for the mass spectrometers.

16.2.2 Analytical GC columns are clipped or replaced when the existing column shows signs of excessive degradation or the inability to properly resolve chromatographic peaks. Excessive peak tailing, poor responses, and baseline disturbances may also indicate that the column needs to be replaced.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

17.1 Data are evaluated qualitatively and quantitatively using a software program such as Waters MassLynx, or equivalent data system.

17.2 Data are reviewed, and a hard copy is generated. If manual integrations are made, a hard copy of the manual integration is printed and initialed by the analyst and included with the raw data.

17.3 Additional supporting documentation, such as totals pages generated by the software may be included with the data.

17.4 Quantitative Analysis

17.4.1 The concentration (ng/L for aqueous, ng/g for solids) of each identified compound in the sample is calculated as follows:

$$PCDD/PCDF = \frac{\left(A_{unk}^{ion1} + A_{unk}^{ion2}\right)}{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times D \times \overline{RF}}$$

Where:

A_{unk} and A_{ES} = the integrated area for each ion monitored.
Q_{ES} = the amount of extraction standard added to the sample in nanograms
W_{unk} = the initial sample aliquot size, in liters for waters and in grams for solids.
D = (% moisture in sample)/100, or 1 for waters
$\overline{RF}$ = Average RF from the ICAL for the compound

17.4.2 The estimated detection limit (EDL) is calculated as follows:

$$EDL_{ppt} = 3 \times \frac{\left(H_{unk}^{ion1} + H_{unk}^{ion2}\right)}{\left(H_{ES}^{ion1} + H_{ES}^{ion2}\right)} \times \frac{Q_{ES}}{W_{unk} \times \overline{RF}}$$

Where:

H_{unk} = the height of the noise present in each ion monitored.

H_{ES} = the height of the extraction standard peak in each ion monitored.
3 = signal-to-noise factor for minimum height of peak.

17.4.3 The estimated maximum possible concentration (EMPC) is calculated in the same manner as a concentration (Section 17.4.1).

17.4.4 The concentration of each extraction and cleanup standard is calculated as follows:

$$ES_{ng} = \frac{\left(A_{ES}^{ion1} + A_{ES}^{ion2}\right)}{\left(A_{JS}^{ion1} + A_{JS}^{ion2}\right)} \times \frac{Q_{JS}}{\overline{RF}}$$

Where:

A_{ES} and A_{JS} = the integrated area for each ion monitored.
Q_{JS} = the amount of injection standard added to the sample in nanograms
$\overline{RF}$ = Average RF from the ICAL for the compound

The cleanup standard concentration is calculated as above, substituting the area of the individual cleanup standard ions for the extraction standard ions.

17.4.5 Percent recovery is calculated as follows:

$$\%R = \frac{R_{ng}}{S_{ng}} \times 100$$

Where:

R_{ng} = the amount of standard recovered in nanograms.
S_{ng} = the amount of standard spiked in nanograms.

18.0 POLLUTION/CONTAMINATION

18.1 Work area should be maintained free of dust and dirt accumulations.

18.2 Fume hoods are utilized to remove fumes and reduce the risk of airborne contaminants to ensure personnel safety. Hoods are monitored in accordance with CF-FC-E-003 for Fume Hood Face Velocity Performance Checks.

18.3 The laboratory area is restricted to authorized personnel.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

19.1 A review process is used to insure the quality of the data. Raw data are reviewed first by the analyst, then by a second (peer) analyst or a data validator. When the analyst is satisfied that the data have been correctly processed and uploaded to the LIMS, a data report is generated from AlphaLIMS. The AlphaLIMS report along with the raw data and supporting documentation, such as a run log and case narrative, are submitted for review to the data validator or another experienced analyst. The reviewer goes through the raw data as if he/she was working it up for the first time and verifies that they are correct. In addition, he/she must make sure that the data have been correctly entered into AlphaLIMS. AlphaLIMS reports may be self-reviewed. If errors are discovered in either the raw data or the AlphaLIMS report, then the two analysts should discuss the differences and how best to resolve them. In some cases, the peer review process may uncover errors that lead to a sample being re-extracted or re-run. In cases such as these, a nonconformance report (NCR) should be completed and submitted to the Quality

department. It is recommended that a copy of the NCR be given to the prep analyst if it involves a re-extraction and that a copy be kept with the original data.

19.2 Once the data review has been completed by the reviewer, the batch is returned to the analyst for corrections (if applicable) and the status is updated from REVW to DONE in AlphaLIMS.

19.3 Data may be transmitted automatically to AlphaLIMS. This automatic "upload" procedure may be activated prior to data review or after data review is complete. In either case, the data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data may require instrument maintenance, re-analysis, re-extraction, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a nonconformance report and/or corrective action should be initiated in accordance with CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective Action.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting is used to determine the appropriate action to take when an initial or continuing calibration, blank and/or laboratory control sample fails to meet the acceptance criteria defined for the method. Troubleshooting may involve one or more of the following actions:

21.1 If analytes in a multi-point calibration fail to meet specified criteria, additional standards for the failing compounds may need to be reanalyzed. If they still do not meet specifications, instrument maintenance or new standards may be required before work is continued.

21.2 If a continuing calibration fails to meet specified criteria, instrument tuning or inlet maintenance may be required. If routine maintenance procedures fail to produce a second consecutive calibration verification within acceptance criteria, then the laboratory must demonstrate acceptable performance after further corrective action with two consecutive calibration verifications, or a new initial calibration must be analyzed.

21.3 If a method blank fails to meet defined criteria, the source of contamination should be found and eliminated before proceeding with analysis.

22.0 RECORDS MANAGEMENT

22.1 Run logs are generated for each instrument each day that the instrument is run. These run logs serve as records of what is run on the instrument, including samples, QC, calibrations, tunes, etc. Additional information is provided in the run log, including the analyst's initials, run date and time, and file name.

22.2 Raw data are stored in the lab in filing cabinets and/or boxes as long as there is space available. When space runs out, the data are boxed and sent to storage.

22.3 All records generated as a result of this procedure are maintained as quality documents in accordance with CF-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Sample extracts that have been run are temporarily stored in case they have to be reanalyzed. Once space is no longer available to keep them in the lab, they are moved to Waste Disposal where they are handled and disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

24.0 REFERENCES

24.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 8290A, "Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatorgraphy/ High Resolution Mass Spectrometry (HRGC/HRMS)," Rev. 1, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

24.2 Method 1613, "Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS," Rev. B, Oct. 1994. USEPA, Office of Water, Engineering and Analysis Division, 401 M Street SW, Washington, D.C. 20460.

24.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb. 2007. Method 0023A, "Sampling Method for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofuran Emissions From Stationary Sources," Rev. 1, Dec. 1996. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

24.4 National Environmental Laboratory Accreditation Conference (NELAC) Standard, July 2003.

25.0 HISTORY

Revision 1: Section 15.3.1.2 added.

Revision 2: Absolute RT information added in 14.2.1.4; Calibration limit exceedance information added in section 15.3.1.2; Table 8 footnote describing RRT window adjustment to column used.

Revision 3: Method 0023A requirements added.

Revision 4: 2378-TCDF confirmation procedure and requirements added.

TABLE 1: METHOD ANALYTES AND PQLs

Analyte	Solid/Tissues (pg/g)	Aqueous (pg/L)	Air (pg)	CAS Number*
2378-TCDD	1	10	10	1746-01-6
12378-PeCDD	5	50	50	40321-76-4
123478-HxCDD	5	50	50	39227-28-6
123678-HxCDD	5	50	50	57653-85-7
123789-HxCDD	5	50	50	19408-74-3
1234678-HpCDD	5	50	50	35822-39-4
OCDD	10	100	100	3268-87-9
2378-TCDF	1	10	10	51207-31-9
12378-PeCDF	5	50	50	57117-41-6
23478-PeCDF	5	50	50	57117-31-4
123478-HxCDF	5	50	50	70648-26-9
123678-HxCDF	5	50	50	57117-44-9
234678-HxCDF	5	50	50	60851-34-5
123789-HxCDF	5	50	50	72918-21-9
1234678-HpCDF	5	50	50	67562-39-4
1234789-HpCDF	5	50	50	55673-89-7
OCDF	10	100	100	39001-02-0

* Chemical Abstract Services number

TABLE 2: MASS DESCRIPTORS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	50	10
1	2	305.8987	50	10
1	3	315.9419	50	10
1	4	304.9824	50	10
1	5	304.9824	(Lock)	10
1	6	317.9389	50	10
1	7	319.8965	50	10
1	8	321.8936	50	10
1	9	327.8847	50	10
1	10	331.9368	50	10
1	11	333.9339	50	10
1	12	339.8597	50	10
1	13	341.8568	50	10
1	14	375.8364	50	10
2	1	339.8597	50	10
2	2	341.8568	50	10
2	3	351.9	50	10
2	4	353.897	50	10
2	5	355.8546	50	10
2	6	357.8517	50	10
2	7	366.9792	50	10
2	8	366.9792	(Lock)	10
2	9	367.8949	50	10
2	10	369.8919	50	10
2	11	409.7974	50	10
3	1	373.8207	50	10
3	2	375.8178	50	10
3	3	380.976	50	10
3	4	380.976	(Lock)	10
3	5	383.8639	50	10
3	6	385.861	50	10
3	7	389.8156	50	10
3	8	391.8127	50	10
3	9	401.8559	50	10
3	10	403.853	50	10
3	11	445.7555	50	10
4	1	407.7818	50	10
4	2	409.7788	50	10
4	3	417.8253	50	10
4	4	419.822	50	10
4	5	423.7767	50	10
4	6	425.7737	50	10
4	7	430.9728	50	10
4	8	430.9728	(Lock)	10
4	9	435.8169	50	10
4	10	437.814	50	10
4	11	479.7165	50	10
5	1	441.7427	50	10
5	2	443.7398	50	10
5	3	454.9728	50	10
5	4	454.9728	(Lock)	10
5	5	457.7377	50	10
5	6	459.7348	50	10
5	7	469.778	50	10
5	8	471.775	50	10
5	9	513.6775	50	10

TABLE 3: THEORETICAL ION RATIOS AND CONTROL LIMITS

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6[a]	0.51	0.43	0.59
7	1.05	0.88	1.20
7[b]	0.44	0.37	0.51
8	0.89	0.76	1.02

[a] Used only for ^{13}C-HxCDF
[b] Used only for ^{13}C-HpCDF

TABLE 4: 1613B LIMITS FOR TETRA ONLY TESTS

Compound Name	Test Conc. (pg/µL)	CCAL Limits (pg/µL)	OPR Limits (pg/µL)	Sample Limits (pg/µL)
2,3,7,8-TCDD	10	8.2 - 12.3	7.3 - 14.6	-
2,3,7,8-TCDF	10	8.6 - 11.6	8.0 - 14.7	-
$^{13}C_{12}$-2,3,7,8-TCDD	100	85 - 117	25 - 141	31 - 137
$^{13}C_{12}$-2,3,7,8-TCDF	100	76 - 131	26 - 126	29 - 140
$^{37}Cl_4$-2,3,7,8-TCDD	10	8.3 - 12.1	3.7 - 15.8	4.2 - 16.4

TABLE 5: INITIAL CALIBRATION CONCENTRATIONS

Analyte	Concentration (pg/uL) CS-0.5	CS-2	CS-3	CS-4	CS-5
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
Extraction Standards					
^{13}C-2378-TCDD	100	100	100	100	100
^{13}C-2378-TCDF	100	100	100	100	100
^{13}C-12378-PeCDD	100	100	100	100	100
^{13}C-12378-PeCDF	100	100	100	100	100
^{13}C-23478-PeCDF	100	100	100	100	100
^{13}C-123678-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDD	100	100	100	100	100
^{13}C-123478-HxCDF	100	100	100	100	100
^{13}C-123678-HxCDF	100	100	100	100	100
^{13}C-123789-HxCDF	100	100	100	100	100
^{13}C-234678-HxCDF	100	100	100	100	100
^{13}C-1234678-HpCDD	100	100	100	100	100
^{13}C-1234678-HpCDF	100	100	100	100	100
^{13}C-1234789-HpCDF	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200
Cleanup Standards					
^{37}Cl-2378-TCDD	0.25	2	10	40	200
Injection Standards					
^{13}C-1234-TCDD	100	100	100	100	100
^{13}C-123789-HxCDD	100	100	100	100	100

TABLE 6: METHOD 1613B LCS LIMITS

LCS Recovery Limits		
Analyte	**Amount Spiked**	**Limit**
	(pg/uL)	**(pg/uL)**
2378-TCDD	10	6.7-15.8
12378-PeCDD	50	35-71
123478-HxCDD	50	35-82
123678-HxCDD	50	38-67
123789-HxCDD	50	32-81
1234678-HpCDD	50	35-70
OCDD	100	78-144
2378-TCDF	10	7.5-15.8
12378-PeCDF	50	40-67
23478-PeCDF	50	34-80
123478-HxCDF	50	36-67
123678-HxCDF	50	42-65
123789-HxCDF	50	39-65
234678-HxCDF	50	35-78
1234678-HpCDF	50	41-61
1234789-HpCDF	50	39-69
OCDF	100	63-170
^{13}C-2378-TCDD	100	20-175
^{13}C-12378-PeCDD	100	21-227
^{13}C-123478-HxCDD	100	21-193
^{13}C-123678-HxCDD	100	25-163
^{13}C-1234678-HpCDD	100	26-166
^{13}C-OCDD	200	26-397
^{13}C-2378-TCDF	100	22-152
^{13}C-12378-PeCDF	100	21-192
^{13}C-23478-PeCDF	100	13-328
^{13}C-123478-HxCDF	100	19-202
^{13}C-123678-HxCDF	100	21-159
^{13}C-123789-HxCDF	100	17-205
^{13}C-234678-HxCDF	100	22-176
^{13}C-1234678-HpCDF	100	21-158
^{13}C-1234789-HpCDF	100	20-186
^{37}Cl-2378-TCDD	10	3.1-19.1

TABLE 7: METHOD 1613B ES (SAMPLES & LMB) RECOVERY LIMITS

Compound Name	Amount Spiked (pg/µL)	Limits %
$^{13}C_{12}$-2,3,7,8-TCDD	100	25 - 164
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25 - 181
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32 - 141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28 - 130
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23 - 140
$^{13}C_{12}$-OCDD	200	17 - 157
$^{13}C_{12}$-2,3,7,8-TCDF	100	24 - 169
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24 - 185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21 - 178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26 - 152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26 - 123
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	28 - 136
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	29 - 147
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28 - 143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26 - 138
$^{37}Cl_4$-2,3,7,8-TCDD	10	35 - 197

TABLE 8: METHOD 1613B CONTINUING CALIBRATION LIMITS

Compound Name	CCAL (pg/µL)	Limits (pg/µL)	Compound Name	CCAL (pg/µL)	Limits (pg/µL)
2,3,7,8-TCDD	10	7.8 - 12.9	$^{13}C_{12}$-2,3,7,8-TCDD	100	82 - 121
1,2,3,7,8-PeCDD	50	39 - 65	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	62 - 160
1,2,3,4,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	85 - 117
1,2,3,6,7,8-HxCDD	50	39 - 64	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	85 - 118
1,2,3,7,8,9-HxCDD	50	41 - 61	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	72 - 138
1,2,3,4,6,7,8-HpCDD	50	43 - 58	$^{13}C_{12}$-OCDD	200	96 - 415
OCDD	100	79 - 126	$^{13}C_{12}$-2,3,7,8-TCDF	100	71 - 140
2,3,7,8-TCDF	10	8.4 - 12	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	76 - 130
1,2,3,7,8-PeCDF	50	41 - 60	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	77 - 130
2,3,4,7,8-PeCDF	50	41 - 61	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	76 - 131
1,2,3,4,7,8-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	70 - 143
1,2,3,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	73 - 137
2,3,4,6,7,8-HxCDF	50	44 - 57	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	74 - 135
1,2,3,7,8,9-HxCDF	50	45 - 56	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	78 - 129
1,2,3,4,6,7,8-HpCDF	50	45 - 55	$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	77 - 129
1,2,3,4,7,8,9-HpCDF	50	43 - 58	$^{37}Cl_4$-2,3,7,8-TCDD	10	7.9 - 12.7

OCDF	100	63 - 159

TABLE 9: METHOD 1613B RELATIVE RETENTION TIME LIMITS

Compound	RRT Reference	RRT Limits
2,3,7,8-TCDF	13C -2,3,7,8-TCDF	0.999 - 1.002
2,3,7,8-TCDD	13C -2,3,7,8-TCDD	0.999 - 1.001
1,2,3,7,8-PeCDF	13C -1,2,3,7,8-PeCDF	0.999 - 1.001
2,3,4,7,8-PeCDF	13C -2,3,4,7,8-PeCDF	1.000 - 1.001
1,2,3,7,8-PeCDD	13C -1,2,3,7,8-PeCDD	0.999 - 1.001
1,2,3,4,7,8-HxCDF	13C -1,2,3,4,7,8-HxCDF	0.999 - 1.001
1,2,3,6,7,8-HxCDF	13C -1,2,3,6,7,8-HxCDF	0.999 - 1.001
1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDF	1.000 - 1.001
2,3,4,6,7,8-HxCDF	13C -2,3,4,6,7,8,-HxCDF	0.999 - 1.001
1,2,3,4,7,8-HxCDD	13C -1,2,3,4,7,8-HxCDD	1.000 - 1.001
1,2,3,6,7,8-HxCDD	13C -1,2,3,6,7,8,-HxCDD	0.999 - 1.001
1,2,3,7,8,9-HxCDD	13C -1,2,3,6,7,8,-HxCDD	1.006 - 1.008
1,2,3,4,6,7,8-HpCDF	13C -1,2,3,4,6,7,8-HpCDF	0.999 - 1.001
1,2,3,4,7,8,9-HpCDF	13C -1,2,3,4,7,8,9-HpCDF	1.000 - 1.001
1,2,3,4,6,7,8-HpCDD	13C -1,2,3,4,6,7,8-HpCDD	0.999 - 1.001
OCDF*	13C -OCDD	1.007 - 1.009
OCDD	13C -OCDD	1.000 - 1.001
13C -2,3,7,8-TCDF	13C -1,2,3,4-TCDD	0.995 - 0.997
13C -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	1.011 - 1.013
37Cl -2,3,7,8-TCDD	13C -1,2,3,4-TCDD	1.011 - 1.013
13C -1,2,3,7,8-PeCDF	13C -1,2,3,4-TCDD	1.073 - 1.075
13C -2,3,4,7,8-PeCDF	13C -1,2,3,4-TCDD	1.093 - 1.095
13C -1,2,3,7,8-PeCDD	13C -1,2,3,4-TCDD	1.099 - 1.101
13C -1,2,3,4,7,8-HxCDF*	13C -1,2,3,7,8,9-HxCDD	0.970 - 0.972
13C -1,2,3,6,7,8-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.973 - 0.975
13C -1,2,3,7,8,9-HxCDF	13C -1,2,3,7,8,9-HxCDD	1.008 - 1.010
13C -2,3,4,6,7,8,-HxCDF	13C -1,2,3,7,8,9-HxCDD	0.986 - 0.988
13C -1,2,3,4,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.990 - 0.992
13C -1,2,3,6,7,8-HxCDD	13C -1,2,3,7,8,9-HxCDD	0.993 - 0.994
13C -1,2,3,4,6,7,8-HpCDF	13C -1,2,3,7,8,9-HxCDD	1.050 - 1.051
13C -1,2,3,4,7,8,9-HpCDF	C -1,2,3,7,8,9-HxCDD	1.104 - 1.106
13C -1,2,3,4,6,7,8-HpCDD*	C -1,2,3,7,8,9-HxCDD	1.085 - 1.087
13C -OCDD	C -1,2,3,7,8,9-HxCDD	1.205 - 1.207

* Due to the use of the DB-5MS column, these compounds exhibit slightly different elution times, resulting in RRT limits which vary from the method.

FIGURE 1: 2378-TCDD CHROMATOGRAPHIC SEPARATION

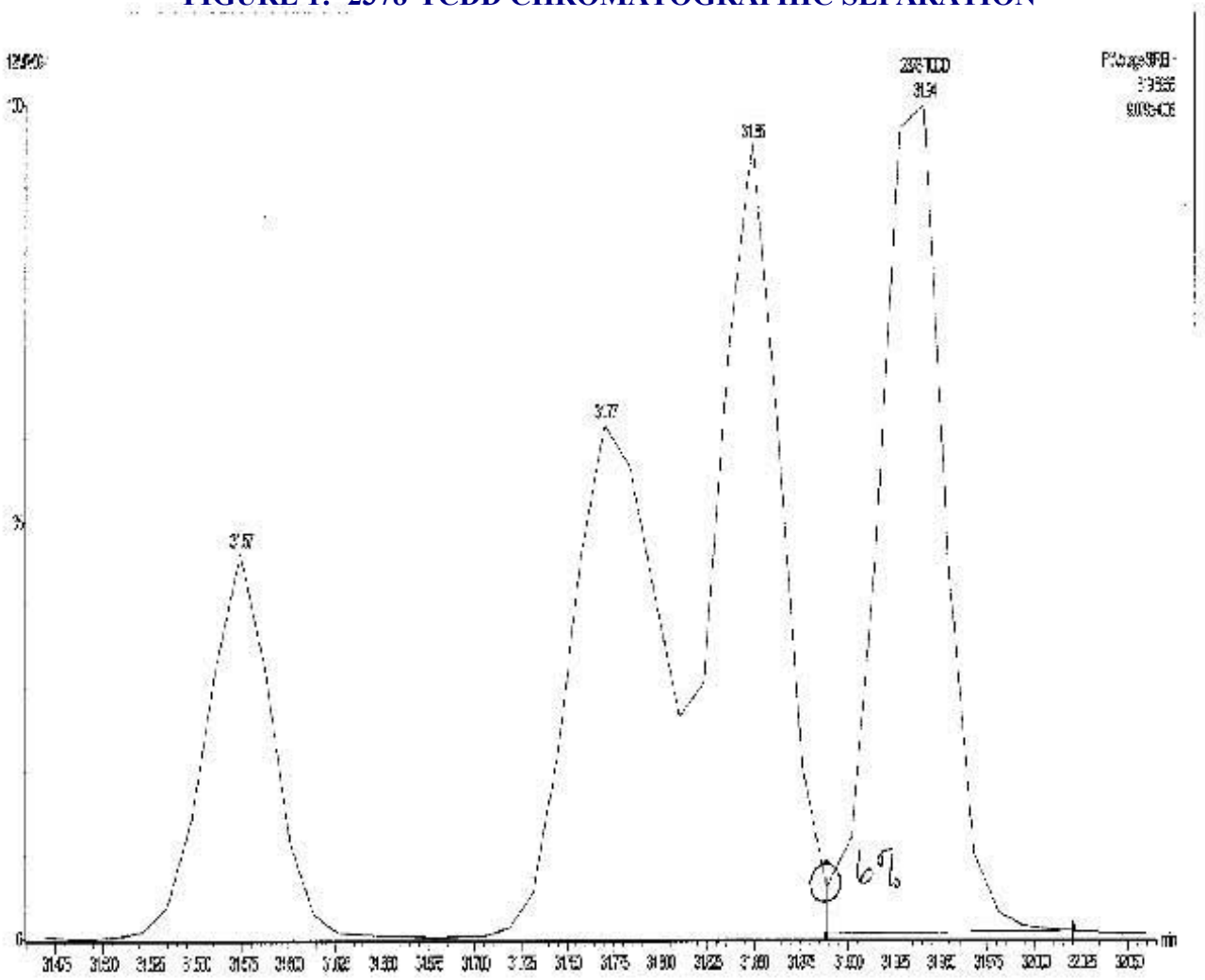
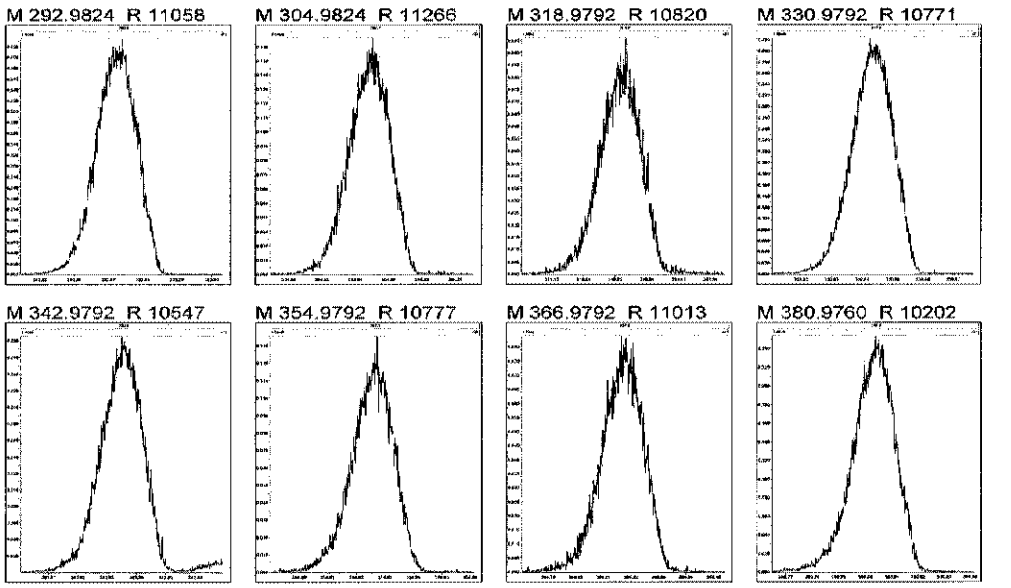


FIGURE 2: INSTRUMENT RESOLVING POWER (EXAMPLE)



VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

CAPE FEAR ANALYTICAL, LLC

QUALITY ASSURANCE PLAN

(CF-QS-B-001 REVISION 4)

PROPRIETARY INFORMATION

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Quality Assurance Plan Approval Signatures:

CFA

CFA's Document Control Officer certifies this document to be a true copy of the fully executed original.

W. M. Larkins

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SECTION 1
INTRODUCTION

Section 1 - Introduction

Cape Fear Analytical, LLC (CFA) is a privately owned environmental laboratory dedicated to providing personalized client services of the highest quality. Our mission is to employ innovative thinking and state-of-the-art analyses to solve analytical challenges. Well trained professional personnel and modern reporting tools will be an integral part of the businesses success. Implementation of a quality control and assurance program will provide a focus for productive work and the basis of state and regulating agency certifications.

CFA was established as an analytical testing laboratory in 2009. Our analytical laboratory uses state of the art equipment and methods to provide data of the highest quality and related support services to meet the needs of our clients.

This Quality Assurance Plan (QAP) provides an overview of our quality assurance program for analytical services. Outlined in this plan are the responsibilities, policies, and processes essential to maintaining client satisfaction and our high quality of performance. The Quality Manager is responsible for revising, controlling, and distributing the QAP. It is reviewed and updated at least annually.

Everyone on our staff is expected to understand the policies, objectives, and procedures that are described in this plan and to fully appreciate our commitment to quality and their respective roles and responsibilities with regard to quality. We also expect any analytical subcontractors we employ to perform in accordance with the quality assurance requirements delineated in this plan.

This Quality Assurance Plan has been prepared according to the standards and requirements of the US Environmental Protection Agency (EPA), ANSI/ISO/IEC 17025-2005, and the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards effective July 2003.

1.1 Quality Policy

CFA's quality policy is a demonstrated commitment to excellence in professional practice and to the quality of the data provided to its clients. The laboratory strives to provide high quality, personalized services that enable its clients to meet their analytical testing needs quickly and cost effectively. All CFA employees are required to be familiar with the quality management system and the necessity for documentation and implementation of its policies and procedures in meeting client and regulatory requirements. Continual improvement to effective, ethical implementation of all quality systems measured is expected throughout the laboratory operations.

We define quality as "consistently meeting the needs and exceeding the expectations of our clients in a highly ethical manner." As such, we strive to:

- meet or exceed client and regulatory requirements
- be technically correct and accurate
- be defensible within contract specifications
- provide services in a cost-effective, timely and efficient manner

At CFA, quality is emphasized at every level—from the owners and directors down to the newest of employees. Management's commitment to good professional practice and to the quality of our testing services for our customers is demonstrated by their dedication of personnel and resources to develop, implement, assess, and continually improve our technical and management operations.

The purpose of CFA's quality assurance program is to establish policies, procedures, and processes to meet or exceed the expectations of our clients. To achieve this, all personnel that support these services to our clients are introduced to the program and policies during their initial orientation, and annually thereafter during company-wide training sessions.

CFA's management is committed to compliance with and continual improvement of our quality assurance program. The program is designed to comply with the guidelines and specifications outlined in the following:

- NELAC 2003
- ANSI/ISO/IEC 17025-2005
- Department of Defense Quality Systems Manual
- Current U.S. EPA CLP statements of work for organic analyses

1.2 Quality Goals

CFA's primary goals are to:

- Ensure that all measurement data generated are scientifically and legally defensible, of known and acceptable quality per the data quality objectives (DQOs), and thoroughly documented to provide sound support for environmental decisions.
- Ensure compliance with all contractual requirements, environmental standards, and regulations established by local, state and federal authorities.

Additional goals include:

- A comprehensive quality assurance program to ensure the timely and effective completion of each measurement effort.
- A commitment to excellence and improvement at all levels of the organization.
- Early detection of deficiencies that might adversely affect data quality.
- Adequate document control.
- Effective quality assurance objectives for measurement systems and for quality data in terms of accuracy, precision, completeness, and comparability through the use of proven methods.
- The establishment of procedures that demonstrate that the analytical systems are in a state of statistical control.
- The implementation of corrective actions and improvements to ensure the integrity of data.
- Reduction of data entry errors through automated data handling procedures.
- The development and implementation of good laboratory practices and standard operating procedures (SOPs).
- Ability to customize quality assurance procedures to meet a client's specific requirements for data quality.
- Good control of instruments, services, and chemical procurement.
- A continuously capable laboratory information management system (AlphaLIMS).
- Validated and documented computer hardware and software.

1.3 Key Quality Elements

A sound quality assurance program is essential to our ability to provide data and services that consistently meet our high standards of integrity. The key features of our program are:

- A formal quality policy and QAP.
- Management review.
- Stated data quality objectives.
- A comprehensive employee training program.
- Ethics policy and education program.
- Internal audits and self-evaluations.
- A closed-loop corrective action program.
- State-of-the-art facilities and instruments.
- Adherence to standard operating procedures.
- EPA/NIST traceable reference materials.
- Electronically based document control.
- Chain of custody and electronic sample tracking.
- Inter-laboratory comparison programs.
- Formal laboratory accreditations.
- The evaluation of subcontractor laboratories.
- Statistical controls for analytical precision and accuracy.
- Replicate, method blank, matrix spike, internal standards, and surrogate measurements.
- The preventive maintenance of instrumentation and equipment.
- Independently prepared blind standard reference materials.
- Multi-level review processes.
- Focus on client satisfaction.
- Electronic tracking of client commitments, nonconformances and corrective actions.
- Trend analysis of nonconforming items.

1.4 Management Reviews

The effectiveness of the Quality System is reviewed at least annually by Senior Management. These reviews address issues that impact quality, and the results of the reviews are used to develop and implement improvements to the system. Records of the review meetings are maintained as quality documents.

1.5 Disposition of Client Records

In the event that the laboratory should change ownership, the responsibility for the maintenance and disposition of client records shall transfer to the new owners. In the unlikely event that the laboratory ceases to conduct business, clients shall be notified and asked to provide instructions as to how their records should be returned or disposed. If a client does not provide instructions, those records will be maintained and

disposed in a manner consistent with regulations and good laboratory practices for quality records.

1.6 Supporting Documents

Our laboratory operations and the quality of our analytical data comply with the specifications described in the documents listed in Appendix A.

1.7 Definitions

Applicable definitions are listed in Appendix B.

1.8 Test Methods

The following test methods are performed at Cape Fear Analytical:

- SW846 Method 8290A
- EPA Method 1613B
- EPA Method 1668B
- SW846 Method 8315A
- SW846 Method 8260B
- EPA DLM02.1
- EPA CBC01.1

SECTION 2
ORGANIZATION, MANAGEMENT, AND PERSONNEL

Section 2 - Organization, Management, and Personnel

The chart found in Appendix C depicts our organization, chain of command and flow of responsibility. The illustration in this appendix is designed to ensure the overall quality and cost efficiency of our company's analytical products and services. Our relationship to our parent corporation is shown by the dotted lines on the organizational chart in appendix C. We have the use of the parent corporation's VP of Operations to help with items such as laboratory direction, the evaluation of new work, and management review. CFA accesses the Quality Director of the parent corporation to provide help with annual quality systems review, allocation of additional quality resources to support heavy work loads in order to maintain quality, and vendor and subcontractor audit support. CFA depends upon its parent corporation for IT support, maintenance of AlphaLIMS and electronic deliverables.

Our structure is based on a customer-focused approach that follows a project from the point of initial contact to the final invoicing of work. This approach includes expertise in project management, sample receipt and custody, sample preparation and analysis, data review, and data packaging. CFA's Technical Director serves also as the laboratory Quality Manager. Our Quality Manager monitors the adherence of these departments to the Quality Assurance Program.

The general responsibilities associated with the following position levels are discussed in this section:

- Laboratory Director
- Technical Director
- Client Services Manager
- Production Manager
- Laboratory and Technical Staff

An overview of CFA's employee training protocol is also provided at Section 2.7.

2.1 Laboratory Director

The Laboratory Director is responsible for the daily operations of the laboratory and client services (see Section 2.4). He/she is also responsible for strategic planning, profitability and growth, personnel management and business development. Other responsibilities include the following:

- Ensuring that the individuals who staff our technical and quality positions have the necessary education, training, and experience to competently perform their jobs.
- Ensuring that all staff members receive ancillary training, as needed, to enhance performance in assigned positions.
- Budgeting, staffing, managing, and equipping the laboratory to meet current and future analytical program requirements.
- Monitoring and meeting profitability and growth objectives of the division.
- Establishing and implementing short and long range objectives and policies that support CFA's goals.
- Overseeing the implementation and overall effectiveness of our Quality Assurance Plan, health and safety initiatives, and environmental programs.
- Authorizes stop-work and the return-to-work orders after a major out-of-control situation
- Managing production and cost control activities.
- Supervising all personnel employed in the laboratory.
- Ensuring development of capabilities in response to new or revised regulations, instrumentation and procedures, and quality assurance initiatives.
- Ensuring compliance with regulatory standards, including ISO/IEC 17025 and the Department of Defense Quality Systems Manual.
- Representing the company to the public and to clients.
- Ensuring the appropriate delegation of authorities during periods of absence.

The Laboratory Director also manages the implementation and ongoing development of our Human Resources. Responsibilities include:

- Administration, orientation, and indoctrination of all new employees.

- Administration and compliance with Federal, State, and Local employment regulations.
- Sourcing candidates for all functional positions to maintain and strengthen the technical services provided by CFA.
- Management of occupational health and safety as it relates to Federal, State, and OSHA regulations.

2.2 Technical Director

The Technical Director is ultimately responsible for the technical content and quality of work performed within each department. Other responsibilities include:

- Defining the minimum level of qualification, experience, and skills necessary for positions in each department.
- Establishing and implementing policies and procedures that support our quality standards.
- Ensuring that technical laboratory staff demonstrates initial and continuing proficiency in the activities for which they are responsible.
- Documenting all analytical and operational activities of the laboratory.
- Ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system, properly labeled, and stored.
- Documenting the quality of all data reported by the laboratory.
- Developing internal mechanisms and measurements to improve efficiency.
- Overseeing activities designed to ensure compliance with laboratory health and safety requirements.
- Allocating the resources necessary to support an effective and ongoing quality assurance program.
- Ensuring compliance with regulatory standards, including ISO/IEC 17025 and the Department of Defense Quality Systems Manual.
- Maintaining MDL, PQL, LOD and LOQ studies in the laboratory LIMs

Due to high volume and the variety of analytical tests performed in the laboratory, the Technical Director has the assistance of a Production Manager (see Section 2.5).

The Technical Director also oversees our physical facility, laboratory safety program, and instrumentation. This includes the following responsibilities:

- Planning, evaluating, and making recommendations for facility maintenance.
- Implementation of the Chemical Hygiene program.
- Aiding in the Installation, maintenance, repair, and modification of analytical instrumentation.
- Providing technical expertise and training in instrumentation operation, calibration, and maintenance.
- Monitoring and ensuring regulatory compliance for waste management operations and off-site disposal.

The Technical Director at CFA also manages the implementation and maintenance of our quality systems in a timely, accurate, and consistent manner. These responsiblilties may be found in Section 3.

2.3 Quality Systems Review

The effectiveness of the Quality System is reviewed on a regular basis during management meetings, which are held as frequently as needed. These meetings address issues that impact quality, and the subsequent discussions are used to design and implement improvements to the system. At least annually, a management assessment of CFA's Quality System is conducted and reported. The Quality Manager maintains records of these assessments.

2.4 Client Services Manager

Project Managers (PMs) serve as primary liaisons to our clients. Employees function as PMs, under the guidance of the Client Services Manager, to manage the company's interaction with clients. They are the client's first point of contact and have responsibility for client satisfaction and for communicating project specifications and changes to the appropriate laboratory areas.

In addition to overseeing project management, other responsibilities include:

- Retaining clients and soliciting new work.
- Managing multiple sample delivery orders and preparing quotes.
- Working with clients to define analytical methodologies, quality assurance requirements, reports, deliverables, and pricing.

- Overseeing sample management and informing laboratory staff of the anticipated arrival of samples for analysis.
- Conducting a review of client documents (i.e. quotes, invoices, routine and specialized reports).
- Working with the accounting team on invoicing and collection issues.
- Working with the Laboratory Director and Production Manager to project workloads and determine schedules.

2.5 Production Manager

The Production Manager is a critical link between project management, lab personnel, and support staff. He/she reports to the Laboratory Director and has the following responsibilities:

- Planning and coordinating the operations of the laboratory to meet client expectations.
- Scheduling sample preparation and analyses according to holding times, quality criteria, and client due dates.
- Ensuring a multi-level review of 100% of data generated by the laboratory.
- Coordinating nonconformances and corrective actions in conjunction with the Quality Manager.
- Serving as a technical resource to the laboratory, including data review.
- Managing special projects, reviewing new work proposals, and overseeing the successful implementation of new methods.
- Monitoring and controlling expenses incurred by the laboratory such as overtime and consumables.
- Providing performance and career development feedback to laboratory staff.
- Ensuring compliance with regulatory standards, including ISO/IEC 17025 and the Department of Defense Quality Systems Manual.

2.6 Laboratory and Technical Staff - General Requirements

At CFA, every effort is made to ensure that the laboratory is sufficiently staffed with personnel who have the training, education, and skills to perform their assigned jobs competently.

Depending upon the specific position, laboratory personnel are responsible for:

- Complying with quality assurance and quality control requirements that pertain to their group and/or technical function.
- Demonstrating a specific knowledge of their particular function and a general knowledge of laboratory operations.
- Understanding analytical test methods and standard operating procedures that are applicable to their job function.
- Documenting their activities and sample interactions in accordance with analytical methods and standard operating procedures.
- Implementing the quality assurance program as it pertains to their respective job functions.
- Identifying potential sources of error and reporting any observed substandard conditions or practices.
- Identifying and correcting any problems affecting the quality of analytical data.

2.7 Employee Training

To ensure that our clients receive the highest quality services possible, we train our employees in the general policies and practices of the company, as well as the specific operating procedures relative to their positions. We conduct and document this training according to CF-HR-E-002 for Employee Training and CF-QS-E-017 for Maintaining Technical Training Records.

New employees participate in a company orientation shortly after they are hired. During orientation they receive information on quality systems, ethics/data integrity, laboratory safety, and employment practices. Each new employee is also provided a manual that reiterates our policies on equal opportunity, benefits, leave, conflicts of interest, employee performance, and disciplinary action. Employees can access standard operating procedures, the Quality Assurance Plan, Safety, Health, and Chemical Hygiene Plan, and the Laboratory Waste Management Plan on CFA's Intranet.

Other training provided on an ongoing basis may include:

- Demonstration of initial proficiency in analytical methods and training to SOPs conducted by a trainer who has been documented as qualified

and proficient in the process for which training is being provided.

- Demonstration of continued analyst proficiency is updated annually, usually during the first quarter of each year. Proficiency is demonstrated using the same processes as those used for initial Demonstration of Capability. (Refer to Section 8.3.1.)
- Company-wide, onsite training.
- Courses or workshops on specific equipment and analytical techniques.
- University courses.
- Professional and trade association conferences, seminars, and courses.

Documentation of employee training is the joint responsibility of the employee and the Quality Manager or Production Manager. If an SOP is revised during the course of the year, training to the revised SOP must be documented.

2.8 Ethics and Data Integrity

Employees attend ethics education programs that focus on the high standards of data integrity and ethical behavior mandated by our company and expected by our clients.

The annual ethics training includes:

- Specific examples of unethical behaviors for the industry and for the laboratory.
- Explanation of Internal Auditing for unethical behaviors and practices.
- CFA use of electronic audit functions using instrument and AlphaLIMS software.
- Examples of consequences of inappropriate or unethical behaviors/practices.

All employees sign an Ethics and Data Integrity Agreement that reflects their commitment to always performing their duties with these high standards. (Refer to Appendix F.)

2.9 Confidentiality

The laboratory maintains the confidentiality and proprietary rights of information including the type of work performed and results of analysis. Laboratory personnel and staff are informed of this policy and sign a confidentiality agreement.

A confidentiality statement accompanies the electronic transfer of data from CFA via telefacsimile (fax) or electronic mail systems (email). Government affiliated auditing agencies have access to pertinent laboratory records. However, contract, third party, and client auditors have access only to those records that may be applicable to their inspection and shall not be granted access to client records that may be considered in conflict with their interests, unless prior authorization has been given by the submitting client. Confidential information may be purged of references to client identity, project and/or sample identity by the laboratoryso that records may be provided to other entities (e.g. auditors) for review.

SECTION 3
QUALITY SYSTEMS

Section 3 - Quality Systems

Our Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures necessary to plan, implement, and assess the work we perform. CFA's QA Program establishes a quality management system (QMS) that governs all of the activities of our organization.

CFA's quality management system is designed to conform to the requirements specified in the standards referenced in Appendix A. Essential elements of our quality management system are described in this section.

3.1 Quality Manager

The Quality Manager is responsible for managing CFA's Quality Assurance Program. This person functions independently of the systems he/she monitors. For the purposes of this document, CFA's Technical Director assumes the role of the Quality Manager.

Following is a summary of the responsibilities of this position:

- Reports to the Laboratory Director
- Demonstrates strict adherence to and support of the company ethics policy
- Serves as management's representative for quality
- Responsible for the implementation and maintenance of the QMS
- Responsible for ensuring communication takes place at all levels within CFA, pertaining to issues included in this section.
- Responsible for evaluating the effectiveness of the quality system through various quality measures such as internal and external assessment, non-conformances and management review
- Initiates and recommends preventive action and solutions to quality problems
- Implements appropriate action to control quality problems until solutions are implemented and verified to be effective
- Verifies that effective solutions are implemented
- Authorizes stop-work and return-to-work orders after a major out-of-control situation
- Demonstrates knowledge of the Quality System as defined by NELAC, ANSI/ISO/IEC 17025 and DoD.
- Plans, schedules and participates in CFA's client audits, internal audits, and subcontractor audits
- Conducts conformance audits as necessary to verify implementation and closure of audit action items
- Serves as liaison to client and third party auditors
- Coordinates laboratory responses to audit reports and prepares final response
- Monitors progress of corrective actions
- Demonstrates the ability to evaluate data objectively without outside influence
- Has knowledge of analytical methods
- Ensures the monitoring of balances and weights, and temperature regulation of ovens, water baths, and refrigerators
- Coordinates the monitoring of DI water system and volatile organics storage coolers
- Writes and reviews quality documents and standard operating procedures
- Provides training in quality systems, ethics, confidentiality and good laboratory practices.
- Evaluates effectiveness of training and makes recommendations when additional training or re-training is required
- Manages laboratory certification and licensing processes
- Coordinates the receipt and disposition of external and internal performance evaluation samples.
- Developing and evaluating quality assurance policies and procedures pertinent to our laboratory functions, and communicating these with the division directors and managers.
- Ensuring that laboratory activities are in compliance with local, state, and federal environmental laws and regulations.

- Manage performance, evaluation and implementation of MDL, PQL and SPC determinations.
- Reviewing project-specific quality assurance plans.
- Performing independent quality reviews of randomly selected data reports and laboratory practices including facitilies and instruments.
- Communicating system deficiencies, recommending corrective action to improve the system, and defining the validity of data generated during out of control situations.
- Administering our document control system.
- Designating quality systems authorities in times of absence to one or more appropriately knowledgeable individuals.

3.2 Quality Documents

Our Quality Systems policies and procedures are documented in the QA Plan (CF-QS-B-001) and other supporting documents. CFA's management approves all company quality documents. Pre-approval is secured for any departures from such documents that may affect quality. Any departures are documented and provided to the client.

In addition to the QA Plan, Quality Systems allows for QA Project Plans (QAPjP) and includes standard operating procedures and any other quality assurance program requirements defined by individual contracts. The QA Plan describes the quality standards that we apply to our laboratory operations. We use Quality Assurance Project Plans to specify individual project requirements. The QA Plan and supporting documents are verified to be understood and are implemented throughout the laboratory fractions to which they apply.

Finally, our Standard Operating Procedures (SOPs) are used to describe in detail those activities that affect quality. SOPs are prepared, authorized, changed, revised, released, and retired in accordance with CF-ADM-E-001. SOPs are accessible electronically via CFA's Intranet.

3.3 Document Control

The control of quality documents is critical to the effective implementation of our Quality Systems. We define and control this process in accordance with CF-DC-E-001 for Document Control. Responsibilities for document control are divided between the Production Manager and the Quality Manager.

The Production Manager is responsible for:

- Supporting the development and maintenance of controlled documents that apply to their respective departments.
- Ensuring documentation that the affected employees are aware of revisions to documents or manuals.

The Quality Manager is responsible for:

- Electronic maintenance of all records required for control, re-creation, and maintenance of analytical documentation.
- Maintenance of electronic copies of archived data and the electronic log of how they were determined.
- Demonstrating strict adherence to and support of the company ethics policy as it pertains to Document Control.
- Managing the system for the preparation, authorization, change, revision, release, and retirement of the Quality Manual, QAP, project plans, and standard operating procedures.
- Ensuring that current controlled documents are accessible via CFA's Intranet.
- Managing a system to document current revision numbers and revision dates for all distributed documents and manuals.
- Managing a system to identify the nature of document revisions.

Maintaining hard or electronic copies of obsolete documents and original copies of controlled documents.

3.4 Controlled Document Review

Internally generated controlled documents undergo a multi-level review and approval process before they are issued. These levels include a procedural review, technical and/or quality review and final authorization by the appropriate manager or director. To ensure that new or revised standard operating procedures are not implemented prematurely, SOPs are effective upon the date of the final approval signature.

3.5 Review of New Work

Prior to submitting proposals for new work, all available information, including but not limited to, statements of work (SOW), requests for proposal (RFP) and client communications are reviewed by the project manager, Quality Manager, and if necessary, the Laboratory Director. This review is performed in order to

ascertain the appropriateness of CFA's resources and facilities.

3.6 Quality Records

Quality records provide evidence that specified quality requirements have been met and documented. We generate them in accordance with applicable procedures, programs, and contracts. Quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- Audit records
- Run logs, instrument data, and analytical logbooks
- Instrument, equipment, and building maintenance logs
- Material requisition forms
- Monitoring logs
- Nonconformance reports and corrective actions
- Method development and start-up procedures including method detection limit studies
- Technical training records
- Waste management records
- Standard logs
- Software validation documentation
- Standard Operating Procedures (SOPs)
- Sample collection and field data

Our quality records are:

- Documented in a legible manner.
- Indexed and filed in a manner conducive to ready retrieval.
- Stored in a manner that protects them from loss, damage, and unauthorized alterations.
- Retained and disposed in the identified time period.

The generation, validation, indexing, storage, retrieval, and disposition of our quality records are detailed in CF-QS-E-008 for Quality Records Management and Disposition. The quality records of subcontracted services are also required to meet the conditions established in this SOP.

3.7 Internal and Supplier Quality Audits

We conduct internal audits annually to verify that our operations comply with the requirements of our QA program and those of our clients. We perform supplier audits as necessary to ensure that they too meet the requirements of these programs. Both internal and supplier audits are conducted in accordance with CF-QS-E-001 for the Conduct of Quality Audits.

3.7.1 Audit Frequency

Internal audits are conducted at least annually in accordance with a schedule approved by the Quality Manager. Supplier audits are contingent upon the categorization of the supplier, and may or may not be conducted prior to the use of a supplier or subcontractor (Refer to CF-QS-E-001.) Type I suppliers and subcontractors, regardless of how they were initially qualified, are re-evaluated at least once every three years.

Additional internal and supplier audits may be scheduled if deemed necessary.

3.7.2 Audit Team Responsibilities

Internal and supplier audits are conducted by qualified staff under the direction of the Quality Manager. A qualified audit team member shall have the technical expertise to examine the assigned activities.

We do not allow staff to audit activities for which they are responsible or in which they are directly involved. It is the responsibility of the Quality Manager to ensure that such conflicts of interest are avoided when the audit team is assembled.

Management has a significant role in the internal audit process, including:

- Provision of audit personnel
- Empowerment of the audit team with authority to make the audit effective
- Development and implementation of timely corrective action plans

3.7.3 Identification and verification of OFIs

Opportunities for Improvement are identified conditions that may adversely affect the quality of products or services. Several examples of objective evidence are used to support an OFI, which might be classified as a finding, concern, observation, and/or recommendation.

The Quality Manager may initiate a Nonconformance Report (NCR) or Corrective Action Request and Report (CARR) referencing the OFI. The NCR or CARR is then entered into the NCR system per CF-QS-E-012 for NCR Database Operation.

Implementation of a corrective action is later verified by a re-audit of the deficient area, review of new or revised documents, or, if the OFI does not warrant immediate action, the corrective action may be verified during the next scheduled audit.

3.8 Managerial and Audit Review

Laboratory management reviews the audit process at least annually. This ensures the effectiveness of the corrective action plan and provides the opportunity to introduce changes and improvements.

We document all review findings and corrective actions. Implementation plans and schedules are monitored by the Quality Manager.

3.9 Nonconformances

Processes, materials, and services that do not meet specifications or requirements are defined as nonconforming. Such nonconformances can include items developed in-house or purchased from vendors, samples received from clients, work in progress, and client reports.

At CFA, we have a nonconformance reporting system (NCR) that helps us prevent the entry of defective goods and services into our processes and the release of nonconforming goods and services to our clients. Our NCR system provides a means for documenting the disposition of nonconforming items and for communicating these to the persons involved in the process affected by the adverse condition(s).

Nonconformances are documented according to CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items. We regularly review SOPs, client complaints, and quality records, including completed NCRs, to promptly identify conditions that might result in situations or services that do not conform to specified quality requirements.

Our Quality Manager processes, categorizes and trends nonconformances. Trending information may be provided to management.

3.10 Corrective Action

There are two categories of corrective action at CFA. One is corrective action implemented at the analytical and data review level in accordance with the analytical SOP. The other is formal corrective action documented by the Quality Manager in accordance with CF-QS-E-002. Formal corrective action is initiated when a nonconformance reoccurs or is so significant that permanent elimination or prevention of the problem is required.

We include quality requirements in most analytical SOPs to ensure that data are reported only if the quality control criteria are met or the quality control measures that did not meet the acceptance criteria are documented.

Formal corrective action is implemented according to CF-QS-E-002 for Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement and documented according to CF-QS-E-012 for NCR Database Operation.

Any employee at CFA can identify and report a nonconformance and request that corrective action be taken. Any CFA employee can participate on a corrective action team as requested by the Quality Manager or Production Manager. The steps for conducting corrective action are detailed in CF-QS-E-002.

In the event that correctness or validity of the laboratory's test results is doubted, the laboratory will take corrective action. If investigations show that the results have been impacted, affected clients will be informed of the issue in writing within 21 calendar days of the discovery.

3.11 Performance Audits

In addition to internal and client audits, our laboratory participates in annual performance evaluation studies conducted by independent providers. We routinely participate in the following types of performance audits:

- Proficiency testing and other inter-laboratory comparisons.
- Performance requirements necessary to retain certifications (Appendix D).
- Evaluation of recoveries of certified reference and in-house secondary reference materials using statistical process control (SPC) data.

- Evaluation of relative percent difference between measurements through SPC data.

NOTE: Once PE samples have been prepared in accordance with the instructions provided by the PE vendor, they are managed and analyzed in the same manner as environmental samples from clients. The analytical and reporting processes for PE samples are not handled differently than for authentic samples. SOP CF-QS-E-013 more completely describes the laboratory policies and practices for managing proficiency evaluation samples.

We also participate in a number of proficiency testing programs for federal and state agencies and as required by contracts. It is our policy that no proficiency evaluation samples be analyzed in any special manner.

Our annual performance evaluation participation generally includes a combination of studies that support the following:

- Water Pollution (WP). Biannual program for waste methodologies.
- Water Supply (WS): Biannual program for drinking water methodologies.
- MAPEP (Mixed Analyte Performance Evaluation Program) administered by the Department of Energy.

At CFA, we also evaluate our analytical performance on a regular basis through statistical process control acceptance criteria. Where feasible, this criterion is applied to both measures of precision and accuracy and is specific to sample matrix.

We establish environmental process control limits for the LCS, MS and MSD at least annually. CFA's current process control limits are maintained in LIMS. The upper and lower control limits (UCL and LCL respectively) for precision are plus or minus three times the standard deviation from the mean of a series of relative percent differences. For DoD QSM projects the laboratory initially uses the limits provided in the current DoD QSM tables and when unavailable, uses the laboratory derived limits. When the laboratory has enough data points to establish statistical limits, it will derive these limits in accordance with its normal procedures, using DoD QSM project sample results.

CFA's procedure for establishing precision and bias at the LOQ is to collect data from LOQ and LOQV determinations until there are enough points to generate valid statistical limits. These limits are reported with the LOQ results in data packages. Where there is not adequate laboratory data available, the laboratory uses 50 -150% for UCL and LCL, with 45% - 155% ME limits.

CFA also measures precision through the use of matrix duplicates and/or matrix spike duplicates. The upper and lower control limits (UCL and LCL respectively) for precision are the same as those used for the LCS, in accordance with the DoD QSM.

For non-DoD QSM projects,the upper and lower control limits (UCL and LCL respectively) for precision are plus or minus three times the standard deviation from the mean of a series of relative percent differences.

Accuracy is measured through laboratory control samples and/or matrix spikes, as well as surrogates and internal standards. The UCLs and LCLs for accuracy are plus or minus three times the standard deviation from the mean of a series of recoveries. Specific Instructions for out-of-control situations are provided in the applicable analytical SOP.

3.12 Essential Quality Control Measures

Some quality control measures are method-specific. There are, however, general quality control measures that are essential to our quality system. These quality measures include:

- Monitoring of negative and positive controls
- Defining variability and reproducibility through duplicates
- Ensuring the accuracy of test data including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, etc.
- Evaluating test performance using method detection limits and quantitation limits or range of applicability such as linearity
- Selecting the appropriate method of data reduction
- A copy of CFA's Ethics and Integrity Agreement is provided in Appendix F.

SECTION 4

FACILITIES

Section 4 - Facilities

Our laboratory is designed with a full-service approach to handling environmental needs. The layout provides dedicated space for organic extractions, semi-volatile organic analyses, volatile organic analyses and dioxin/PCB analyses.

The laboratory and support offices occupy approximately 5,400 square feet engineered to meet the stringent quality control and utility requirements of the modern environmental laboratory. Records are temporarily stored on-site then warehoused in a secured building off-site. The diagram in Appendix H depicts the layout of the laboratories.

Discussed in this section are:

- Facility security
- Utility services and deionized water
- Prevention of contamination
- Assessment of contamination

4.1 Facility Security

Our facility features secured laboratory and storage areas. Restricted entry assures sample integrity and client confidentiality, which satisfies clients' and potential national security interests.

Visitors cannot gain entry without being escorted through the laboratory by authorized personnel. A designated sample custodian and a bar-coded chain-of-custody provide a second level of security.

4.2 Utility Services

The laboratory area is equipped with the following utilities:

- Cold water
- Hot water
- Deionized water
- Compressed air
- Vacuum (at selected stations)
- 110 Volt AC
- 208 Volt AC (at selected stations)
- Specialty gases (as required)

4.2.1 Deionized Water

We have one fully functioning deionized water system which services the whole laboratory. DI water is made from city water flowing through a deionization system capable of producing 5 gallons per minute of Type II laboratory water.

We monitor the unit by using the service/replacement indicator light on the filter assemblies. Because the laboratory requires water of purity such that scaling of glassware and analytes of interest are not detected, locally purchased distilled water may also be used.

4.3 Prevention of Contamination

Work areas that are free of sample contaminants, constituents and measurement interferences are important to the generation of quality data. With this in mind, we designed our laboratory to prevent contamination and reinforce this design with good laboratory practices.

In addition to keeping our work areas free of dust and dirt accumulations, policies and features that prevent or minimize contamination include:

- Segregation of volatile and semi-volatile laboratories to minimize potential contamination associated with the use of commonly required solvents.
- Fume hoods to remove fumes and reduce the risk of aerosol and airborne contaminants and personal safety hazards are monitored in accordance with CF-FC-E-003 for Fume Hood Face Velocity Performance Checks.
- Restricted access to the laboratory (authorized personnel only).
- Designated area for glassware preparation wherein all glassware used in sample prep and analysis is cleaned according to CF-LB-E-003 for Glassware Preparation.
- Segregated storage areas for volatiles samples.
- Production, use, and monitoring of Type II DI water.
- Tracking and trending of any significant sample and/or reagent spills using the AlphaLIMS NCR system, allowing efficient analysis of any potential contamination.

4.4 Assessment of Contamination Levels

We evaluate contamination resulting from the following sources on the basis of quality assurance and quality control data derived from the analytical method and method blanks:

- Sample containers
- Reagent water
- Reagents and solvents
- Sample storage
- Chemical and physical interference
- Constituent carryover during analysis

Contamination in each of the volatile storage coolers is monitored by the weekly analysis of water blanks during periods of sample storage. Four DI water blanks are placed in the cooler/freezer at the beginning of each month with one being analyzed each week. If the concentration of any target analyte exceeds the PQL, corrective action is implemented to eliminate the source of contamination, evaluate the effect of samples stored in the cooler/freezer, and to notify clients, if necessary.

SECTION 5

EQUIPMENT AND REFERENCE MATERIALS

Section 5 - Equipment and Reference Materials

CFA's ability to efficiently generate data that are reproducible, accurate, and legally defensible is attributable to our use of high-quality instruments, equipment, and reference materials.

Provided in this section are:

- CFA's policies governing instruments, equipment, and reference materials
- Identification of instrumentation and support equipment
- Procurement protocol

5.1 General Policies

It is our policy to purchase instrumentation, equipment and high-quality reference materials that meet or exceed the method and regulatory requirements for the analyses for which we are accredited. If we need to use instruments or equipment not under our permanent control, we ensure that it also meets these standards.

Instrumentation and equipment are placed into service on the basis of ability to meet method or regulatory specified operating conditions such as range and accuracy. All laboratory instrumentation and testing equipment is maintained in accordance with standard operating procedures (SOPs).

Instrumentation and equipment is used in a manner that assures, where possible, that measurement uncertainty is known and consistent with specified quality requirements. Instruments and equipment are taken out of service and segregated or labeled as such under the following conditions:

- Mishandling and/or overloading
- Results produced are suspect
- Demonstrated defect or malfunction

Tagged or segregated instruments and equipment remain out of service until repaired and documented by test, calibration, or verification to perform satisfactorily. Instruments that are in service and normally calibrated prior to and during use are not tagged.

Each item of equipment, including reference materials is, if appropriate, labeled, marked or otherwise identified to indicate its calibration status. We maintain records for each major item of equipment, instrumentation, and all reference materials significant to quality performance. These records are often in the form of maintenance logs, which are kept in accordance with CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

Documentation included in these records includes but is not limited to:

- Equipment name
- Manufacturer's name
- Type identification
- Serial number or other unique identification
- Date received and date placed in service (if available)
- Current location
- Condition when received (if known)
- Manufacturer's instruction, where available
- Dates and results of calibrations and or verifications
- Date of next calibration and/or verification, where written procedures do not specify frequency
- Details of maintenance carried out to date and planned for the future
- History of any damage, malfunction, modification or repair

5.2 Instrumentation and Support Equipment

Appendix G lists the instruments we use for the analysis of environmental samples. Where feasible, our instruments are equipped with autosamplers that improve efficiency and facilitate consistent sample introduction to the sample detector. They are also connected to an area network to facilitate data transfer.

Devices that may not be the actual test instrument but are necessary to support laboratory operations are referred to as support equipment. We also maintain this equipment in proper working order. Support equipment utilized at CFA includes:

- balances
- ovens
- refrigerators
- freezers
- water baths

- temperature measuring devices
- volumetric dispensing devices
- muffle furnaces
- grinders and homogenizers
- hot plates and heating mantles.

Guidelines for the required calibration and evaluation of this equipment are discussed in Section 7.

SOPs are used to describe our procedures for all routine analyses performed by our labs. These procedures include step-by-step instructions for sample collection, storage, preparation, analysis, instrument calibration, quality control, disposal, and data reporting.

5.3 Control of Purchased Items

Materials, equipment, and services that affect the quality of our products are designated as Quality Materials, Equipment, and Services and are purchased from pre-approved suppliers whenever possible. Suppliers are approved and documented according to CF-QS-E-001 for the Conduct of Quality Audits.

At CFA, we maintain documentation of specific quality requirements for materials and services. Records that document the quality of a product or service may include:

- certificates of analysis and traceability
- verifications of chemical quality
- inspections of equipment or materials
- verifications or inspections of vendor product specifications

SECTION 6

HEALTH AND SAFETY

Section 6 - Health and Safety

CFA maintains a safe work environment and promotes healthy work practices. CF-LB-N-001 is the laboratory Safety, Health and Chemical Hygiene Plan. Procedures outlined in the plan are consistent with OSHA, CERCLA and the EPA.

All employees are trained in the safety practices applicable to their job functions. This training is conducted in accordance with CF-HR-E-002 for Employee Training.

6.1 Fire Safety

Our facility is equipped with a fire alarm system designed to detect smoke in all areas of the facility. Certain high-risk areas, such as, the cold and ambient storage areas, organic sample preparation lab, and solvent storage are additionally equipped with automatic halon systems. Fire blankets and dry chemical extinguishers are located at strategic points throughout the lab. We routinely inspect these extinguishers in accordance with CF-FC-E-004. Lab personnel are trained in the proper use and selection of fire extinguishers. In order to decrease the risk of fire, bulk solvents are stored in a protected outdoor corral.

6.2 Evacuation

In the unlikely event of a fire (or other emergency), we have defined evacuation routes depicted in Appendix H. This diagram is posted in pertinent areas of the facility.

6.3 Safety Equipment

Safety equipment, including safety glasses, lab coats, safety goggles and protective gloves, is available to all employees as needed. Eyewashes and overhead showers are located in the laboratory. We routinely inspect these as directed in CF-FC-E-002 for Testing Emergency Eyewash and Shower Equipment.

SECTION 7

MEASUREMENT, TRACEABILITY, AND CALIBRATION

Section 7 - Traceability and Calibration

Traceability of measurements and the calibration of testing equipment are imperative to our ability to produce accurate and legally defensible data. As such, we have implemented procedures to ensure that equipment calibration and measurement verification are traceable to nationally recognized standards.

Where possible, calibration certificates provide traceability to national standards of measurement. Calibration certificates provide measurement results and any associated uncertainty of measurement, and/or a statement of compliance with the identified specification. Calibration certifications are maintained as quality records.

When traceability to a national standard is not applicable, verification of measurement is achieved through inter-laboratory comparisons, proficiency tests, or independent analyses.

The following measurement and traceability practices are described in this section:

- Calibration criteria for support equipment
- General requirements
- Balances
- Temperature-sensitive devices and temperature monitoring
- Air displacement pipets
- Instrument calibration
- Calibration verification

7.1 Calibration Criteria for Support Equipment

This section addresses calibration protocols for support equipment, including balances, temperature - sensitive equipment, and air displacement pipets. The general criteria applicable to the calibration of support equipment are as follows:

- Equipment is maintained in proper working order. Records of all maintenance activities including service calls are kept.
- Calibrations or verifications over the entire range of use, using NIST-traceable references when available, are conducted at least annually.
- Temperature measuring devices with digital readouts are calibrated quarterly.
- Prior to use each day, balances, ovens, freezers, refrigerators, incubators, and water baths are checked with NIST-traceable references (where possible) in the expected use range.
- If prescribed by the test method, additional monitoring is performed for a device used in a critical test (such as an incubator or water bath).
- Reference standards of measurement such as Class S or equivalent weights or traceable thermometers may be used for calibration when demonstrated that their performance as reference standards will not be invalidated.
- Reference standards of measurement are calibrated by a body that can provide, where possible, traceability to a national standard.
- Reference standards and measuring and testing equipment are subject to in-service checks between calibrations and verifications, in accordance with ANSI/ISO/IEC 17025-2005.
- Mechanical volumetric dispensing devices used for quantitative measurements are checked daily for accuracy prior to use.
- If results of calibration and verification are not within the specifications for the equipment's application, then:
    1. The equipment is removed from service until repaired
    2. Under certain conditions, a deviation curve may be prepared. All measurements are corrected for the deviation, recorded and maintained.

7.1.1 Balances

Our balances undergo annual calibration, maintenance, and cleaning. Each balance is labeled with a serial number, service date, date of next service, and signature of the service technician.

Balances are set up, calibrated, and operated in the range required by the analytical method in accordance with CF-LB-E-002 for Balances. Prior to using a balance, the analyst is responsible for checking its calibration.

Calibration and calibration verification are performed using weights that are or have been calibrated against Class S or equivalent weights. These weights are

traceable to NIST and calibrated annually by a calibration service provider that meets the requirements of the ANSI/ISO/IEC 17025-2005 standard.

Calibration and calibration verification are recorded in the balance calibration logbook. If the calibration or calibration verification does not meet the specified acceptance criteria, the balance is recalibrated. If the calibration criteria are still not met, the balance is removed from service and tagged as such.

7.1.2 Refrigerators, Freezers, Ovens, Water Baths, and Similar Devices

Careful control of temperature is often central to the production of acceptable data. Temperature excursions beyond the established limits may invalidate a procedure and the associated data. Constant monitoring in accordance with CF-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, and Other Similar Devices assures us that regulatory and/or method temperature requirements are being met.

We measure temperatures with thermometers that are verified annually against a NIST-traceable thermometer. The NIST traceable thermometers are independently verified at least annually by a verification service that meets the requirements of the ANSI/ISO/IEC 17025-2005 standard. The protocol for thermometer verification is described in CF-QS-E-007. We monitor the temperature of the following equipment according to CF-LB-E-004:

- Refrigerators and freezers used to store samples, standards, and other temperature-sensitive materials
- Ovens
- Water baths

We monitor the temperatures of refrigerators and freezers prior to use each day. The temperatures of ovens, water baths, and other devices used as part of an analytical process must be monitored prior to, during, and immediately after use.

Temperature measurements are documented on logs specific to each piece of equipment. The logs are posted on or near each refrigerator, freezer, water bath, oven, or other temperature control device. Each log includes the following information:

- Date and time of each measurement
- Initials of person taking measurement
- Acceptance limits for device being monitored
- Whether device conforms with specifications at time of measurement
- Name, location, and number of device being monitored
- Notation of any out-of-control condition

When the process to maintain and document temperatures within acceptance limits does not conform to specifications, a nonconformance report (NCR) is issued. Appropriate action is then taken to disposition the nonconformance according to CF-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

Examples of nonconformances are:

- Failure to maintain process temperature within acceptance limits
- Failure of device to achieve calibration
- Total failure of temperature control device
- Failure to monitor the temperature as required

7.1.3 Air Displacement Pipets

Air displacement pipets offer a level of precision and accuracy exceeded only by Class A transfer pipets. Due to the use of disposable tips, these pipets eliminate the possibility of cross-contamination.

We calibrate air displacement pipets quarterly using five replicate measurements of a frequently used volume setting in accordance with CF-LB-E-010 for Maintenance and Use of Air Displacement Pipets.

The acceptance criteria for each measurement are based on the average of the five calibration measurements. Tolerance limits for commonly used verification volumes and accuracy and precision checks are included in the pipet calibration logbook. Calibrations are traceable to each pipet using the unique identification found on its label.

If a pipet does not meet the calibration tolerance limits, it is removed from service until it again demonstrates compliance after being cleaned and/or repaired. Analysts whose jobs may require the use of air displacement pipets are trained in their proper use and calibration.

7.2 Instrument Calibrations

To ensure that the data generated by an instrument are accurate, we calibrate the instrument using standards containing known concentrations of target analytes. The stability of the instrument over the calibration range is

verified by the analysis of a continuing calibration verification standard (CCV) obtained from a second source whenever possible.

Traceability of calibration, calibration verification, and other quality control standards to the recognized standard are documented per CF-LB-E-007 for Laboratory Standards Documentation. Individual identification numbers are assigned to each source standard and each subsequent intermediate and working standard prepared.

The identification number makes it possible to trace a standard to a parent standard and ultimately to the source standard and its Certificate of Analysis. The date each standard is prepared, the protocol used in the preparation, the person preparing the standard, and the standard's expiration date are documented in the appropriate standards log, maintained in AlphaLIMS. The information is accessible via the standard ID number.

We record standard and reagent ID numbers on instrument run logs, analytical logbooks, sample preparation logs, and instrument raw data. Calibration standards that are used in the analysis of a particular sample or group of samples can be traced to NIST, US EPA, or other nationally recognized standards.

7.2.1 Initial Instrument Calibration

Calibration procedures for specific instruments, and the frequencies of performance for defined methods, are described in the applicable operating or analytical SOP. Calibration is discussed in general terms in CF-QS-E-014 and includes standard laboratory practices and formulas used for determinations made by these practices. General guidelines include:

- Preparation of calibration curves as specified in the reference method.
- If a test method does not specify the number of calibration standards, the minimum number is two, not including blanks, with one at the lowest quantitation limit. The reference SOP must establish the initial calibration requirements.
- Verification of initial calibrations with a standard obtained from a second source (unless one is not available).

7.3 Calibration Verification

When an initial calibration curve is not established on the day of analysis, the integrity of the curve should be verified prior to sample analysis. Verification requires the analysis of a standard from a second source. The standard concentration should be at the method-defined level. If not specified, a standard at a mid-level concentration may be used.

If the calibration verification does not meet acceptance criteria, the analytical procedure is stopped and evaluated, and appropriate corrective measures are taken. Calibration verification (CCV) must be acceptable before any samples are analyzed. CCVs are analyzed at a frequency described in the referenced method. If an instrument consistently drifts outside the acceptance criteria before the next calibration, the frequency is increased.

If the recovery of a CCV does not meet the acceptance criteria and routine corrective actions fail to produce a second consecutive check within acceptance criteria, a new initial calibration curve should be constructed. Analytes of interest found in corresponding environmental samples may be reported, however, only if all of these criteria are met:

1. CCV recovery for target analyte exceeds the acceptance criteria (biased high)
2. Target analyte in the environmental sample is not detected at a concentration exceeding the level required by client contract (i.e., MDL, PQL).

Non-detects that meet these criteria are also referred to as "passable non-detects."

If the CCV recovery does not meet the acceptance criteria, and samples are found to contain target analytes at concentrations above the PQL, the affected samples must be re-analyzed.

SECTION 8

ANALYTICAL METHODS AND STANDARD OPERATING PROCEDURES

Section 8 - Analytical Methods and Standard Operating Procedures (SOPs)

We provide a wide array of parameters including dioxins/furans and PCB congeners, volatile organics and extractable organics. The procedures we use to determine these parameters are consistently executed due to our extensive system of SOPs and our training requirements for analytical staff.

A list of our SOPs and the analytical methods they represent (if applicable) is provided in Appendix I. Discussed here are:

- Selection of analytical methods
- Standard operating procedures
- Method validation and initial demonstration of capability
- Sample aliquots
- Data verifications
- Standard and reagent documentation and labeling (Refer to Section 10.1)
- Computers and data requirements

8.1 Selection of Analytical Method

CFA's Project Managers are ultimately responsible for selecting the test codes and methods assigned to a client based on client requirements and sample collection techniques. In selecting methods, our goal is to meet the specific needs and requirements of the client while providing data that are scientifically valid.

When the use of a specific test method is mandated, only that method is used. If the analysis cannot be performed by the client-requested method, we notify the client. We do not perform method substitutions without the client's consent. We recommend that clients who submit data to regulatory agencies also obtain the agency's approval of method modifications.

When clients have specific process or reporting deviations from CFA's standard practices, the laboratory may document the deviations in contracts, case narratives and/or with specific work instructions from the Project Management Team to the laboratory. Approval of the deviations is made after consideration of all safety and quality concerns have been resolved by CFA's management.

A Project Management AlphaLIMS Manual (CF-CS-M-001) is available to assist those managing client projects in selecting test codes and methods and communicating the client's analytical and data reporting specifications.

8.2 Standard Operating Procedures (SOPs)

We determine each parameter by the protocol detailed in the corresponding SOP. The defined protocol originates from the analytical method or methods referenced in the SOP and may incorporate regulatory and client requirements. Descriptions of the methods we employ can be found in:

- EPA SW-846, 3rd Edition, Revision III
- American Society for Testing and Materials (ASTM)
- Standard Methods for the Examination of Water and Wastewater (SM)
- Code of Federal Regulations (CFR) Titles 40 and 49
- EPA CLP

Laboratory sections have access to CFA's SOPs to ensure that each operational system and analytical procedure is performed in a uniform manner. SOPs are controlled according to CF-DC-E-001 for Document Control and are posted on the Intranet by the Quality Manager.

We write and issue SOPs in accordance with CF-ADM-E-001 for the Preparation, Authorization, Change, Revision, and Release of Standard Operating Procedures. A technical and/or quality review is made of each new or revised SOP prior to its implementation.

Technical reviews ensure that procedures are technically sound and method-compliant, and are conducted by a senior analyst or data reviewer. The quality review is an independent review by a member of management and ensures that the quality requirements of the method, regulatory agencies, and CFA are adequately and accurately identified.

SOPs are modified when:

- Instruments or equipment change
- An error is identified
- Improvements in technology and/or reagents need to be incorporated

- Reference methods are revised or discontinued

Proposed revisions are submitted for review on Documentation Initiation and Revision Request (DIRR) forms. Changes are not implemented without a technical and quality review.

We review our SOPs at least annually and revise them as necessary. Analytical SOPs either contain or reference other SOPs that contain:

- reference method
- applicable matrix or matrices
- method detection limit
- scope and application including parameters to be analyzed
- method summary
- definitions
- interferences and limitations
- specific safety requirements
- required equipment and supplies
- reagents and standards
- sample collection, preservation, shipment, and storage
- quality control
- calibration and standardization
- procedure
- calculations
- method performance
- pollution prevention
- data assessment and acceptance criteria for quality control measures
- corrective actions for out of control or unacceptable data
- waste management
- references
- tables, diagrams, flowcharts, validation data
- identification of any modifications we have made to the published procedure
- history of changes to the SOP

Administrative SOPs typically do not require all of the above sections. CFA's SOP CF-ADM-E-001 discusses more fully the laboratory practices and policies related to SOP creation and maintenance.

8.3 Method Validation and Initial Demonstration of Capability

An initial demonstration of method performance is required before a new analytical method is implemented and any time that there is a significant change in instrumentation or methodology. Exempted from this requirement are and any tests for which spiking solutions are not available.

We conduct the initial demonstration as described in Section 8.3.1. Records of initial demonstration are maintained in accordance with CF-QS-E-008 for Quality Records Management and Disposition. These records are available upon request.

After we demonstrate our ability to perform a specific analysis, we continue to demonstrate method performance through the analysis of laboratory control samples and performance evaluation samples.

If spiking solutions or quality control samples are not available, an analyst is trained by a qualified trainer to conduct the analysis. Analyst capability and proficiency is evaluated by the Production Manager before the analyst is qualified to perform the analysis on client samples. The evaluation is documented and maintained according to CF-QS-E-017 for Maintaining Technical Training Records.

8.3.1 Procedure for Initial and Continuing Demonstrations of Capability (IDOC and CDOC)

We conduct initial demonstrations of capability for mandated analytical or EPA reference test methods following the procedure outlined below. This procedure is adapted from the EPA test method published in 40 CFR part 136, Appendix A and the 2003 NELAC Standard. IDOCs are completed whenever there is a change in instrument type, method or personnel. CDOCs are completed annually.

Step 1: A quality control sample is obtained from an outside source (if possible). If one is not available, the sample may be prepared internally using stock standards that are prepared independently from those used in instrument calibration. The concentration is not known to the analyst.

Step 2: The QC sample is diluted in a volume of clean matrix. Sufficient volume of the diluted QC sample is prepared so that at least four aliquots of the required method are analyzed. Alternatively, four matrix spike

samples may be evaluated for levels of precision and accuracy.

Step 3: Four aliquots of the diluted quality control sample are prepared and analyzed according to the analytical test method. This may occur concurrently or over a period of days.

Step 4: With the results obtained from the analysis of the diluted QC sample, the average recovery (x) in the appropriate reporting units (such as µg/L) and the standard deviation of the population sample (n-1) (in the same units) are calculated for each parameter of interest.

Step 5: For each parameter, the standard deviation (s) and the average recovery (x) are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria. If "s" and "x" for all parameters meet the acceptance criteria, analysis of samples may begin. If any one parameter exceeds the acceptance range, the performance is unacceptable for that parameter.

Step 6: When one or more tested parameters fail one or more of the acceptance criteria, we locate and correct the source of the problem and repeat the test for every parameter of interest.

Other options for successful IDOCs are the following:

- PT Study- successful analysis of a PT Sample. The PT sample may be single-blind to the analyst or double blind to the laboratory.
- Supervised Analysis- where other options are not practical, supervised analysis of a procedure may be used to demonstrate capability.
- Analysis of authentic sample with results statistically matching those obtained by another trained analyst.
- Other – this option may be used for certain personnel having sufficient analytical skills to develop a new procedure, as deemed appropriate by the supervisor or Quality Assurance personnel.

8.4 Sample Aliquots

When obtaining aliquots from a sample, it is imperative that the subsamples be representative of the parent sample. This ensures that the results obtained from the analysis of the aliquots are representative of the entire parent sample, not just the subsample. We employ different techniques to obtain subsamples. CFA's SOP for sample compositing is CF-LB-E-031. The appropriate techniques for obtaining sample aliquots for designated analyses are discussed in the applicable SOPs.

8.5 Data Verification

All of the data we include in final reports to our clients undergoes extensive data verification. At CFA, we have a multi-level review process that takes place in all areas of the laboratory beginning with sample login. This process and the responsibilities of each level of review are delineated in the procedure for data review and packaging, CF-LB-E-005.

8.5.1 Sample Login:

Samples are analyzed by the methods and for the target analytes identified when samples are logged into our database. If there is an error in this entry that is not promptly identified, the incorrect analytical method may be used or certain analytes may not be determined.

To prevent this, the person who enters the information into the database is generally the client's assigned Project Manager. This entered information is reviewed against the client confirmation letter and/or chain of custody. If errors are identified, they are immediately corrected.

8.5.2 Data Validation in the Laboratory

The multi-level review process in our laboratory includes initial review by the analyst, a final review by the data reviewer, and a completeness review by the project manager.

Our analytical data reviews ensure that:

- The analytical procedures comply with current SOPs.
- Quality control samples are analyzed at the frequency specified in the SOP or client specifications.
- The acceptance criteria for quality control samples are met, including recoveries of matrix spikes and laboratory control samples, the relative percent difference for matrix duplicates, matrix spike duplicates, laboratory control sample duplicates, and concentrations of target analytes in the method blank.
- Instrument data, run logs, and logbooks are reviewed to ensure that all method quality control

criteria were met (e.g., initial calibration and continuing calibration verifications).

- Documentation is sufficient to reconstruct the analytical procedure.
- Data are maintained according to CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.
- Raw data are in agreement with the computer generated batch sheets and data reports.
- The calculations, dilution factors, concentration reported, and nominal concentrations are verified.
- Comments, qualifiers, or nonconformances for noncompliant or questionable data are documented.
- Data generated when the analytical process appears to be out of statistical control are not reported.

8.5.3 Validation of Data Reports and Packages

Before data is reported to the client, the data report is reviewed for package accuracy, completeness, and client specifications. Responsibilities for review are dependent upon the type of report or package being generated. (Refer to Section 11 for Laboratory Report Formats.)

The Project Manager (PM) reviews client information for accuracy, completeness and the addition of pertinent comments made by the laboratory about the analysis or sample. The PM also reviews data for consistency as described in the Project Management AlphaLIMS Manual, CF-CS-M-001.

The data validator reviews the analyst-prepared case narrative for accuracy and to assure its consistency with the information included on the certificate of analysis and Quality Control Summary Report. Each laboratory fraction of data is reviewed by that fraction's data validator. The data are then compiled into a final data package.

A minimum of 10% of all DoD ELAP data packages will be reviewed by the Quality Manager (or designee) for technical completeness and accuracy. This review is not required to be performed prior to release of the data to the client. The Quality Manager, or designee, must maintain documentation of the reviews to establish the minimum requirement having been met.

8.6 Standard and Reagent Documentation and Labeling

The documentation and labeling of standards and reagents is addressed in CF-LB-E-007 for Laboratory Standards Documentation, and in Section 10.1 of the QAP, Recordkeeping System and Design.

8.7 Computer and Electronic Data Related Requirements

Our Information Management System (IT) SOPs describe the way in which we manage our software programs and hardware systems. Control of software development and modification activities is described in CF-IT-E-003 for Requirements, Design, Operation, Validation, and Removal of Hardware and Software Systems. All development and revision activities are validated, verified, and controlled with revision software or other procedures prior to production use.

Analytical software that is purchased from a vendor is validated and verified in accordance with CF-IT-E-005 for Requirements, Design, Operation, Validation, and Removal of Applications. Documentation requirements are also described in this SOP.

SECTION 9

SAMPLE HANDLING, ACCEPTANCE, RECEIPT, AND INTERNAL CHAIN OF CUSTODY

Section 9 - Sample Handling, Acceptance, Receipt, and Internal Chain of Custody

The way we receive and handle samples is critical to providing our clients with data that are of the highest quality and are legally defensible. We have strict policies that govern the acceptance and receipt of a sample, sample handling and integrity, maintenance of the internal chain of custody, and storage of the sample upon completion of the required analytical processes. This section describes the policies and practices that we employ, including the following:

- Agreements to perform analysis
- Proper labeling of submitted samples
- Chains of custody
- Sample receipt procedures
- Sample tracking
- Sample storage
- Sample disposal
- Emergency Sample Storage

9.1 Agreement to Perform Analysis

Before we accept samples, we should have an agreement with the client that specifies the analytical methods, the number of samples to be analyzed, the price for the analysis, the date by which the client will receive results, and the reporting format. Any special requirements the client may have, such as non-routine methods and reporting limits, should be part of that agreement.

An agreement to perform analysis should be in one of three forms, further detailed in our the SOPs for Delegated Authoriity to Commit the Company and Request for Proposal (RFP) and Contract Review (CF-CO-E-002 and CF-CO-E-003):

- Client confirmation letter (CCL) between the client and project manager for a specific group of samples. This letter includes the cost, turn-around time, requested analysis, sample matrix, number of samples, and type of client report.
- Sample acceptance by the Project Manager from an established client based on previously agreed conditions and confirmed by the client's submission of the sample(s).
- Contractual agreement for analytical services over a designated time period or project that delineates the agreed upon specifications.

When the laboratory agrees to perform analyses with exceptional departures from normal processes, these exceptions are clearly defined in the client-laboratory agreement.

9.2 Sample Labels and Chain of Custody Forms

Once an agreement is established, we assume joint responsibility with the client to ensure that the samples submitted are properly labeled and accompanied by full and complete documentation that includes chain of custody and, where possible, material safety data sheets. Samples that are submitted without proper documentation may be refused.

Sample labels should include:

- client sample identification
- location, date, and time of collection
- collector's name
- chemical preservatives used
- constituents of interest (if space permits)

When requested, we ship labeled sample containers with appropriate preservatives and a chain of custody to the client for use during sample collection. There are several advantages to using these containers, including:

- Dedication of appropriate type sample container for the intended analyte or analytical method.
- Proper sample preservation for analytical test
- Traceability of bottle lot number to the manufacturer's certification that the containers are clean and show no signs of contamination.

Chain of custody forms include the following information and are initiated at the time of sample collection:

- name and address of client
- client sample identification
- date and time of sample collection

- sample matrix
- description of sampling site location
- number of containers
- methods, chemical and physical constituents for which the analyses are to be conducted
- preservatives
- date and signature of person who collected the sample
- date of transfer and signature of person relinquishing sample to the laboratory.

Our standard chain of custody forms are available to our clients and are included with each shipment of pre-labeled and preserved containers. CFA chain of custody forms should always be used unless otherwise agreed to by contract.

9.3 Sample Conditions

In addition to properly documenting sample container labels and the chain of custody form, we need to make sure that samples meet the established requirements for analytical testing. This is particularly critical for samples that are being analyzed to meet regulatory requirements.

Samples should be collected in the appropriate type of container, preserved as directed, and stored under the conditions specified in the analytical method or established regulatory guidelines. In addition, samples should be submitted with sufficient time to conduct the specified analysis within the regulatory or method holding time. Aliquots should be of sufficient volume to perform the requested analyses. A summary of these conditions and holding times for routine analyses can be found in Appendix J.

9.4 Sample Receipt

Samples submitted to us are received in a central sample receiving area by our sample custodian or login clerk. Every sample is subject to the protocols established in CF-SR-E-001 for Sample Receipt, Login and Storage.

Our sample custodian acknowledges receipt of a sample by signing the chain of custody and recording the date and time custody was transferred from the client to the laboratory. The date, time, and person receiving the sample are also recorded on a standard or client-specific Sample Receipt Checklist (SRC) form.

The sample custodian is also responsible for noting the condition of a sample upon its arrival. This information is recorded on both the sample chain of custody and the Sample Review Receipt form. As detailed in CF-SR-E-001, the sample custodian should:

- Document whether custody seals are present and intact on the outside of the sample cooler.
- Inspect all sample containers for integrity.
- Document any unusual physical damage or signs of tampering with custody seals.
- Place any samples that appear to be leaking or have unusual odor under the fume hood while notifying the responsible project manager.
- Review the chain of custody submitted by the client for completeness.
- Compare descriptions and other information on the sample container labels to that listed on the chain of custody.
- Verify the sample is within the regulatory holding time for the analyses.
- Measure and record the temperature of the cooler using the temperature blank (if provided) or a representative sample container.
- Measure and record the pH of all sample aliquots submitted for analyses that require chemical preservation to a specific pH except those submitted for Volatile Organic Compounds. (VOA) The pH preservation of VOA samples is verified in the analytical laboratory.
- Check water samples for residual chlorine if dioxin/furan or PCB analysis is requested. If residual chlorine is present, preserve wih sodium thiosulfate as directed in CF-SR-E-001, and document on the SRC.
- Verify that there are adequate sample aliquots for the requested analyses.
- Verify that appropriate sample containers were used for requested analyses.

If the sample custodian discovers any abnormalities or departures from standard conditions, the PM is informed immediately. The PM will then notify the client as quickly as possible so that a decision can be made to proceed with the analysis or submit another sample or additional sample aliquots.

Common abnormalities or departures from standard conditions include:

- Sample containers with signs of damage, leaking, or tampering.
- Incomplete/missing chain of custody.

  NOTE: If a sample has no chain of custody, the sample custodian should initiate one. "INITIATED ON RECEIPT" should be documented on the chain of custody.
- Discrepancies between the information on the chain of custody and the sample container labels.
- Method or regulatory holding time is exceeded.
- Sample is not preserved to the method or regulatory-required pH.
- The sample container does not meet method or regulatory criteria.
- The sample temperature exceeds or falls below the thermal preservation regulation or method requirement of 0° - 6° C.

  NOTE: If a sample is hand-delivered to the laboratory immediately after collection with evidence that the chilling process has begun (arrival on ice), the sample shall be deemed acceptable.

9.5 Sample Tracking

We track the samples we receive by a unique laboratory identification number that is automatically assigned when information pertaining to the sample is first entered into our database. Pursuant to CF-SR-E-001, the following information is entered for each sample received:

- client and/or project code
- client sample ID
- sample matrix
- chemical preservative (if applicable)
- date and time of collection
- date received
- initials of person making entries
- number of containers submitted for the sample
- requested analyses
- pertinent observations or comments affecting the sample analysis or rejection
- container type

As soon as this information is entered, AlphaLIMS automatically assigns a unique number to the sample and its containers. We use these numbers to track the location of a sample container and to link to any subsamples and subsequent leachates and extracts.

The unique laboratory identification number is printed on a durable barcode label that contains the client identification, sample date and time. Once labeled, the sample container's identification number is uploaded into the database by scanning the barcode. Information included in the database at the time of sample scanning is the container's storage location, bottle type and volume, physical characteristics of the bottle, preservative, and the initials of the person entering this information. Entering of this information into the database is an important part of initiating our electronic internal chain of custody.

9.6 Internal Chain of Custody

Chain of custody procedures ensure traceability and sample integrity. Our legal and evidentiary chain of custody protocol establishes a continuous record of the physical possession, storage, and disposal of sample containers, collected samples and aliquots, and sample digestates or extracts.

The internal chain of custody starts with the scanning of a container's barcode label into an electronic database while identifying the location of the sample and the person having custody, or placing the sample in a secured storage area. If we supply the containers, the chain of custody may begin when the containers are provided to the client.

With regard to the internal chain of custody, a sample is defined as being in someone's custody if:

- It is in one's actual physical possession, or
- It is in one's view after being in one's physical possession, or
- It is in one's possession and then is locked up so that no tampering may occur, or
- It is kept in a secured area restricted to authorized personnel only.

The protocol for ensuring sample integrity using the internal chain of custody is detailed in CF-LB-E-012 for Verifying the Maintenance of Sample Integrity. The electronic internal chain of custody works in conjunction with the chain of custody submitted by the client with a sample to:

- Account for all time associated with a sample, its subsamples, and extracts or digestates from the time the sample is received at CFA to its disposal
- Identify all individuals who physically handled the sample
- Provide evidence that the sample was stored in accordance with method and regulatory protocols

The electronic internal chain of custody is stored in AlphaLIMS so that information demonstrating the proper maintenance of custody can be provided to the client on the data reports or electronic data deliverables.

9.7 Sample Storage

In order to ensure the maintenance of sample integrity, all aliquots are stored in secured areas designated for sample storage. The storage location of each sample aliquot can be tracked using the internal chain of custody. Areas designated for sample storage include:

- Main cooler where most samples requiring maintenance at a temperature range of 0° - 6° C are stored.
- Volatiles coolers and freezers for samples to be analyzed for volatile contaminants.
- Ambient storage for samples not requiring refrigeration.
- Freezers for the storage of samples requiring maintenance at a temperature of $\leq$-10°C (i.e. tissues).

The temperature of each refrigerated sample storage unit is monitored daily and documented per CF-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens and Other Similar Devices. In addition, the main cooler is monitored twenty-four hours a day by temperature sensor that is connected to our main security system. If the temperatures exceed the required range, an alarm is sounded and the security system notifies the laboratory manager or his designee immediately. This allows corrective actions to be initiated promptly.

Prior to and immediately after analysis, samples and their leachates and extracts are stored in compliance with the requirements of the requested analytical methods and CF-SR-E-001 for Sample Receipt, Login, and Storage. If a single aliquot is supplied for analyses by several methods, the most stringent analytical storage requirements are applied to the sample.

If samples are to be analyzed for volatile organic compounds, they are stored in designated volatile coolers that are maintained at a temperature range of 0°- 6° C. No sample aliquots are stored in these refrigerators unless they are to be analyzed for volatiles. These storage units are monitored on a weekly basis for contamination by the analysis of volatile cooler storage blanks.

At the beginning of each month, four 40 mL vials are filled with treated deionized water, which is used for volatile method blanks, and placed in each volatiles cooler. Each week, one vial is analyzed by EPA 8260C and the data are reported to the Quality Department. If the analysis reveals evidence of potential contamination, appropriate corrective actions are immediately implemented.

After all analyses are complete and results are submitted to the client, sample aliquots are transferred to the sample archive area. They are stored in this area until they are disposed.

9.8 Sample Disposal

Our policies concerning sample disposal are described in the Laboratory Waste Management Plan, CF-LB-G-001 and can be divided into two categories: those governing the disposal of sample laboratory waste, and those directing the disposal of remaining sample aliquots after the completion of all analyses.

9.8.1 Laboratory waste

Unless otherwise requested by contract, laboratory sample waste is collected throughout the laboratory in designated satellite containers found in sample collection and accumulation areas. Sample wastes are segregated based on the type of analysis by which they were generated and by matrix. This contains certain process contaminants thus decreasing the amount of waste material that may be labeled hazardous. It also ensures that solid and aqueous wastes are not mixed.

The composited sample wastes then undergo hazardous waste characterization. The analyses allow CFA to properly characterize the waste according to EPA regulations.

Sample waste is disposed in accordance with the Laboratory Waste Management Plan, CF-LB-G-001.

9.8.2 Remaining Sample Aliquots

Samples not consumed during the sample preparation or analytical procedures are either returned to the client in accordance with CF-SR-E-002 for Transportation and Shipping of Samples and Pre-Preserved Sample Containers or disposed pursuant to the Laboratory Waste Management Plan. Samples are returned to a client under the conditions and terms agreed to by contract. A chain of custody listing the laboratory waste technician as the relinquishing party is enclosed with each set of samples being returned to a client. Unless otherwise specified by the client, all samples are shipped by an approved package carrier. The procedure for shipment is delineated in CF-SR-E-002 for Transportation and Shipping of Samples and Pre-Preserved Sample Containers.

It is our policy to hold samples for a minimum of thirty days after invoicing and before disposal, unless otherwise specified by contract or if the sample is part of litigation. If the sample is part of litigation, disposal of the physical sample shall occur only with concurrence of the affected legal authority, sample data user, and/or client.

When sample analyses are complete and regulatory and/or contractual holding times have expired, samples are moved from their storage locations to the sample archives. Samples that are to be returned to the client or held for an extended time period are segregated from the other samples.

When internal or client-specified storage time expires, samples with like matrices are composited into appropriate containers. The composites are then subject to the same treatment and disposal protocol as described in 9.8.1. Samples that are approved for disposal are scanned into our database and assigned the status of "Disposed."

9.9 Emergency Sample Storage

In the event of an impending disaster (such as a hurricane), the laboratory will make arrangements to store samples and their extracts at an approved off-site facility. CF-QS-E-001 is used to identify acceptable storage facilities that meet the requirements of the laboratory policies pertaining to proper storage conditions and documented chain-of-custody.

SECTION 10
RECORDS

Section 10 - Records

Our quality records provide the documentation we need to support analytical results and conclusions. Documented evidence that quality assurance and quality control requirements have been met is critical to providing data that fulfill the specifications of applicable procedures, programs, and contracts.

As described in Section 3 of this Quality Assurance Plan (QAP), quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- External, supplier, and internal audits
- Run logs
- Instrument data and analytical logbooks
- Instrument, equipment and building maintenance logs
- Material requisition forms
- Monitoring logs
- Nonconformance reports
- Corrective actions
- Method development and start-up procedures including MDL, LOD and LOQ verification studies
- Training records
- Waste management records
- Standard logs
- Software validation
- Standard operating procedures (SOPs)
- Sample collection and field data

Our procedures to provide a legal and evidentiary chain of custody are described in Section 9 of this QAP. Described in this section are:

- Recordkeeping system and design
- Records management and storage
- Sample handling records
- Records of support activities
- Analytical records
- Administrative records

10.1 Recordkeeping System and Design

We manage, maintain and store our quality records according to CF-QS-E-008 for Quality Records Management and Disposition. The protocols established in this document work in conjunction with those for specific types of records addressed in other SOPs to govern our record keeping system. Our record keeping system allows the historical reconstruction of all laboratory activities that produce analytical data.

We facilitate historical reconstruction by maintaining the following records and information, from the time a sample is received until it is disposed.

- A master list of all employee signatures and initials is maintained by the Quality Manager. This allows the identification of any CFA personnel who accept, handle, analyze, prepare, review, store, or dispose of a sample, its subsamples, associated data and reports, and other related documentation.
- If we provide bottles and containers to a client or sampling personnel, these records are kept in accordance with CF-SR-E-002 Transportation and Shipping of Sample and Pre-preserved Sample Containers.

The person or agency responsible for collecting a sample is documented on the chain of custody and entered into AlphaLIMS. Other records supporting the acceptance of a sample may include:

- Date and time of sample receipt
- Person accepting sample
- Condition of sample upon receipt
- Client chain of custody
- Electronically generated sample ID numbers specific to each sample aliquot and linked to the client's sample description, sample collection and receipt information, and analyses to be performed.
- Identification of each person who has custody of a sample, its subsamples, extracts, or leachates, as requested. (This is provided through the internal chain of custody procedures described in Section 9.)

Documentation that materials purchased for use in the analysis or preparation of samples meet

specifications is maintained in accordance with CF-RC-E-001 for Receipt and Inspection of Material and Services.

Records of equipment calibrations are maintained and traceable by date and unique ID number to a specific analysis. These records include certifications of calibration and service that have been initialed or signed.

Our thermometers are verified against the NIST traceable thermometer and records of this verification are maintained as described in CF-QS-E-007 for Thermometer Verification. Records of the daily calibration verifications of our analytical balances are kept in accordance with CF-LB-E-002 for Balances. The calibration records for our air-displacement pipets are maintained in pipet calibration logs specific to each pipet according to CF-LB-E-010 for Maintenance and Use of Air Displacement Pipets.

When methods and/or regulations specify that samples, subsamples, extracts, and/or leachates be stored at designated temperatures, or when the method itself has temperature sensitive steps, we document those temperatures on monitoring logs at the frequency defined in the corresponding SOPs. We can trace the specific storage location of a sample through the internal chain of custody.

We require that the initials of all personnel responsible for monitoring temperatures be recorded in the temperature monitoring logs pursuant to CF-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens and Other Similar Devices. The logs are reviewed for completeness in accordance with CF-QS-E-005 for Review of Monitoring Device Logs.

Documentation on the instruments and equipment used for the analysis of samples is recorded in run logs, laboratory logbooks, instrument data and/or sample preparation logs. Routine or corrective maintenance that is performed on equipment or instruments is recorded in the maintenance log specific to the instrument. We document these records in accordance with CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

The standards containing known quantities of target analytes that we use in instrument calibration, calibration verification, and as quality control samples, such as matrix spikes and laboratory control samples, are documented according to CF-LB-E-007 for Laboratory Standards Documentation. These records contain the following information:

- Protocol by which each standard was prepared
- Traceability of each child standard to its parent
- Date each standard was prepared
- Initials of person preparing the standard
- Expiration dates
- Concentration of each standard
- Certificate of Analysis, provided by the supplier (if available)

This information allows us to document that the standards used were prepared in accordance with the established protocol, produced using source standards that meet the method and regulatory criteria, and used prior to their expiration date.

If required, reagents used in the preparation, dilution, and analysis of samples are verified to be free of interferences or target analytes. We record these verifications in the reagent logs in accordance with CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

Analytical and sample preparation methods applied to each sample aliquot are documented via the internal chain of custody, method information, and information recorded in lab notebooks, sample preparation logs, run logs, and instrument data. The laboratory protocol we employ during analysis is dictated by the SOP in effect at the time the sample was analyzed or prepared by a specific method.

Run logs, laboratory notebooks, instrument data and sample preparation logs are used to document the preparation and analysis of samples and the associated instrument calibrations. These logs and notebooks are governed by CF-LB-E-009 for Run Logs and CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices. As stated in these SOPs, sample preparation and analytical records that are not electronically generated should be:

- Legible
- Recorded in permanent ink
- Corrected using one line marked through the error, initialed and dated
- Initialed by the responsible party

We maintain electronic records for each analytical batch. These records include the ID numbers of each client and quality control sample prepared and/or analyzed together, the method of preparation and analysis, and the matrix of the samples included in the batch.

Through our electronic statistical process control system (SPC), the acceptance criteria applied for all quality control (QC) samples are stored and maintained as described in CF-QS-E-014. The acceptance limits for target analytes are method, matrix, and time-period specific, which allow us to generate the criteria applied to QC samples associated with identified client samples.

Our Quality Manager maintains the records of nonconformances and corrective actions associated with specific samples, batches, and processes. We maintain these records according to CF-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and CF-QS-E-002 for Conducting Corrective/Preventative Action and Identifying Opportunities for Improvement.

Electronic data records are maintained in a secured database designed to protect the integrity of the data. Data that are uploaded directly from instruments and that are manually entered are backed up by a second system.

Permanent records of electronic data deliverables are maintained along with the corresponding sample preparation and analytical data review records. This documentation includes the initials of the reviewer and date of the review.

Records of the data we report to our clients are maintained in a manner that protects client confidentiality, as well as any potential national security concerns. These records include copies of certificates of analysis, quality control summary reports, case narratives, CLP forms, and other information we provided to the client. The copies may be paper or electronic.

Records of samples being disposed or returned to the client are documented in accordance with CF-SR-E-002 for Transportation and Shipping of Samples and Pre-Preserved Sample Containers. Such records include the date samples are returned or disposed, the destination of the samples, and name of the person transferring the samples.

10.2 Record Storage

We store quality records in compliance with CF-QS-E-008 for Quality Records Management and Disposition. The records are:

- Stored in a secured area to maintain data integrity and protect client confidentiality, including any national security concerns.
- Kept in areas where they are protected from fire loss, environmental deterioration, and, in the case of electronic records, electronic or magnetic sources.
- Indexed and filed in a manner allowing for ready retrieval.
- Retained for an identified period of time that equals or exceeds five years as determined by applicable law and client contract requirements.

Electronic data records are stored on compact disks.

All of the hardware and software we need to reconstruct data is maintained according to CF-IT-E-003 for Requirements, Design, Operation, Validation and Removal of Hardware and Software Systems Used by CFA. Records that are stored or generated by network or personal computers have either hard copy or write-protected backup.

10.3 Sample Handling Policy

Records of all procedures applicable to samples are maintained in our possession. These records include documents that pertain to:

- Preservation, including sample container and holding time
- Sample identification, receipt, acceptance or rejection, and login
- Sample storage and tracking including shipping receipts, transmittal forms, routing and assignment records
- Sample preparation (ID codes, cleanup and separation protocols, volumes, weights, instrument printouts, meter readings, calculations, reagents)
- Sample analysis
- Standard and reagent origin, receipt, preparation, and use
- Equipment receipt, use, specification, operating conditions and preventive maintenance

- Instrument calibration frequency and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Method performance criteria including expected quality control requirements
- Quality control protocols
- Electronic data security, software documentation and verification, software and hardware audits, backups and records of any changes to automated data entries
- Disposal of hazardous samples

10.4 Records of Laboratory Support Activities

In addition to sample handling records, we maintain the following:

- Original raw data for calibrations, samples and quality control measures, including worksheets and data output records (chromatograms, strip charts, and other instrument readout records)
- A written description of or reference to the specific method used, including the computational steps used to translate parameter observations into a reportable analytical value
- Copies of final reports
- Archived standard operating procedures
- Correspondence relating to project-specific laboratory activities
- Corrective action reports, audits and audit responses
- Proficiency test results

10.5 Analytical Records

We document and maintain analytical records, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs according to CF-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices, and CF-LB-E-009 for Run Logs.

The information that is documented in analytical records includes:

- Laboratory sample ID code
- Date and time of analysis
- Instrument ID and operating conditions/parameter (or reference to such data)
- Method of analysis
- All calculations
- Dilutions
- Initials of analyst or operator
- Units of measurement

Our policy is to produce and maintain analytical records that are:

- Accurate
- Reviewed and verified
- Legible and understandable
- Traceable and authentic to their source
- Grouped in a contemporary manner with data entered and information recorded as it is obtained

10.6 Administrative Records

A number of pertinent records are maintained by the Quality Manager, including:

- Staff qualifications and experience.
- Training records, including initial demonstrations of proficiency. (Refer to procedure CF-HR-E-002 for Employee Training.)
- A log of names, initials and signatures for individuals having responsibility for initialing laboratory records.

We monitor continuing demonstrations of proficiency through AlphaLIMS per CF-HR-E-002 for Employee Training.

SECTION 11

LABORATORY REPORT FORMAT AND CONTENTS

Section 11 - Laboratory Report Format and Contents

Accurate data are of little benefit to a client unless they are reported in a format that is easy to interpret and provides all pertinent information relating to the analysis of a sample. At CFA, we have developed certificate of analysis report formats that meet the different needs of our clients, yet provide all of the information necessary to satisfy regulatory requirements while allowing for the interpretation of the data. Each format provides accurate, clear, unambiguous and objective data.

In addition to a certificate of analysis, a client can request and receive an extended data package. This package may include any of the following: certificates of analysis; summaries of quality control; case narratives; instrument data; sample preparation data; measurement traceability and calibration information; and electronic data deliverables. If clients require the reporting of data following the established contract laboratory protocol (CLP), we can provide a CLP-like data package that will meet their needs.

It is important that the certificate of analysis format and data package requirements be discussed with the client prior to our acceptance of the samples. Project Managers are responsible for establishing an agreement with the client concerning data reporting and the potential cost to the client for data packages and/or specialized reporting. Our analytical data are reported to three significant figures unless otherwise required by client contract.

Laboratory reports and data packages are stored and transmitted in a manner that protects client confidentiality and potential matters of national security. No reports or data packages are released to persons or organizations outside CFA without the express consent of the client. If directed by a regulatory agency or subpoenaed to submit documents to a court of law, we will notify the client of the demand and the records being released.

The following elements of report formats and data packages are described in this section:

- Certificates of analysis (C of A)
- Quality control summary reports (QCSR)
- Analytical case narratives
- Electronic data deliverables (EDDs)
- Types of data packages and reporting formats
- Review of data packages and reports

11.1 Certificates of Analysis

We have two primary C of A report formats, Level 1 and Level 2. Both contain the following information when applicable:

- Title
- CFA address and phone number
- Name of PM or person serving as the primary client contact
- Barcode identification of the C of A
- Number of page and total number of pages
- Name and address of client, where appropriate
- Project name or code if applicable
- Client-provided sample description
- Unique laboratory ID number for the sample
- Sample matrix
- Characterization and condition of the sample where relevant
- Date of receipt of sample
- Date and time of sample collection, if provided
- Date and time of sample analysis, reanalysis, and/or sample preparation
- Initials of analyst and person responsible for sample prep
- Analytical batch number
- Sample analysis and preparation methods (or unambiguous description of any non-standard method used)
- Reference to sampling procedure
- Additions to or deviations or exclusions from the test method, and other information relevant to a specific test, such as environmental conditions and the use and meaning of data qualifiers
- Nonconformances that affect the data
- Whether data are calculated on a dry weight or wet weight basis
- Identification of the reporting units, such as µg/L or mg/kg

- Statement of the estimated uncertainty of the test result, if applicable
- Signature and title of the person(s) accepting responsibility for the content of the C of A
- Date C of A was issued
- Clear identification of data provided by outside sources, such as air temperature or ambient water temperature
- Identification of the reporting limit (RL) or practical quantitation limit (PQL) for each analyte, if applicable.

If a portion of the sample analysis is subcontracted, the C of A will identify the subcontractor or applicable accreditation number, and the data that was determined by the subcontracting laboratory.

Level 2 Certificates of Analysis contain the following additional information:

- Dilution factors
- Method detection limits
- Surrogate recoveries and the acceptance criteria for all organic analyses
- Estimated concentrations determined for nondetects and appropriate "U" and "J" qualifiers for nondetects and concentrations that fall between the MDL and PQL respectively.

Once issued, a C of A is not altered unless a subsequent C of A is identified as a revised report.

11.2 Quality Control Summary Report (QCSR)

The quality control data that demonstrate the sample preparation and/or analytical efficiency of the batch are summarized on a QCSR. The data reported on the QCSR may be limited to a sample delivery group contained in the batch or may include all quality control for the batch. Information reported on QCSR includes:

- Quality control sample ID number
- Type of quality control sample
- Concentrations determined, where applicable, for method blanks, matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples, serial dilutions, and laboratory control sample duplicates
- Acceptance criteria for matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples, and laboratory control sample duplicates
- Nominal concentrations of matrix spikes, matrix spike duplicates, LCSs, and LCS duplicates
- Concentration of parent sample for the matrix spikes, matrix spike duplicates, or sample duplicates
- Percent recoveries for LCS and matrix spikes
- Relative percent differences for the matrix spike duplicates, matrix duplicates, and LCS duplicates
- Analytical batch number with which the quality control data is associated
- Parent sample numbers for matrix spikes, matrix duplicates, and matrix spike duplicates
- Sample or sample delivery group ID
- Project code
- Date issued, page numbers/total number of pages
- Identification of recoveries or relative percent differences that do not meet the acceptance criteria

11.3 Analytical Case Narratives

Analytical case narratives are written by an analyst or data validator to describe the overall conditions affecting the analysis of a batch or a specific sample in the batch. Case narratives usually include:

- Sample delivery group ID number
- Analytical batch number
- Methods of preparation and analysis
- Sample matrix
- Initial of person preparing and/or reviewing the narrative
- Specific sample ID numbers
- Identification and description of batch quality control samples including parent sample identification
- Affirmation that all sample preparation conditions specified by the method or regulatory agencies were met or identification of specific deviations
- Affirmation that all analysis criteria specified by the method or regulatory agencies were met or identification of specific deviations
- Instrumentation employed if applicable and verification of its calibration
- Summary of batch quality control as compared to acceptance criteria
- Identification of nonconformances
- Pertinent comments and observations of factors that affect sample data quality

11.4 Electronic Data Deliverables (EDDs)

Electronic data deliverables are generated according to client specifications. EDDs use programs supplied by the client or created internally by our EDD team. Internally generated EDDs are usually written in Perl and/or PL/SQL.

11.5 Types of Data Packages and Reports

We offer three levels of data reports and the ability to design packages to meet the needs of our clients. The levels of data reports are summarized in Table 1.

Table 1: Data Report Formats

Level	Contents
1	Level 1 C of A
2	Level 2 C of A plus QCSR, narratives, and any required QC forms
3	Level 2 plus raw data

If a client so requests, the above reports can be accompanied by EDDs, case narratives, copies of associated nonconformance reports, and other support documentation. The client's specific requirements are communicated to the laboratory and data reviewers through AlphaLIMS.

CFA's SOP CF-CS-E-002 for The Internal Review of Contractually Required Quality Criteria for Client Package Delivery defines preparation and review of the package.

11.6 Review of Data Reports, EDDs, and Data Packages

Level 1 and Level 2 data reports are reviewed for accuracy and completeness by the PM . Level 3 data packages are reviewed in the laboratory by a data reviewer, who is responsible for reviewing specific fractions of the data package for accuracy, consistency, and completeness in accordance with the SOP for that lab area.

SECTION 12

SUBCONTRACTING ANALYTICAL SAMPLES AND OUTSIDE SUPPORT SERVICES

Section 12 - Subcontracting Analytical Samples and Outside Support Services

The subcontracting of samples to other facilities may occur when:

- The client has requested analytical services for which we are not certified or do not offer as a routine product.
- The regulatory or method holding times and/or client due dates are in danger of not being met as the result of instrument malfunction or the unexpected influx of a large group of samples.

No samples are subcontracted without the client's consent. The laboratories selected to receive subcontracted samples are expected to meet the following criteria:

- Demonstrated technical capability to provide data that meet and conform to our quality standards.
- Established certification, if available, for the requested analyses.
- Successful proficiency evaluation results, if available.
- Commitment to meet time requirements for delivery of results to the client.
- Agreement to provide all documentation requested in conjunction with the analysis.
- NELAP or ISO/IEC 17025 accreditation for the analysis if required by the client.

We audit potential subcontractors for technical and administrative compliance as directed in CF-QS-E-001 for Conduct of Quality Audits. An audit may be in the form of a book/desk audit or an on-site review.

If there is evidence of a technical, administrative, or quality deterioration, the laboratory is removed from our list of approved subcontractor laboratories pending further evaluation, which may include an on-site audit. Once the laboratory again demonstrates compliance with CFA's standards, it can be reclassified as an approved subcontractor laboratory.

At CFA, we have a multi-faceted and trained staff. There are occasions, however, when it may be necessary to obtain the services of professionals outside of CFA. This may be due to such things as sample workload, introduction of a new instrument or method requiring special knowledge, or employee leave of absence.

Any outside support services or service personnel are subject to the same scrutiny as a subcontract laboratory. If a service fails to meet our standards for excellence, the appropriate parties are promptly notified. If immediate corrections are not implemented and services are not of adequate quality to maintain confidence, the contract is canceled.

SECTION 13
CLIENT SATISFACTION

Section 13 - Client Satisfaction

Meeting the needs and expectations of our clients is essential to meeting our commitment to be the environmental laboratory of first choice. An important part of meeting this commitment involves receiving and resolving client concerns and complaints.

Client complaints that question the quality of laboratory data or data deliverables are directed to Quality Systems. These concerns are responded to with input from the laboratory, or support group as may be needed.

The types of complaints, area(s) affected, and any impacts on quality are trended on a quarterly basis. This information is available to management at any time.

We use AlphaLIMS to monitor client complaints, nonconformances and corrective actions. Every complaint is entered into the system upon receipt and assigned an internal and external due date. The external due date is often established by client contract. The internal due date allows time for the Quality Manager to review the response and transmit it to the client on or before the due date.

If we notice a trend that significantly affects the quality of our data, a corrective action is initiated following CF-QS-E-002 for Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement. The implementation and verification of the corrective action affirms an effective and permanent solution. Corrective Action investigations are verified for effective implementation during subsequent internal audits of the laboratory area where changes were made.

APPENDIX A: REFERENCES

- National Environmental Laboratory Accreditation Program, (NELAP), 2003 NELAC Standard
- 40 CFR Part 136, October 1984, Part VII, EPA 600 Series Methodologies for the Analysis of Organic Contaminants.
- EPA Requirements for Quality Assurance Project Plans (QAPPs), US EPA QA/R5.
- US Department of Defense Quality Systems Manual for Environmental Laboratories, Versions 3 and 4.1.
- ANSI/ISO/IEC 17025-2005
- DOE G414/1 – 3, 11/3/04 Suspect and Counterfeit Items

APPENDIX B: DEFINITIONS

The following definitions are used throughout the text of our Quality Systems Plan. These definitions were reprinted from "Definitions for Quality Systems," NELAC, July 1, 1999.The original source of each definition is provided.

AlphaLIMS: CFA's Laboratory Information Management System.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in the requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

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. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Aliquot: A discrete, measured, representative portion of sample taken for analysis. (DoD, EPA QAD Glossary)

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. (NELAC)

Analyte: The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and are analyzed together. (EPA Risk Assessment Guide for Superfund, OSHA Glossary)

Analytical Detection Limit: The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given confidence interval. (NELAC Quality Systems Committee)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (NELAC Quality Systems Committee)

ANSI: American National Standards Institute--this consensus standards body approves standards as a guide to aid the manufacturer, the consumer and the general public who may be concerned with its scope and provisions.

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples 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 to 20 environmental samples of the same NELAC-defined 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 24 hours. An analytical batch is composed of prepared environmental samples (extracts, leachates or concentrates) that are analyzed together as a group using the same calibration curve or factor. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

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 subject 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. NOTE: Blank correction is not performed on organic analytes. (ASQC)

Blind Sample: A subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibrate: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration: The set of operations that establish, under specified conditions, the relationship between values indicated by a measuring device, or the correct value of each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number of and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC Quality Systems Committee)

Confirmation: Verification of the presence of a component through the use of an analytical technique that differs from the original test method. These may include: (NELAC)

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures

Corrective Action: 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 are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form. (EPA-QAD)

Detection Limit: The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. Refer to Method Detection Limit. (NELAC)

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 is 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)

Holding Time (Maximum Allowable Holding Time): The maximum time that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136)

Initial and Continuing Demonstrations of Capability: Procedures to establish the ability of the laboratory to generate acceptable accuracy and precision which is included in many of the EPA's analytical test methods. In general, the procedure includes the addition of a specified concentration of each analyte in each of four separate

aliquots of laboratory pure water or authentic samples. These are carried through the analytical procedure and the percent recovery and the standard deviation are compared to specified limits. (40 CFR Part 136, 2003 NELAC)

Internal Standard: A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

ISO/IEC 17025: The International Organization for Standardization and International Electrotechnical Commission form this specialized system for worldwide standardization. Members of ISO or IEC participate in the development of International Standards through technical committees established by their organization to deal with particular fields of activity. Other international organizations, government and non-government, also take part in development of these standards. The ANSI/ISO/IEC 17025-2005 is approved as an American National Standard and covers general requirements for the competence of testing and calibration laboratories.

Laboratory: A body that calibrates and/or tests.

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.
2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing at or from a permanent location, from a temporary facility, or a mobile facility. (ISO 25)

Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC Quality Systems)

Limit of Detection (LOD): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reliably detected. CFA has established LOD = 2 x MDL.

Limit of Quantitation (LOQ): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reported with a specific level of confidence. The LOQ is set at or above the concentration of the lowest initial calibration standard. The laboratory must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported.

CFA uses the following guidance (LOD < LOQ):

When LOD < PQL, PQL = LOQ

When LOD > PQL, LOQ is raised to next lowest calibration standard

Matrix: The component or substrate that contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- ◊ Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. Includes surface water, groundwater, and effluents.
- ◊ Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- ◊ Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt-water source.
- ◊ 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.

- ◊ Air Samples: Media used to retain the analyte of interest from an air sample such as sorbent tubes, summa canisters, XAD resin or PUFs. Each medium shall be considered as a distinct matrix. (Quality Systems)

Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate 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. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

May: Denotes permitted action, but not required action. (NELAC)

Method Blank (MB): 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 containing an analyte of interest 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. (NELAC)

Method Detection Limit (MDL): The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater that zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: Denotes an item that is required to be met. (Random House College Dictionary)

Negative Control: Measures taken to ensure that a test, its components, or the environment does not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of National Environmental Laboratory Accreditation Program (NELAP). Also known as The NELAC Institute (TNI).

Performance Audit: The routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

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. (NELAC)

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. (NELAC)

Preservation: Refrigeration and or reagents added at the time of sample collection to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

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 extermal source. (NELAC, Section 2.1)

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 in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories. (NELAC)

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed. (EPA-QAD)

Pure Reagent Water: Shall be water in which no target analytes or interferences are present at a concentration that would impact the results when using a particular analytical test method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality within a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the need of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may also be called a Quality Assurance Plan or a Quality Plan. NOTE: The quality manual may call up other documentation relating to the laboratory's quality arrangements. (CFA)

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. (ANSI/ASQC E-41994)

Quantitation Limits: The value at which an instrument can accurately measure an analyte at a specific concentration that includes the maximum or minimum levels, concentrations, or quantities of a target that can be quantified with the accuracy required by the data user. These values establish the upper and lower limits of the calibration range. (NELAC with DoD clarification)

Range: The difference between the minimum and the maximum set of values. (EPA-QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes that have been transcribed verbatim, dated and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 -2.1)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM - 6.08)

Requirement: Denotes mandatory specification; often designated by the term "shall." (NELAC)

Sample: Portion of material collected for chemical analysis, identified by a single, unique term. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis. (DoD)

Selectivity: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (NELAC Quality Systems)

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC Quality Systems)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there will be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: A known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Standard Operating Procedure (SOP): A written document that details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Standard Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method. (NELAC)

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.4)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

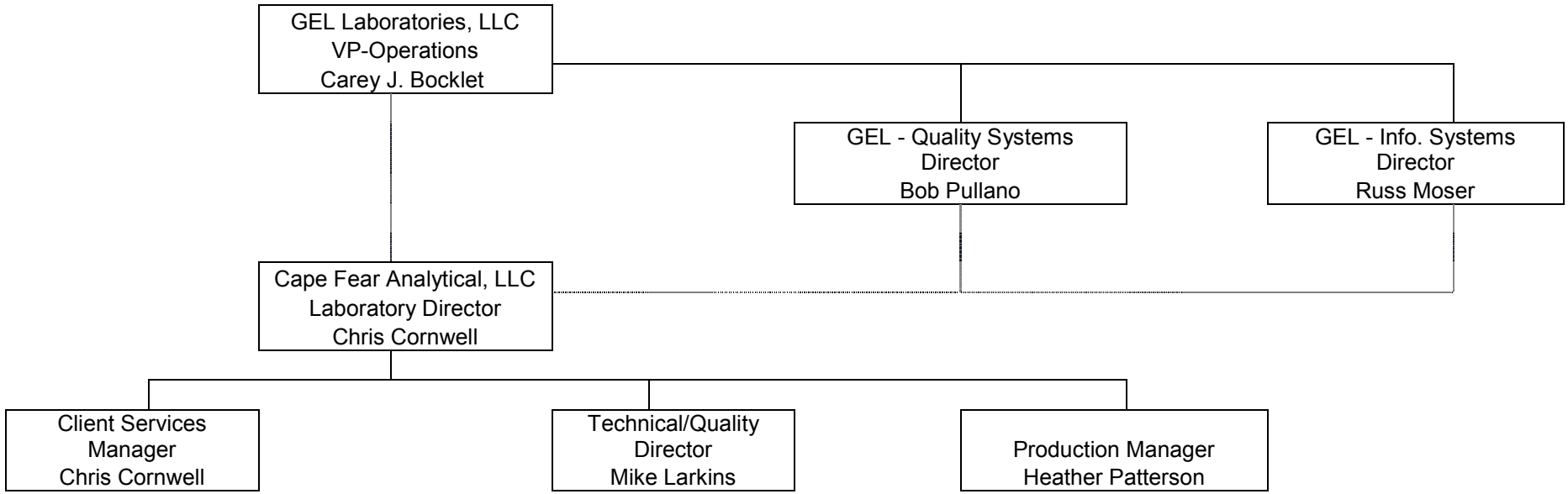
Validation: The process of substantiating specified performance criteria.

Verification: Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: Verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, to repair, to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

APPENDIX C: CORPORATE ORGANIZATION CHART



APPENDIX D: CERTIFICATIONS

Cape Fear Analytical, LLC maintains environmental laboratory certification from NELAP in Utah and secondary in California. We expand our list of certification as needed.

Original Scope of Accreditations is maintained in the Quality Assurance work area. Electronic copies are available in .pdf form on the CFA intranet. *Please call to confirm the status of any certification of interest to you.*

- National Environmental Laboratory Accreditation Program (NELAP) - Primary issued through the State of Utah, Department of Health, Bureau of Laboratory Improvement;
  Secondary NELAP accreditation issued through the States of California, New York and New Jersey

- North Carolina Department of Health and Human Services, North Carolina State Laboratory Public Health Environmental Sciences, Safe Drinking Water.

- Pennsylvania Department of Environmental Protection, Registered to perform analysis on Air samples

APPENDIX E: ESSENTIAL QUALITY CONTROL REQUIREMENTS

At CFA, we enforce strict adherence to quality control measures. Quality control measures for each type of analysis are delineated in the associated standard operating procedure and include those specified in the identified analytical method. Client requests for additional quality control agreed to by us will be communicated to the laboratory by the Project Manager and performed accordingly.

All quality control measures are assessed and evaluated on an ongoing basis. We use these measures to establish statistically derived quality control acceptance criteria. The acceptance criteria are used to evaluate whether the analytical process is in control and to assist us in establishing the validity of the data. Our procedures for handling out- of-control situations are written in the analytical standard operating procedure.

Method-specific quality measures are described in the appropriate standard operating procedure. Essential but general quality control requirements are summarized in the sections below for chemical testing, including organic analyses.

E1 Chemical Testing

This section includes our quality control requirements for organic analyses, and discusses:

- Negative controls
- Positive controls
- Analytical variability and reproducibility
- Method evaluation
- Method detection limits
- Data reduction
- Quality of standards and reagents
- Selectivity
- Constant and consistent test condition

E1.1 Negative controls

We implement a negative control at least once per analytical batch of samples having the same matrix, and where, if applicable, the same extraction or preparation method is employed. The negative control is a method blank that we use to determine the presence of contamination. If discovered, we must investigate the source of contamination and take measures to correct, minimize, or eliminate the source if:

1. The concentration of target analyte exceeds the established practical quantitation limit and exceeds a concentration greater than 1/10 of the measured concentration of any sample in the analytical batch;
2. The concentration of a target analyte in the method blank exceeds that present in the samples and is greater than 1/10 of the specified regulatory limit.

If a method blank is indicative of contamination, we must assess each sample in that batch against the above criteria to determine if the data are acceptable. Any sample associated with a contaminated method blank shall be reprocessed for analysis, as needed, or the results will be reported with appropriate data qualifiers.

E1.2 Positive Control - Method Performance

E1.2.1 Laboratory Control Sample (LCS)

Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicate that the analytical system is “out of control.” Any affected samples associated with an out-of-control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes, as necessary.

Frequency: The LCS is analyzed at a minimum of 1 per preparation batch. Exceptions would be for those

analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

Composition: The LCS is a controlled matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. NOTE: The matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a medium containing known and verified concentrations of analytes such as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the method. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked.

a) For methods that include 1-10 targets, spike all components;
b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;
c) For methods with more than 20 targets, spike at least 16 components.

NOTE: Unless otherwise noted in project quality assurance plans or if components interfere with an accurate assessment, all Department of Defense projects will have LCS, MS, and MSD that contain all target analytes.

Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery. The laboratory shall document the calculation for percent recovery. The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory determines internal criteria or utilizes client specified assessment criteria.

An LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be “out of control” should be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes as necessary.

E1.2.2 Sample Specific Controls

The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge laboratory performance. Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); Post Spike (PS) and Post Spike Duplicate (PSD) sample duplicates; and surrogate spikes.

E1.2.3 Matrix Spike; Matrix Spike Duplicates, Post Spike ; Post Spike Duplicates:

Purpose: Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.

Frequency: The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e. g. Data Quality Objectives) or as specified by the required mandated test method.

Composition: The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period.

a) For methods that include 1-10 targets, spike all components;
b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;
c) For methods with more than 20 targets, spike at least 16 components.

Evaluation Criteria and Corrective Action: The results from matrix spike/matrix spike duplicate and post spike/post spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD).

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. For matrix spike or post spike results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.

E1.2.4 Matrix Duplicates:

Purpose: Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.

Frequency: The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e. g. Data Quality Objectives) or as specified by the mandated test method.

Composition: Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.

Evaluation Criteria and Corrective Action The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another statistical treatment (e. g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments.

Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

E1.2.5 Surrogate Spikes:

Purpose Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample

	preparation/extraction, they provide a measure of recovery for every sample matrix.
Frequency	Except where the matrix precludes its use or when not available, or is not a method requirement, surrogate compounds are added to all samples, standards, and blanks for all appropriate test methods.
Composition:	Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.
Evaluation Criteria and Corrective Action:	The results are compared to the acceptance criteria as published in the mandated test method or determined using statistical process controls (SPC). Where there are no established criteria, the laboratory determines internal criteria and documents the method used to establish the limits.
	Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria include appropriate data qualifiers.

E1.3 Method Evaluation

The following procedures, as described in the other sections of the QAP, are in place in order to ensure the accuracy of the reported result:

- Procedure for initial demonstration of analytical capability performed initially (prior to the analysis of any samples) and if there is a significant change in instrument type, personnel, matrix or test method. Refer to Section 8.
- Procedures for initial and continuing calibration protocols as specified in Section 7.
- Procedures for utilizing proficiency test samples to evaluate the ability of a procedure and/or analyst laboratory to produce accurate data as specified in Section 3.

E1.4 Method Detection Limits

Method detection limits (MDLs) are determined as described in CF-LB-E-001 for The Determination of Method Detection Limits. This procedure is based on that established in 40 CFR Part 136, Appendix B. The laboratory processes for determining Practical Quantitation Limit (PQL), Limit of Detection (LOD) and Limit of Quantitation (LOQ) is also discussed in the SOP CF-LB-E-001.

Where possible, MDL studies are conducted for aqueous, solid and tissue matrices using a clean matrix appropriate to the test method (such as laboratory pure reagent water, Ottawa sand, glass beads or corn oil). MDL studies for the majority of routine parameters are conducted by:

- analyzing a minimum of seven replicates of the lowest calibration standard
- determining the standard deviation of the seven replicates
- multiplying the standard deviation by 3.143 (based on six degrees of freedom and representing a 99% confidence level) to obtain the calculated MDL.

If the MDL study is being conducted for a new method or target analyte, the following steps are taken:

- the MDL is estimated based on information provided in the method or analytical experience
- a standard with a concentration three to five times the estimated MDL is prepared and analyzed a minimum of seven times
- the MDL is calculated as above based on the standard deviation and degrees of freedom
- the MDL is evaluated for reasonableness by verification through analysis of a prepared standard solution two to three times the calculated MDL.

MDL studies are not performed for any target analyte for which spiking solutions are not available .

Practical quantitation limits (PQLs) are determined by either multiplying the MDL by approximately 2 to 10 or are equal to that of the lowest calibration standard. Concentrations of a target analyte determined to be greater than its PQL are defined as quantitative results. All quantitative reported results are preceded by or bracketed by calibration or calibration verification standards.

The LOD is calculated using MDL x 2 = LOD. CFA Laboratories performs quarterly verifications of LODs by spiking a quality system matrix at approximately two to three times the detection limit (for a single-analyte standard) or one to four times the detection limit (for a multi-analyte standard). LOD Verifications are performed on all instruments used to report samples requiring LOD reporting.

The LOD is compared to the PQL and if LOD < PQL, then LOQ = PQL. If LOD > PQL, then LOQ is established at the next lowest level of the calibration curve. CFA Laboratories performs quarterly verifications of LOQs by spiking a quality system matrix at approximately one to two times the claimed LOQ.

The laboratory demonstrates precision and bias at the LOQ by calculating Statistical Process Control (SPC) limits using LOQ measurements, as discussed in SOP CF-QS-E-14, Quality Assurance Measurement Calculations and Processes.

All MDL studies conducted by the laboratory are submitted to the Quality Group for an independent review. Upon acceptance of the MDL study, the MDLs reported to clients via our computer system are updated unless otherwise specified by contract. PQLs are also updated as directed by the new MDLs or changes to procedures.

All data pertaining to the study and the calculation of MDLs is maintained as quality records by the Quality Manager.

E1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, are documented in the individual analytical standard operating procedures. CFA's policy governing the manual integration of chromatographic data is detailed in CF-LB-E-017, Procedure and Policy for Manual Integration. Manual integrations of chromatographic peaks can only be performed in accordance with CF-LB-E-017. This ensures that the integrations are done in a consistent and technically justifiable manner while meeting the requirements set forth under the Good Automated Laboratory Practices.

SOP CF-QS-E-014, Quality Assurance Measurement Calculations and Processes, discusses the use of laboratory data in statistical determinations and includes discussion of Estimation of Total Analytical Uncertainty, Statistical Process Control (SPC) Limits, and Calibration of Instrumentation. Understanding of the procedures used for data generation and reduction is an important part of an analyst demonstrating proficiency in an analytical procedure. All analysts and technicians responsible for generating curves and using curve-generated data are trained to this SOP per CFA annual and interim training requirements.

E1.6 Quality of Standards and Reagents

The quality of standards used in instrument calibration or quality control samples and reagents used in sample preparation and/or analysis must meet the criteria described in Section 7. In methods where the purity is not specified, analytical grade reagents are used. Reagents of lesser purity than those specified by the test method are never used. Upon receipt and prior to use, the labels on the container are checked to verify that the purity of the reagents meets the documented requirements of the particular test method.

The quality of water sources is monitored and documented as described in Section 4. The quality of water used in sample preparation or analysis meets the method-specified requirements. The type of water available in the laboratory is described in Section 4.

E1.7 Selectivity

Absolute and relative retention times aid in the identification of components in chromatographic analyses and in evaluation of the effectiveness of a column in separating constituents. The procedures governing retention time widows are documented in the applicable analytical SOP and meet all regulatory and method requirements.

In addition to retention time windows, the acceptance criteria for mass spectral tuning are also documented in the appropriate analytical SOP. In all cases, the acceptance criteria meet or exceed those specified in the analytical methods.

Unless stipulated in writing by the client, confirmations may be performed to verify the compound identification of positive results detected on a sample. Such confirmations are performed on a second analytical column for organic tests. All confirmation is documented.

E1.8 Constant and Consistent Test Conditions

CFA's implementation of standard operating procedures that specify quality criteria including initial and continuing calibrations assures that our test instruments consistently operate within the specifications required of the application for which the equipment is used.

In addition to the specifications applied to instrumentation, glassware used for sample preparation or analysis is cleaned in a manner that reduces the potential for positive or negative interferences. Glassware is prepared in accordance with CF-LB-E-003 for Glassware Preparation.

This SOP details the procedures used to clean the following groups of glassware:

- Reusable bottles and plasticware
- Glassware used for determination of dioxins/furans or PCB congeners
- Glassware used in the determination of other organic compounds
- Generic glassware used in all other analyses

If the method specifies that the glassware be stored in a particular manner, this requirement is documented in the appropriate analytical SOP.

CAPE FEAR ANALYTICAL, LLC

ETHICS and DATA INTEGRITY AGREEMENT

I. I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Cape Fear Analytical, LLC.

II. I agree that in the performance of my duties at Cape Fear Analytical, LLC:

A. I shall not intentionally report data values that are not the actual values obtained;

B. I shall not intentionally report data that does not meet method or procedural specifications unless that data is properly qualified through comments or other notations in the analytical report.

C. I shall not intentionally report dates and times of data analyses that are not the actual dates and times of data analyses; and,

D. I shall not intentionally represent another individual's work as my own.

III. I agree to inform Cape Fear Analytical, LLC of any accidental or intentional reporting of non-authentic data by myself or by other employees in a timely manner.

IV. I will not knowingly participate in any questionable activities or violations of the Procurement Integrity Act during purchasing or sales activities. I will report any questionable activities to Cape Fear Analytical management. This includes discussions on analytical services, pricing and contracts, vendor pricing or other essential business information to anyone outside of The GEL Group, Inc. family.

This Ethics and Data Integrity Agreement has been explained to me by the Quality Manager and I have been provided the opportunity to ask questions on any part of this agreement that I did not understand. It has also been explained to me that any violation of this agreement conducted during work performed under a subcontract or direct contract to a government agency could subject me to potential prosecution.

I understand that violation of this policy subjects me to disciplinary action, up to and including termination of my employment with The GEL Group, Inc and/or Cape Fear Analytical, LLC.

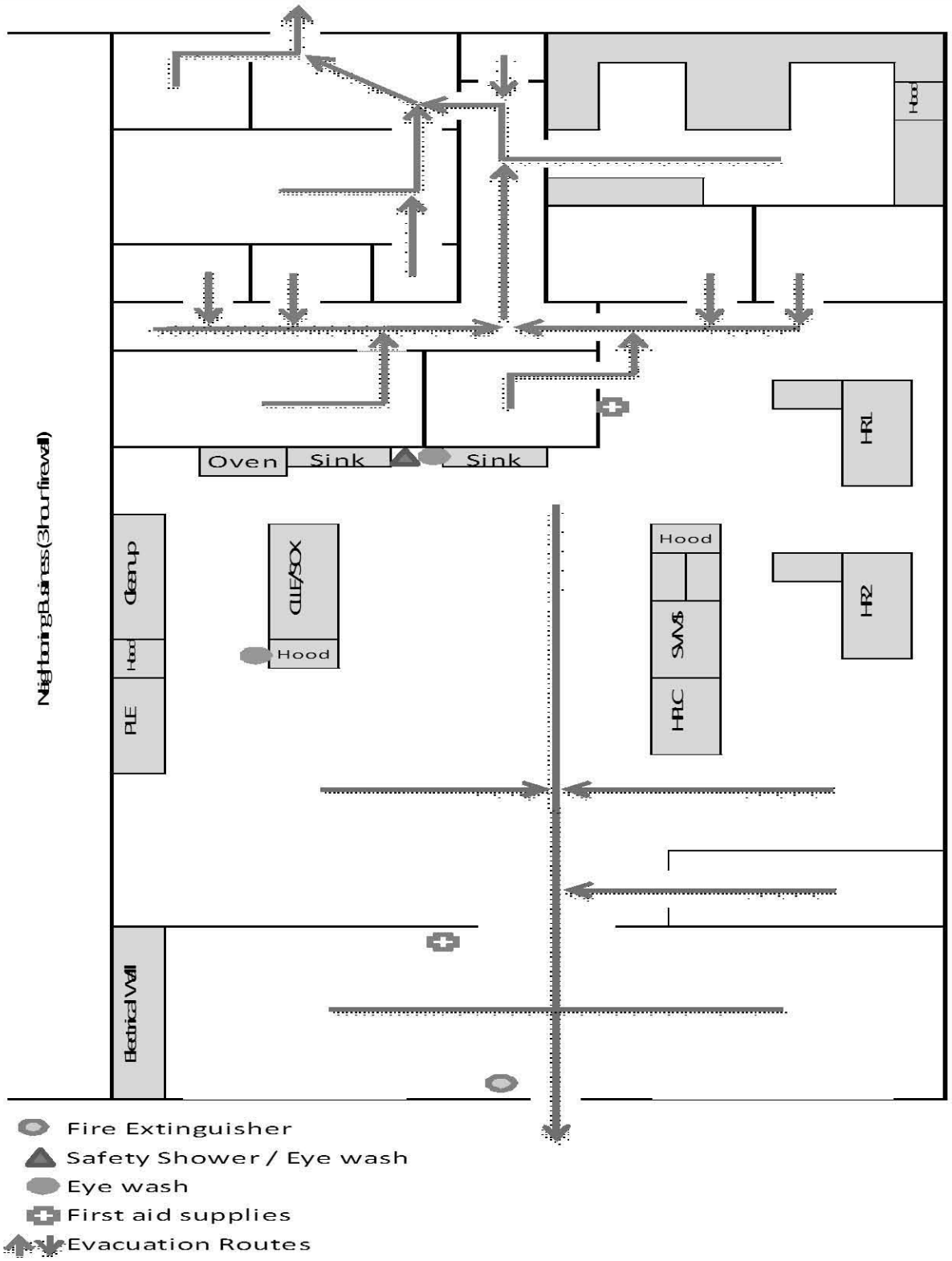
Employee Signature_____ Date:__

Trainer Signature _____ Date:

APPENDIX G: EQUIPMENT LIST

# of Units	Equipment	Model #	Purchase Date	ID/Serial #
2	Waters Autospec Premier High Resolution Mass Spectrometer w/Agilent 7890 Gas Chromatograph w/Leap GC PAL Autosampler	Premier; GC PAL	Jan-09 Jun-09	P750; 161809 P763; 161815
1	Agilent 1100 HPLC	1100	Feb-09	DE23916965; DE14927774; DE13203140; DE21001560; DE14917300; JP40724089
1	Zymark Turbovap	LV	Jan-09	TV9940N9139
1	Powerware Uninterrupted Power Source (UPS)	9390	Feb-09	FX451DAAD1; EX422CAA05
1	Baxter Tempu-Con drying oven	H6620-5A	Mar-09	1089-0265
1	Ohaus Balance - 400 g	Scout Pro SP402	Mar-09	7129320329
1	Ohaus Balance - 4000 g	Scout Pro SP4001	Mar-09	7129370194
4	Kewaunee Fume Hood	H05	Feb-09	Not Listed
1	Chromalox extractions heater/pump/controller	NA	Jan-09	NA
1	Cooling Technology extractions chiller	MPCA-03X	Mar-09	29005-02
1	Cooling Technology HRMS chiller	ICA-12	Apr-09	Not available
26	Heating mantles and controllers	Glas-Col PowerTrol	Feb-09	Various
1	Air compressor	4B233E	Mar-09	L4/7/2008-00469
1	VWR Sonicator	B1500A-DTH	Mar-09	CB0808DUA01271
1	Agilent Technologies 7890 GC-ECD	7890A	Jun-09	ECDCF.I; CN10842125
1	Hewlett Packard GC-MSD with Arcon Autosampler	HP5973/6890	Jun-09	MSDCF.I;US71191100 VOACF.I;US71191095 13332 (Arcon AS)

APPENDIX H: FACILITIES WITH EVACUATION ROUTES



APPENDIX I: STANDARD OPERATING PROCEDURES AND ANALYTICAL METHODS

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
CF-ADM-E-001	Preparation, Authorization, Change, Revision, and Release of SOPs	N/A
CF-CO-E-002	Delegated Authority to Commit the Company	N/A
CF-CO-E-003	Contract Review	N/A
CF-CS-E-002	Internal Review of Contractually Required Quality Criteria for Client Package Delivery	N/A
CF-CS-M-001	Project Management AlphaLIMS Manual	N/A
CF-DC-E-001	Document Control	N/A
CF-FC-E-002	Testing Emergency Eyewash and Shower Equipment	N/A
CF-FC-E-003	Fume Hood Face Velocity Performance Checks	N/A
CF-FC-E-004	Inspection of Fire Extinguishers	N/A
CF-HR-E-002	Employee Training	N/A
CF-IT-E-003	Requirements, Design, Operation, Validation and Removal of Hardware and Software Systems Used by CFA.	N/A
CF-IT-E-005	Requirements, Design, Operation, Validation and Removal of Applications Used by CFA.	N/A
CF-LB-E-001	The Determination of Method Detection Limits	N/A
CF-LB-E-002	Balances	N/A
CF-LB-E-003	Glassware Preparation	N/A
CF-LB-E-004	Temperature Monitoring and Documentation Requirements for Refrigerators, Ovens, Incubators, and Other Similar Devices	N/A
CF-LB-E-005	Data Review and Validation	N/A
CF-LB-E-007	Laboratory Standards Documentation	N/A
CF-LB-E-008	Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices	N/A
CF-LB-E-009	Run Logs	N/A
CF-LB-E-010	Maintenance and Use of Air Displacement Pipets	N/A
CF-LB-E-012	Verifying the Maintenance of Sample Integrity	N/A
CF-LB-E-015	Control of Laboratory Standards	N/A
CF-LB-E-017	Procedure and Policy for Manual Integration	N/A
CF-LB-E-028	Creation and Maintenance of Case Narratives	N/A
CF-LB-E-031	Subsampling and Compositing of Samples	N/A
CF-LB-G-001	Laboratory Waste Management Plan	N/A

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
CF-LB-N-001	Safety, Health and Chemical Hygiene Plan	N/A
CF-OA-E-001	Dioxin/Furan/PCB Congener Sample Processing	SW846 8290A, EPA 1613B, EPA 1668B
CF-OA-E-002	Dioxin/Furan Analysis by HRGC/HRMS	SW846 8290A, EPA 1613B
CF-OA-E-003	PCB Congener Analysis by HRGC/HRMS	EPA 1668B
CF-OA-E-004	Formaldehyde Extraction, Derivitization and Analysis by HPLC	SW846 8315A
CF-OA-E-005	DLM02.1 Sample Processing and Analysis	DLM02.1
CF-OA-E-006	CBC01.1 Sample Processing and Analysis	CBC01.1
CF-OA-E-020	Percent Moisture	N/A
CF-OA-E-065	Reagent and Solvent Screening	N/A
CF-QS-B-001	Quality Assurance Plan	N/A
CF-QS-E-001	Conduct of Quality Audits	N/A
CF-QS-E-002	Conducting Corrective/Preventive Action	N/A
CF-QS-E-003	Conflict of Interest Plan	N/A
CF-QS-E-004	Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items	N/A
CF-QS-E-005	Review of Monitoring Device Logs	N/A
CF-QS-E-007	Thermometer Verification	N/A
CF-QS-E-008	Quality Records Management and Disposition	N/A
CF-QS-E-011	Method Validation and Initial and Continuing Demonstrations of Capability	N/A
CF-QS-E-012	NCR Database Operation	N/A
CF-QS-E-013	Handling of Proficiency Evaluation Samples	N/A
CF-QS-E-014	Quality Assurance Measurement Calculations and Processes	N/A
CF-QS-E-015	The Use of Logos and Describing Accreditation Status	N/A
CF-QS-E-017	Maintaining Technical Training Records	N/A
CF-RC-E-001	Receipt and Inspection of Materials	N/A
CF-SR-E-001	Sample Receipt, Login, and Storage	N/A
CF-SR-E-002	Transportation and Shipping of Samples and Pre-Preserved Sample Containers	N/A

APPENDIX J: SAMPLE STORAGE AND PRESERVATION REQUIREMENTS

Parameter	Container[1]	Preservation	Holding Time[2]	Min. Volume
Organics				
PCB Congeners	Amber G, teflon-lined cap	0 ≤ 6° C; <-10°C for tissues; waters: 80 mg thiosulfate[3]	1 year for extraction; 45 days after extraction for analysis (1668, CBC)	1000 mL / 50 g
Dioxin/Furan	Amber G, teflon-lined cap	0 ≤ 6° C; <-10°C for tissues; waters: 80 mg thiosulfate[3]	30 days (8290) or 1 year (1613,CBC) for extraction; 45 days after extraction for analysis	1000 mL / 50 g
Formaldehyde	Amber G, teflon-lined cap	0 ≤ 6° C	3 days for derivitization/extraction; 3 days after extraction for analysis	1000 mL / 50 g
Volatile Organic Compounds	Teflon lined septum 40ml vial or Encore sampler container	0 ≤ 6° C, zero headspace, liquids may be preserved with HCl	Unpreserved liquids 7 days Preserved liquids 14 days Encore Sample Containers 48 hrs to preservation (frozen)	Liquids 3 x 40mL Solids 3 x 5g

[1] P = Polyethylene; G = Glass
[2] Samples should be analyzed as soon as possible after collection. The holding times listed are maximum times that samples may be held before analysis and be considered valid.
[3] Used only in the presence of residual chlorine.

APPENDIX C- DQO MEETING MINUTES

DQO Meeting Report
For Development of
UFP-SAP for SWMU 11 – Former Building 225

Date of Meeting:	November 4, 2010
Meeting: Location:	Environmental Office, NSA Crane; Crane, IN
Attendees:	Doug Griffin, IDEM Howard Hickey, NAVFAC Midwest Tom Brent, Crane Environmental Ralph Basinski, Tetra Tech Tony Klimek, Tetra Tech Peggy Churchill, Tetra Tech
Report prepared by:	Tony Klimek - 513-557-5057, prepared: November 18, 2010

Tetra Tech presented background information about SWMU 11 and there was a general discussion about the proposed sampling. Some of the specific issues discussed were as follows:

1. Tom Brent located and provided additional photos of the fire and aftermath. He provided pdf copies on a CD.
2. Background documents such as the "Green Water Report" should be attached to the UFP-SAP.
3. Tom Brent also found a drawing of a concrete tank that was used to hold the green water. Surface soil samples will be collected from the discharge area of the tank downstream of the outlet.
4. IDEM was concerned about the 70,000 lbs of PCP that burned in the 1976 fire. *Note: The proposed sampling includes dioxin/furan analysis of surface soil to address this concern.*
5. Groundwater samples can be collected from a geoprobe; temporary wells are not necessary. *Note: Tetra Tech will collect groundwater sample in accordance with Tetra Tech/Crane SOP.*
6. Dioxin/furan samples from the surface soil can be composited by area. They should also be collected from the sediment and from areas downwind of site. It was not possible to determine the wind direction during the fire by photos, but a report noted there was a northeast wind during the fire. Target samples will be collected from unpaved areas northeast and southwest of the site. Background samples would be perpendicular to this axis, but are not necessary at this time. Dioxins can occur naturally; the UFP-SAP should describe how dioxin levels will be evaluated. If dioxin/furans are determined to be a concern in Phase I sampling, background samples will be collected in Phase II.
7. Primarily, surface soil and sediment samples will be collected. Subsurface soil samples will be collected from the UST area only. If present and available, two layers of sediment will be collected; 0 to 6" bgs and 6" to 12" bgs from each sediment sampling location.
8. Comments on sampling program are as follows:

- Building 225 Subslab. Collect only surface soil (below slab and gravel) from under floor slab. *Note: One composite sample from area will be analyzed for dioxin/furans.*
- Ditch. Collect only surface soil from ditch. *Note: One composite sample from ditch will be analyzed for dioxin/furans.*
- Adjacent Areas. Collect only surface soil from adjacent areas. Multiple discrete soil samples will be collected from the grassed areas immediately adjacent to Building 225 (SW, SE, and NE). *Note: One composite sample from each area will be analyzed for dioxin/furans – total of three samples for dioxin/furan analysis.*
- Downwind Areas (New). Composites of surface soil samples from grassed areas NE and SW of the site will be collected and analyzed for dioxin/furans. *Note: Tetra Tech will propose to collect surface soil (0 to 24" bgs) composite samples from two grassed areas NE of the site, and two grassed areas SW of the site - total of four samples for dioxin/furan analysis.*
- UST area. Four surface and subsurface soil samples will be collected from the area. These samples will not be analyzed for dioxin/furans
- Building 2981 Concrete Tank. Two surface soil samples will be collected from the soil downstream of the outlet. *Note: These samples will not be analyzed for dioxin/furans.*
- Drainage channel. Surface sediment samples will be collected from four locations at two depth intervals. If available, sediment samples will be collected from 0 to 6" bgs and 6 to 12" bgs. *Note: The four 0 to 6" bgs sediment samples will be composited, and the four 6" to 12" bgs will be composited, for dioxin/furan analysis - total of two samples for dioxin/furan analysis.*
- Groundwater. Two downgradient (in ditch area northwest of slab) and one upgradient (southeast of slab) groundwater samples will be collected. *Note: The locations of the groundwater samples will be determined by the Tetra Tech Field Operations Leader.*

9. Tom Brent agreed that Tetra Tech could submit the draft UFP-SAP for simultaneous review by both Navy Crane and Navy Chemist.

APPENDIX D- BACKGROUND REPORTS

File Crane

APPENDIX G

"GREEN WATER" REPORT

20 September 1976

Building 225
SWMU 11
CTO 90

DEPARTMENT OF THE NAVY
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA 47522

IN REPLY REFER TO
303-REK:jp
5600/1
20 September 1976

MEMORANDUM

From: 303
To: Distribution List

Subj: "Green Water" Report

Encl: Report Draft

1. Enclosure (1) is the draft of the report for off-station use that I promised. Feel free to comment, edit, castrate, renovate or otherwise alter as you see fit.

2. Send comments to Code 01 for consolidation.

R E Klausmeier

R. E. KLAUSMEIER

Distribution
01
0421
09A
09237 (L. Andrews)
3032

1801-0 ic - Storage of Toxic / Hazardous Materials - Crane

On 13 July fire broke out in Building 225 at the Naval Weapons Support Center, Crane, Indiana. The Center fire department pumped in excess of 600,000 gallons of water onto the blaze which burned out of control for several hours. Building 225 and its contents were destroyed. The building was used for storage of a variety of paints, solvents, dye, wood preservative, ink, gauze and hundreds of other items.

Environmental concern was generated by the observation that runoff water used in fighting the fire was colored a brilliant fluorescent green and was entering an intermittent stream known as Broom Branch. This stream flows into Furst Creek which has as its source Lake Greenwood on this Center. Furst Creek flows off the Center and into White River near Newberry, Indiana (See Figure 1). Broom Branch at that time had little flow other than the water used in fighting the fire. A series of dams (#1, 2 and 3) was constructed to contain the polluted water on the Center (See Figure 2).

On 14 July samples were collected at various points along Broom Branch. The samples were analyzed for the presence of toxic materials. Dead fish were observed in the green water which extended almost down to Dam #2. Analysis confirmed the visual observation that all pollutants were contained above Dam #2 and increased in concentration upstream (See Table 1).

TABLE 1. Data as of 7-14-76 a.m.

	Conc. of Pollutants (mg/l)					
SAMPLE SITE	Zn	Pb	Cu	Na Fluor.		PCP
Above Dam #1	0.69	0.2	0.08	15		>30
Below Railroad	0.08	0	0	30		30
Above Dam #2	0	0	0	0.075		
Above Dam #3	0.01	0	0.01	0		
Below Dam #3	0	0	0.01	0		

Exhaustive analyses failed to reveal any pollutants other than zinc, copper and lead, sodium fluorescein (a green dye) and pentachlorophenol (wood preservative). Of these the metals were confined and the dye reasonably non-toxic leaving pentachlorophenol (PCP) as the primary concern.

Beginning 14 July the water near the building was pumped out and transported to a concrete holding pond. A portion of the stream was treated with HTH (calcium hypochlorite) which decolorized the dye. The HTH treatment was quickly discontinued on the rationale that (a) the residual chlorine was an added toxicant, (b) the reaction products were not known and (c) loss of the green color made it virtually impossible to monitor the location of the polluted water.

Beginning 15 July water from the stream was pumped into fuel storage bladders as a containment procedure. At that time probably less than 100,000 gallons of polluted water was present. On the afternoon of 15 July heavy rains added an estimated 10 to 20 million gallons of water to the watershed. As a result, the stream circumvented Dam #2. Dam #3 was washed away on the morning of 16 July. Visibly green water left the center. It was determined that the visible limit of the dye in the

stream was about 0.2 mg/l. The highest value recorded off the Center was 0.4 mg/l.

The green water moved slowly downstream taking 3 days to reach White River where it was diluted at least 1,000 fold. The waters receded to the point that at 1600 on 17 July flow around Dam #2 was stopped. The water quality at the 3 bridges (Figure 1) and the mouth of Furst Creek was continually monitored. The highest PCP value was <10 ppb recorded at Bridge #2.

Having filled all available fuel bladders, it was decided to release water from the reservoir behind Dam #2 into Broom Branch and control the flow of Furst Creek by manipulating the outflow of Lake Greenwood so that dilution would be sufficient that no green color could be perceived in the water leaving the Center. This procedure was begun at approximately 1300 on 19 July. By both visually and chemically monitoring the water leaving the Station, it was possible to dispose of the water in the reservoir with only one instance being noted where green coloration appeared in the water leaving the Station. At no time during this procedure did the level of PCP in the water leaving the Center exceed 20 ppb. The concentration of PCP in the reservoir above Dam #2 was typically 60-100 ppb.

The contents of the fuel bladders were analyzed and PCP levels from 10-80 ppb found. This water was added to the controlled pump-out without ill effect over a period of several days. The bladders were then reconditioned and returned to stock.

Visual observations revealed a normal aquatic fuana and flora at all points of Furst Creek at all times during discharge of the contaminated water.

At present no polluted water is present in the watershed. A small amount of dye does leach from the railroad bed during heavy rain but no toxicity problems result. A culvert has been installed in Dams #2 and #4 to allow free flow of the stream. Building 225 has been razed and the site cleaned. The residue has been buried in a hydrologically isolated land fill. Several thousand gallons of highly polluted water pumped from the building site are being held in a concrete reservoir awaiting proper disposal. A study is in progress to determine the most cost effective disposal procedure.

FIGURE 1.

Schematic Diagram of Watershed
(Not to scale)

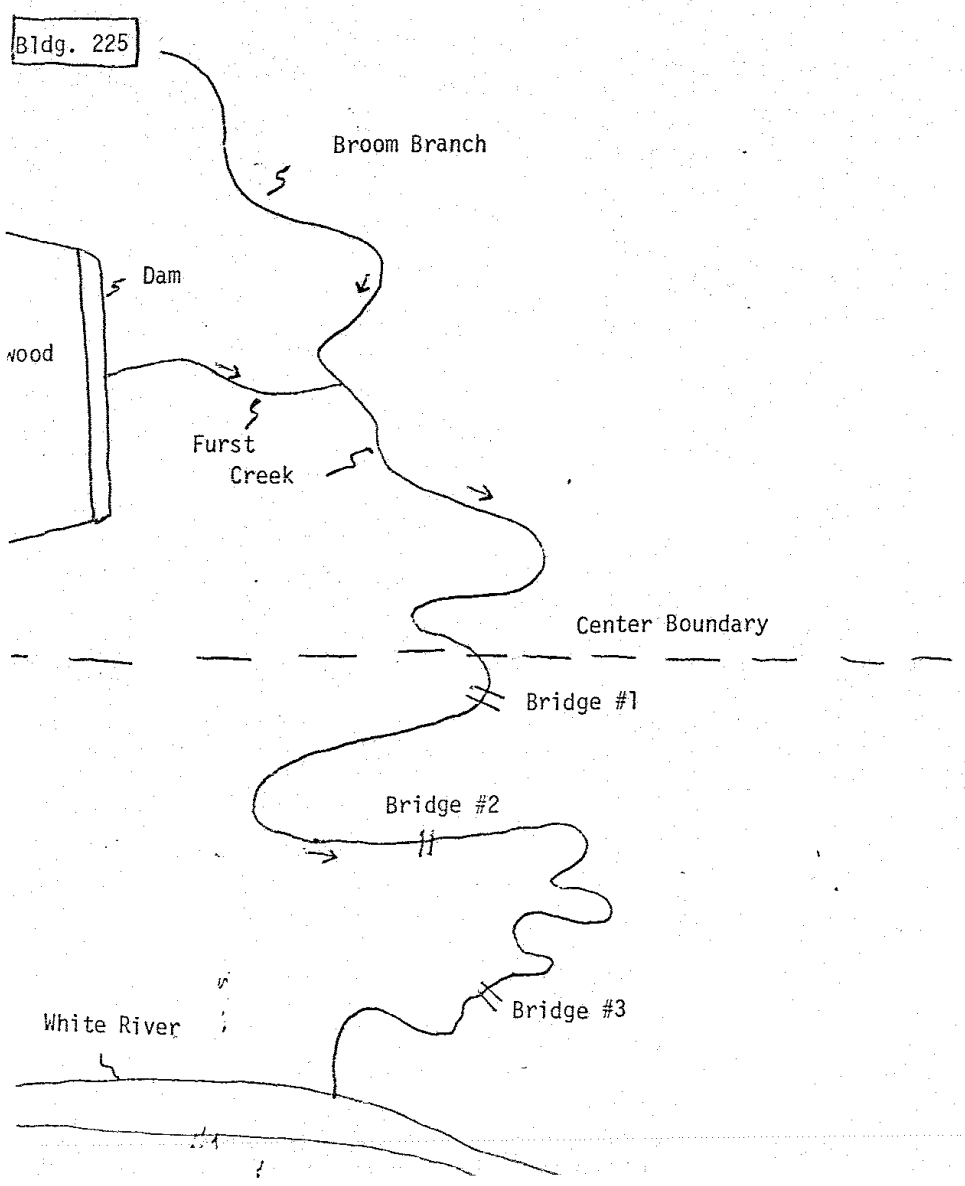
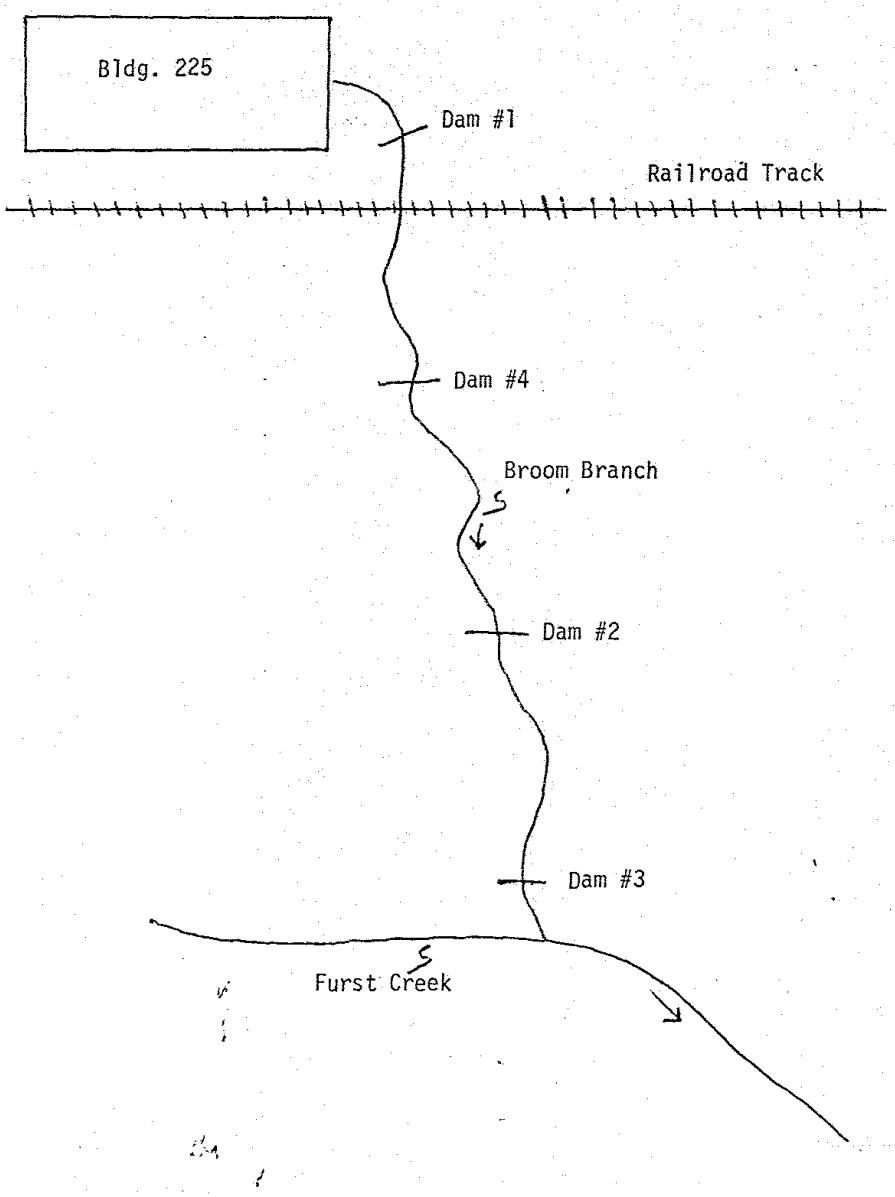


FIGURE 2.

Schematic of Watershed
(Not to scale)



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REPORT OF FIRE INVESTIGATION

BUILDING 225

CRANE NAVAL WEAPONS SUPPORT CENTER

CRANE, INDIANA

14-16 JULY 1976

Enclosure (1)

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REPORT OF FIRE INVESTIGATION
INERT WAREHOUSE #225
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA
14-16 JULY 1976

1. GENERAL

a. Time and Date of Fire: The Fire Department received a telephone call from the base telephone operator at 0655 hours on 13 July 1976.

b. Location of Fire: Bldg. #225, Inert Warehouse, is located on the west side of Highway 101 at the Naval Weapons Support Center.

c. Construction: Bldg. #225 is a one-story 200' - 8" x 50' - 5" structure. Walls are 8" reinforced concrete with metal framed windows and iron doors. The roof is constructed of structural iron trusses covered with corrugated transite forming a high "A". It was ventilated with stack vents at the ridge pole and louvers at each end. A concrete loading platform was on the west side of the building parallel to a railroad siding.

d. Occupancy: Bldg. #225, Inert Warehouse, contained storage of flammable liquids consisting of various amounts of wood preservative, thread, dispersion darex, octadecylamine, tolulene, protective coating adhesive, silicone grease, methyl ethyl ketone, randing seals, adhesive dorax, enamels, paints, lacquers, sodium flueorecinds, parchloroethlene, graphite, trichlorethylene, compound champel, nylon resin, adhesive resin, zinc chloride, stencil paint, tape, paraffin, stannical, devcon, sodium hydroxide, araldite hardener, cellulose nitrate binder, brown wrapping paper, linseed oil, staples, cobalt napthenate, varnish, desicant, primer coating, teflon bottles and caps, solvents, polyurethane foam, gauze, drilling oil, dechlocomethane, plastic adhesive lacquer, molykote, butylacetate, orange shellac, chlorinated rubber, lactite, tricresyl phosphate, xylen, stearic acid, electrial equipment, ceiling compound, styrene monomeric, and pettem cement. It was stored in the following containers: 55-gallon drums, 5-gallon cans, boxes, 50-pound cans, fiber drums, 1-gallon cans, 5-pound jars, 1-pound kits, rolls, pallets, 1-quart cans, bags, 1-pint cans, paper drums, 100-pound bags and 200-pound drums.

e. Exposures: Due to the occupancy of Bldg. #225 exposures existed on the south, west and east sides of the fire building. Refer to paragraph 4.b(4) of this report for complete detailed information on fire exposures.

f. Building services: Unheated building - wiring was rigid conduit, explosion proof fixtures.

g. Weather conditions: Temperature - 58°, humidity - 82%, clear, wind direction - northeast, velocity - seven miles per hour.

2. FIRE PROTECTION FEATURES

a. Water supply and hydrants: An adequate volume of water at sufficient pressure was available from the two fire hydrants near the building for fire fighting purposes.

b. Alarm system: None

c. Portable fire fighting equipment: Ten portable fire extinguishers were located inside of Building 225.

d. Automatic detection and extinguishing devices: None existed in the building. However, automatic sprinkler protection was previously recommended in a fire protection engineering survey report in 1966. This recommendation was incorporated into a FY68 MILCON project but was deleted due to lack of funds.

e. Other: Explosionproof electrical equipment, floor drains, and building construction.

3. FIRE FIGHTING FORCES AVAILABLE

a. Activity Fire Department: Naval Weapons Support Center, Crane has a structural fire department comprised of twenty-seven men; one fire chief, two assistant chiefs, four fire captains, four drivers/operators, twelve fire fighters, one fire inspector supervisor, two fire inspectors and one fire inspector trainee.

The Fire Department responded to the fire with a total of eight men; one captain (acting assistant chief), one captain, two drivers/operators and four fire fighters.

b. Outside Fire Departments: Although mutual aid agreements are in effect, the fire chief did not call for mutual aid due to the distance of travel and the lack of response by volunteer fire fighters. Crane Volunteer Fire Department is five miles from the activity and Owensburg Volunteer Fire Department is eight miles.

c. Other organized forces: The eight-man Naval Weapons Support Center, Crane was supported by the activities auxiliary fire force, various civilian workers and military personnel. Public Works utility personnel responded to the scene and rendered assistance.

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4. STORY OF THE FIRE

a. Circumstances:

(1) Activities prior to the fire: The investigation disclosed that on 30 June 1976 three civilian workers entered Building 225 to remove parachute cords in accordance with base operational procedures. On 7 July 1976, Fire Inspector James Hager was passing Building 225 and noticed the front door open. He took this opportunity to inspect the building. When he went in, he saw one workman from about thirty feet away. He called to the workman and advised him of his intention to inspect the premises. Mr. Hager said he could not identify the workman and did not notice anything out of order. Fire Inspector Hager checked the fire extinguishers and general conditions. Inspector Hager did not see anything unusual during his inspection. This, according to records and witnesses, was the last time anyone was in the building.

(2) Detection and reporting of fire: Mr. Bob Brinson, a car blocker who works in Building 224 which is south of Building 225, stated that at approximately 0650 on Tuesday, 13 July 1976, he noticed smoke coming from the louver of Building 225 and simultaneously he heard what he thought was "small arms fire". He said he went into the office and told his supervisor, Doyle Robertson, that Building 225 was on fire. Mr. Robertson called on the "crash phone" and gave his name and told the operator that Building 225 was on fire and to notify the fire department. Mr. Robertson then said he looked out the window and saw fire coming through the roof on the west wall of Building 225 in about a 10' x 10' area.

At 0655 hours, Fire Captain John Parkes received a call from the operator that Building 225 was on fire and for the fire department to respond.

(3) Attempts to extinguish fire pending arrival of the fire department: None

(4) Unusual conditions leading to or affecting the fire: Hot, humid weather existed in Illinois and Indiana for the previous week prior to the fire. Weather for Crane prior to the fire, up to the day it occurred is as follows:

	Maximum	Minimum	Humidity
Wednesday, 7 July	84°F	60°F	84%
Thursday, 8 July	87°F	72°F	84%
Friday, 9 July	89°F	66°F	86%
Saturday, 10 July	93°F	70°F	87%
Sunday, 11 July	93°F	73°F	76%
Monday, 12 July	92°F	73°F	69%
Tuesday, 13 July	84°F	54°F	82%

Wind direction on 13 July 1976, the day of the fire, was northeast at seven miles per hour.

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(5) Fire spread: The fire extended to grass in the area by flying fifty-five gallon drums and aerosol cans but was quickly extinguished by man power patrolling the area using a 500 GPM, Code 7341, brush attack pumper. Other than that, the fire was confined to the building of origin.

b. Fire fighting operations (See attachment (1) for detailed plan of fire scene.)

(1) Response of personnel and equipment: At 0655 hours, Engine 1, Engine 2 and Captain Charles Hawkins, Acting Assistant Chief, responded to a telephone call from the base operator for a fire in Building 225. The base fire station is 4.2 miles from Building 225. As Acting Assistant Chief Hawkins turned from Highway 5 to Highway 101, a distance of three miles away, he saw a column of smoke in the sky. When he arrived, no one was in the area. He heard no explosions and he observed fire in a small area venting itself through the roof on the west side of Building 225. Engine Company No. 1, commanded by Orval T.Wunder, Fire Captain, arrived right behind the Acting Assistant Chief Hawkins and, as he approached the north side of Building 225, he gave orders to his three-man crew to lay one 2 1/2" line wyed to two 1 1/2" lines and a 2 1/2" line with a constant flow nozzle and proceeded to hydrant 341 on the east side of fire building, east of the open lumber storage. The hose crew then cut the lock on the front iron door for entry but were unable to open the door. They stated "it was hot" and "possibly distorted and it would not open." (NOTE: This was a blessing in disguise, for had the door been open to make entry certainly we would have sustained casualties.) Hose lines were then taken to the west side of the building and directed onto the fire coming through the roof while an alternate way of entry could be found. The fire began to become intense and it was at this point that an attempt to gain entry was abandoned as more explosions were occurring and it was evident that the problem now at hand was to cover the exposures. Subsequently, Engine 1 crew set up a deluge gun at the south east corner of the building fed by two additional 2 1/2" lines supplied from Engine 1. This action was instrumental in cutting down the radiant heat extending to the lumber storage across Highway 101 and in covering the exposure to the south, Building 224, the car blocker shop. The deluge gun was also used to extinguish fire in three wooden utility poles adjacent to the fire building.

Engine 2, commanded by Captain John Parkes, arrived immediately behind Engine 1 with two fire fighters and laid a 2 1/2" line from the west side of the building to hydrant 338. They then hand-stretched a second 2 1/2" line and both lines were positioned on the railroad tracks covering the exposure on the west side: Building 2118, Railroad Repair Storage, Building 2720, Railroad Repair Shop; and Building 2111, Paint Storage Locker. These buildings were one-hundred-and-one feet from the fire building.

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Fire Chief Robert Veth, coming to work for the day shift, arrived at the fire station and was told of the fire at Building 225. He immediately responded and arrived on the fire ground at 0713 hours. Upon his arrival, he found that one-third of the roof was collapsed and fire was "Fireballing" four-hundred feet into the air with fifty-five gallon drums exploding and landing throughout the area. He used a five-hundred GPM pumper to patrol the area to extinguish fires started by these drums and flying fire brands. At 0735 hours, a call was put out for all off-duty Crane fire fighters to respond from their homes. Ten members responded and along with the fire prevention supervisor and inspector, they helped handle hose lines and combat the fire. In all six, 2 1/2" lines were used; two into a deluge gun, three with straight streams and one wyed to two 1 1/2" lines. Several booster lines were used in the area for incipient small fires. The fire was secured at 1114 hours.

(2) Person in direct charge of fire fighting operations: In the early stages of the fire, Captain Charles Hawkins, Acting Assistant Chief, was in command until relieved by Fire Chief Robert Veth at 0713 hours.

(3) Evacuation and/or rescue operations: None

(4) Protection of exposures: The following exposures to Building 225 were:

Exposure No.	Building	Distance	Direction
1	Lumber storage	83'	East
2	Building 224	175'	South
3	Building 2118	101'	West
4	Building 2720	101'	West
5	Building 2111	130'	Northwest

In a normal fire, these buildings would not be considered immediate exposures. However, due to the nature of contents of flammable liquids of Building 225, with the large volume of fire and explosions, these buildings were severely threatened. Two factors in confining this fire to the building of origin were:

(a) Excellent placement of master streams and hose lines; and,

(b) Building 225 - the walls were constructed of 8" concrete poured over reinforcing rods. The strength of these walls absorbed the heat and withheld the force and intensity of the fire channeling it vertically, high into the air, and withholding it from extending horizontally on all sides. Had the building's walls been of any other construction, the results of containing this fire to the building of origin would have been disastrous. (Refer to Attachment 1 for placement of lines to cover exposures.)

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(5) Extinguishing operations: Engine 1 laid a total of four 2 1/2" lines:

One 2 1/2" line straight tip to north side of building.
One 2 1/2" wyed to two 1 1/2" lines to west side of fire.
Two 2 1/2" lines to a deluge gun southeast corner of fire.
Engine 2 laid two 2 1/2" lines - one to east side of fire and one to west side of fire.

(6) Salvage operations: None were performed as all of the contents were destroyed. No overhauling was effected and security and standby lines were left with the fire detail.

(7) Summary of equipment and extinguishing agents used: Fire Department records disclosed that 2,500 feet of 2 1/2" hose, 1,600 feet of $1\frac{1}{2}$" hose discharging a total of 328,000 gallons of water. A deluge gun and various hand tools were also placed in service. It was first thought that AFFF (light water) was used for extinguishment of flammable liquids, however, due to the intensity of the fire and the inability to get close enough because of the radiated heat this idea was abandoned.

(8) Unusual circumstances during fire: The exploding fifty-five gallon drums of various flammable liquids cascading into the air and then exploding all about the area presented a great hazard to all personnel operations at this fire.

5. CAUSE OF FIRE

Deterioration of containers allowed contents to seep out onto cardboard containers and wooden pallets causing spontaneous ignition, Witnesses that discovered the fire and first arriving fire fighters placed the fire at the second doorway, west wall, as illustrated in Attachment 2. Referring to Attachment 3, "Location of Contents", the storage at location 63 was cellulose nitrate binder and brown wrapping paper.

It was also in this area that the interior concrete wall spalled most severely and a "sagging of the metal lintel" over the metal doorway was found. For this metal fatigue to take place, a long period of high temperature had to exist. Further examination of all other lintels did not show this sagging. The one pint and one quart cans in the area where the fire originated were probably "the small arms type fire" reported by the first witness. Arson was ruled out as a possible cause of the fire due to the lack of any hard or significant evidence.

6. LOSS OF LIFE AND PERSONAL INJURY

There was no loss of life or injuries sustained as a result of this fire.

7. PROPERTY LOSS

a. Buildings: For all practical purposes, the building is considered a total loss. Per OPNAVINST 11320.25, the loss is the original plant account value of $41,072.

b. Contents: $300,000

c. Other than Navy property: None

d. Totals:

(1) Navy Property: $341,072

(2) Other than Navy property: None

8. CONCLUSIONS

a. Violation of regulations:

(1) Approximately fifty percent of the material in Building 225 was scheduled for disposal per an inventory check in April 1976. Reportedly, some of the materials could have been in storage for ten years or more. The physical evidence available after the fire indicates severe container deterioration over a long period of time. Obsolete materials should have been disposed of on a routine schedule basis, i.e., quarterly, semi-annually, or yearly, as needed.

(2) Lack of required automatic sprinkler protection resulted in a loss of the entire building and contents.

(3) Unrecorded entrance into the building by a workman six days prior to fire.

b. Failure of equipment: None

c. Delayed alarm: None

d. Evaluation of fire fighting operations: Very good - The ability of the acting assistant chief to realize early in the fire that a fire of major proportions was underway and the fire department's ability to bring to bear the largest size lines and deluge gun, and the placement of these in strategic positions was instrumental in confining the fire to the building of origin.

e. Prevention and/or loss minimization: The size of the fire loss would have been significantly reduced if only useable materials were in storage, and if the building were fully sprinklered.

f. Other conclusions:

(1) In June 1975 a fire occurred in Building 868 due to leaking containers of methyl ethyl ketone peroxide which were stored past their shelf life. Consequently, a serious problem appears to be developing

regarding the storage of outdated hazardous materials. Until a suitable program of prompt disposal of obsolete items can be established and implemented, fires in storage areas can be anticipated periodically at this facility.

(2) This "Inert" Warehouse building contained useable and unuseable storage items consisting of ordinary combustibles (gauze, tape, etc.), flammable liquids, combustible liquids, oils, grease, and caustics. The monetary value in this fire loss would have been drastically reduced under the following conditions:

(a) Storage limited to useable items only.

(b) Physical isolation by way of one-hour rated walls or partitions of hazardous materials (flammable/combustible liquids) from all other storage.

(3) This investigation disclosed many people operating at the fire in shirt sleeves. If a "blast" had occurred, the Navy would have sustained many burn victims. On fires of this type, in the future, all personnel operating within the hazardous area should wear protective clothing.

9. RECOMMENDATIONS

a. Establish and implement a suitable program for the disposal of excess, obsolete, or outdated hazardous materials. The first goal of such a program would be to find any hazardous materials in deteriorating containers and immediately dispose of them.

b. A survey should be conducted by activity personnel of all storage structures to pinpoint the location, quantity, and extent of outdated, obsolete, or excess hazardous materials.

c. Store useable materials in buildings separate from unuseable materials.

d. Purchase a 750/1000 GPM deluge gun with three 2 1/2" intakes and a stacked tip of 1 1/2", 1 3/4", and 2" tip for fire department use.

e. Buildings or structures used for the storage of hazardous materials (flammable/combustible liquids, acids, caustics, chemicals, etc.) must comply with the requirements of NAVSUP Publications 284 and 284.1 and NFPA Standard No. 30. These codes include criteria for sprinkler protection, segregation of materials (flammable, acids, caustics, chemicals, etc.), and appropriate storage arrangements.

f. Reevaluate base procedures for access to secured buildings and update if needed.

Charles B. Gindele

CHARLES B. GINDELE
Area Fire Marshal

Joseph R. Condlin

JOSEPH R. CONDLIN
Fire Protection Engineer

(7)

LOCATION	CONTENTS	QUANTITY	Container
51-1	Wood preservative	2172 lbs.	55 gal. drums
51-2	" "	"	"
51-3	" "	"	"
51-4	" "	"	"
51-7	" "	"	"
51-8	" "	"	"
51-9	" "	"	"
51-10	" "	"	"
52-1	" "	"	"
52-2	" "	"	"
52-3	" "	"	"
52-4	" "	"	"
52-7	" "	"	"
52-8	" "	"	"
52-9	" "	"	"
52-10	" "	"	"
53-1	" "	"	"
53-2	" "	"	"
53-3	" "	"	"
53-4	" "	"	"
53-7	" "	"	"
53-8	" "	"	"
53-9	" "	"	"
53-10	" "	"	"

Attachment (3)

LOCATION	CONTENTS	QUANTITY	CONTAINER
54-1	Empty	-	-
54-2	Empty	-	-
54-3	Acetone	750 gals.	5 gal. cans
54-4	Empty	-	-
54-7	Wood Preservative	2,500 lbs.	55 gal. drums
54-8	Wood Preservative	2,500 lbs.	55 gal. drums
54-9	Wood Preservative	2,500 lbs.	55 gal. drums
54-10	Wood Preservative	2,500 lbs.	55 gal. drums
55-1	Aisle space	-	-
55-2	"	-	-
55-3	"	-	-
55-4	"	-	-
55-7	Thread	1,000 spools	Boxes
55-8	Thread	1,000 spools	Boxes
55-9	Dispersion darex Octadecylamine, practical	75 gals. 185 lbs.	5 gal. cans boxes
55-10	Dispersion darex	75 gals.	5 gal. cans
56-1	Aisle space	-	-
56-2	Aisle space	-	-
56-3	Aisle space	-	-
56-4	Aisle space	-	-
56-7	Thread	5,841 spools	Boxes
56-8	Tolulene	875 gals.	55 gal. drums
56-9	Protective Coating Adhesive	250 gals.	5 gal. cans
56-10	"	"	"

LOCATION	CONTENTS	QUANTITY	CONTAINER
57-1	Silicone Grease	2100 lbs.	50 lb. cans
57-2	Silicone Grease	2100 lbs.	50 lb. cans
57-3	Silicone Grease	2150 lbs.	50 lb. cans
57-4	Silicone Grease	2150 lbs.	50 lb. cans
57-7	Methyl Ethyl Ketone	240 gals.	5 gal. cans
57-8	Banding Seals	22,500	Boxes
57-9	Methyl Ethyl Ketone	555 gals.	55 gal. drums
57-10	Methyl Ethyl Ketone	500 gals.	55 gal. drums
58-1	Silicone Grease	1300 lbs.	50 lb. cans
58-2	Silicone Grease	1300 lbs.	50 lb. cans
58-3	Silicone Grease	1400 lbs.	50 lb. cans
58-4	Adhesive Dorax Blue enamel	102 lbs. 530 gals.	5 gal. cans 5 gal. cans
58-7	Plastic Molding	125 lbs.	Fiber drum
58-8	Sodium Flueoreceinds Installation Instruction sheets	480 lbs. 84,925	100 lb. fiber dr Box
58-9	Parchloroethlene	495 gals.	5 gal. cans
58-10	Parchloroethlene	495 gals.	5 gal. cans
59-1	Silicone Grease	1100 lbs.	50 lb. cans
59-2	Silicone Grease	1100 lbs.	50 lb. cans
59-3	Silicone Grease	1150 lbs.	50 lb. cans
59-4	Silicone Grease	1150 lbs.	50 lb. cans
59-7	Graphite	575 lbs.	Fiber drums
59-8	Protective Coating Adhesive Trichlorethylene	205 gals. 140 gals.	5 gal. cans 5 gal. cans
59-9	Compound Champel	1000 lbs.	5 gal. cans
59-10	Nylon Resin Adhesive Resin	100 gals. 200 gals.	55 gal. drums 55 gal. cans

LOCATION	CONTENTS	QUANTITY	CONTAINER
60-1	Blue enamel	166 gals.	1 gal. cans
60-2	Blue enamel	166 gals.	1 gal. cans
60-3	Blue enamel	166 gals.	1 gal. cans
60-4	Grease	200 lbs.	50 lb. cans
	Zinc Chloride	90 lbs.	5 lb. jars
60-7	Yellow stencil paint	15 gals.	1 gal. cans
	Tape	129 rolls	Box
	Wax paraffin	200 lbs.	Box
60-8	Empty	-	-
60-9	Empty	-	-
60-10	Empty	-	-
61-1	Empty	-	-
61-2	Empty	-	-
61-3	Stannical	90 gals.	5 gal. ca
61-4	Yellow enamel	160 gals.	5 gal. ca
61-7	Devcon	236 lbs.	1 lb. kits
61-8	Enamel	55 gals.	5 gal. car
61-9	Trichlorethylene	100 gals.	5 gal. car
61-10	Trichlorethylene	100 gals.	5 gal. car
62-1	Red enamel	68 gals.	1 gal. car
62-2	Red enamel	69 gals.	1 gal. car
62-3	Red enamel	69 gals.	1 gal. car
62-4	Red enamel	69 gals.	1 gal. car
62-7	Aluminum enamel	120 gals.	5 gal. can
62-8	Sodium Hydroxide	1600 lbs.	Boxes
62-9	Araldite Hardener	1350 lbs.	55 gal. dr
62-10	Empty	-	-

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LOCATION	CONTENTS	QUANTITY	CONTAIN
63-1	Cellulose Nitrate Binder	3525 lbs.	Boxes
63-2	Cellulose Nitrate Binder	3525 lbs.	Boxes
63-3	Cellulose Nitrate Binder	3525 lbs.	Boxes
63-4	Brown wrapping paper	5 rolls	Banded
63-7	Araldite hardener Enamel	900 lbs. 198 gals.	55 gal. 5 gal. c
63-8	Linseed Oil	1030 gals.	5 gal. c
63-9	Araldite	5000 lbs.	55 gal.
63-10	Araldite	5000 lbs.	55 gal.
64-1	Aisle space	-	-
64-2	Aisle space	-	-
64-3	Aisle space	-	-
64-4	Aisle space	-	-
64-7	Enamel	85 gals.	1 gal. ca
64-8	Desicant	9830 lbs.	Fiber dru
64-9	Desicant	9830 lbs.	Fiber dru
64-10	Desicant	9840 lbs.	Fiber dru
65-1	Black enamel	150 gals.	5 gal. ca
65-2	Black enamel	155 gals.	5 gal. ca
65-3	Yellow stencil ink	500 tubes	Boxes
65-4	Yellow stencil ink	500 tubes	Boxes
65-7	Wax paraffin	100 lbs.	Box
65-8	Staples	1650 bxs.	Pallet
65-9	Dope	110 gals.	5 gal. ca
65-10	Cobalt Napthenate	5545 lbs.	55 gal. d

LOCATION	CONTENTS	QUANTITY	CONTAINER
66-1	Adhesive	30 gals.	1 gal. cans
66-2	Adhesive	30 gals.	1 gal. cans
66-3	Adhesive	40 gals.	1 gal. cans
66-4	Yellow stencil ink	500 tubes	Boxes
66-7	Enamel	200 gals.	5 gal. cans
	Staples	1000	Boxes
66-8	Varnish	316 qts.	1 qt. cans
	Electrical tape	1117 rolls	Boxes
66-9	Tape	504 rolls	Boxes
	Insulation tape	1449 rolls	Boxes
	Cloth tape	32 rolls	Boxes
66-10	Tape	96,177 yrds.	Boxes
67-1	Desicant	1,200 bags	Fiber drums
67-2	Desicant	1,200 bags	Fiber drums
67-3	Desicant	1,200 bags	Fiber drums
67-4	Desicant	1,200 bags	Fiber drums
67-7	Sodium Flueoreceinds	500 lbs.	100 lb. fiber drum
	Primer coating	10 gals.	1 gal. cans
	Wax	150 lbs.	Bags
67-8	Teflon bottles and caps	11,000	Boxes
	Varnish	327 gals.	1 gal. cans
	Tape	20 rolls	Box
67-9	Tape	400 rolls	Boxes
67-10	Tape	900 rolls	Boxes
68-1	Solvent	15 gals.	5 gal. cans
68-2	Adhesive	100 gals.	1 gal. cans
68-3	Adhesive	100 gals.	1 gal. cans
68-4	Adhesive	100 gals.	1 gal. cans

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LOCATION	CONTENTS	QUANTITY	CONTAINER
68-7	Brown enamel	250 gals.	5 gal. cans
	Graphite	56 lbs.	Drum
68-8	Brown enamel	250 gals.	5 gal. cans
	Pallet edge protectors	61	Box
68-9	Polyurethane foam	1500 gals.	55 gal. drums
68-10	Polyurethane foam	1500 gals.	55 gal. drums
69-1	Gauze	50,000 ft.	Boxes
69-2	Gauze	50,000 ft.	Boxes
69-3	Gauze	50,000 ft.	Boxes
	Drilling oil	15 pts.	1 pt. cans
69-4	Dechlocomethane	150 gals.	5 gal. cans
	Plastic adhesive lacquer	29 gals.	1 gal. cans
69-7	Desicant	4,800 bags	Fiber drums
	Gauze	3,320 ft.	Boxes
69-8	Yellow enamel	43 gals.	1 gal. cans
69-9	Yellow enamel	25 gals.	1 gal. cans
69-10	Molykote	100 gals.	5 gal. cans
70-1	Gauze	37,500 ft.	Boxes
70-2	Gauze	37,500 ft.	Boxes
70-3	Gauze	37,500 ft.	Boxes
70-4	Gauze	37,500 ft.	Boxes
70-7	Wood preservatives	530 gals.	55 gal. drums
70-8	Wood preservatives	530 gals.	55 gal. drums
70-9	Wood preservatives	530 gals.	55 gal. drums
70-10	Wood preservatives	525 gals.	55 gal. drums
71-1	Lacquer	100 gals.	5 gal. cans
71-2	Lacquer	100 gals.	5 gal. cans
71-3	Lacquer	100 gals.	5 gal. cans
71-4	Lacquer	100 gals.	5 gal. cans

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LOCATION	CONTENTS	QUANTITY	CONTAINER
71-7	Wood preservatives	750 gals.	55 gal. drums
71-8	Wood preservatives	750 gals.	55 gal. drums
71-9	Wood preservatives	750 gals.	55 gal. drums
71-10	Wood preservatives	750 gals.	55 gal. drums
72-1	Lacquer	140 gals.	5 gal. cans
72-2	Lacquer	130 gals.	5 gal. cans
72-3	Lacquer	130 gals.	5 gal. cans
72-4	Butylacetate	85 gals.	5 gal. cans
	Orange shellac	315 lbs.	Fiber drums
	Chlorinated rubber	30 lbs.	Paper drums
	Graphite	91 lbs.	Paper drums
	Pressure tape	3441 rolls	Box
72-7	Lactite	378 gals.	1 gal. cans
	Pressure tape	2860 rolls	Box
72-8	Lacquer	37 gals.	1 gal. cans
	Star varnish	10 gals.	1 gal. cans
72-9	Graphite	2300 lbs.	Paper drums
72-10	Lead Car Seals	53,000	Boxes
73-1	Lacquer	100 gals.	5 gal. cans
73-2	Lacquer	100 gals.	5 gal. cans
73-3	Lacquer	100 gals.	5 gal. cans
73-4	Lacquer	100 gals.	5 gal. cans
73-7	Varnish	315 gals.	5 gal. cans
	Tricresyl Phosphate	10 lbs.	5 lb. cans
73-8	Desicant	2,804 bags	Fiber drums
	Xylen, Technical	25 gals.	5 gal. cans
73-9	Tolulene	400 gals.	55 gal. drums
73-10	Tolulene	400 gals.	55 gal. drums

LOCATION	CONTENTS	QUANTITY	CONTAINER
74-1	Stearic acid	6,000 lbs.	100 lb. bags
74-2	Stearic acid	6,000 lbs.	100 lb. bags
74-3	Stearic acid	6,000 lbs.	100 lb. bags
74-4	Stearic acid	6,000 lbs.	100 lb. bags
74-7	Desicant	18,655 bags	Fiber drums
74-8	Desicant	18,655 bags	Fiber drums
74-9	Desicant	18,655 bags	Fiber drums
74-10	Desicant	18,665 bags	Fiber drums
75-1	Aisle space	-	-
75-2	Aisle space	-	-
75-3	Aisle space	-	-
75-4	Aisle space	-	-
75-7	Orange shellac Wax paraffin	400 lbs. 170 lbs.	Fiber drums Fiber drums
75-8	Chlorinated rubber	4,516 lbs.	Fiber drums
75-9	Empty	-	-
75-10	Empty	-	-
76-1	Miscellaneous electrical equip.	-	-
76-2	Miscellaneous electrical equip.	-	-
76-3	Miscellaneous electrical equip.	-	-
76-4	Miscellaneous electrical equip.	-	-
76-7	Empty	-	-
76-8	Empty	-	-
76-9	Empty	-	-
76-10	Chlorinated rubber Wax paraffin	80 lbs. 100 lbs.	Fiber drum Box

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LOCATION	CONTENTS	QUANTITY	CONTAINER
77-1	Misc. Elect. Equip.	-	-
77-2	Misc. Elect. Equip.	-	-
77-3	Misc. Elect. Equip.	-	-
77-4	Lacquer	128 gal.	1 gal. can
	Placards	39,980	Boxes
77-7	Brown enamel	175 gals.	5 gal. cans
	Aluminum enamel	100 gals.	5 gal. cans
77-8	Pressure tape	3,000 rolls	Boxes
77-9	Electrical tape	920 rolls	Boxes
77-10	Lacquer	30 gals.	5 gal. cans
78-1	Storage shelves	-	-
78-2	Storage shelves	-	-
78-3	Storage shelves	-	-
78-4	Storage shelves	-	-
78-7	Thread	650 spools	Boxes
78-8	Thread	778 spools	Boxes
78-9	Thread	1,029 spools	Boxes
78-10	Enamel	130 gals.	5 gal. cans
79-1	Empty	-	-
79-2	Empty	-	-
79-3	Empty	-	-
79-4	Empty	-	-
79-7	Ceiling compound	16 gals.	1 gal. cans
	Primer	9 gals.	1 gal. cans
	Styrene monomeric	279 lbs.	1 gal. cans.
	Lacquer	204 gals.	1 gal. cans
	Araldite hardener	91 lbs.	5 gal. cans
	Tape	52 rolls	Boxes

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LOCATION	CONTENTS	QUANTITY	CONTAINER
79-8	Lacquer	160 gals.	5 gal. cans
	Tricryesyl	8 gals.	1 gal. cans
79-9	Solvent	370 gals.	5 gal. cans
	Dispersion Darex	75 gals.	5 gal. cans
	Lacquer	50 gals.	5 gal. cans
79-10	Solvent	370 gals.	5 gal. cans
	Lacquer	44 gals.	1 gal. cans
80-1	Empty	-	-
80-2	Empty	-	-
80-3	Orange paint	35 gals.	1 gal. cans
80-4	Pettmen cement	6 gals.	1 gal. cans
80-7	Empty	-	-
80-8	Empty	-	-
80-9	Graphite	4,400 lbs.	200lb. drums
80-10	Blue enamel	130 gals.	5 gal. cans

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FLOOR PLAN

Scale 1/16" = 1'-0"

APPENDIX 1

Attachment (2)

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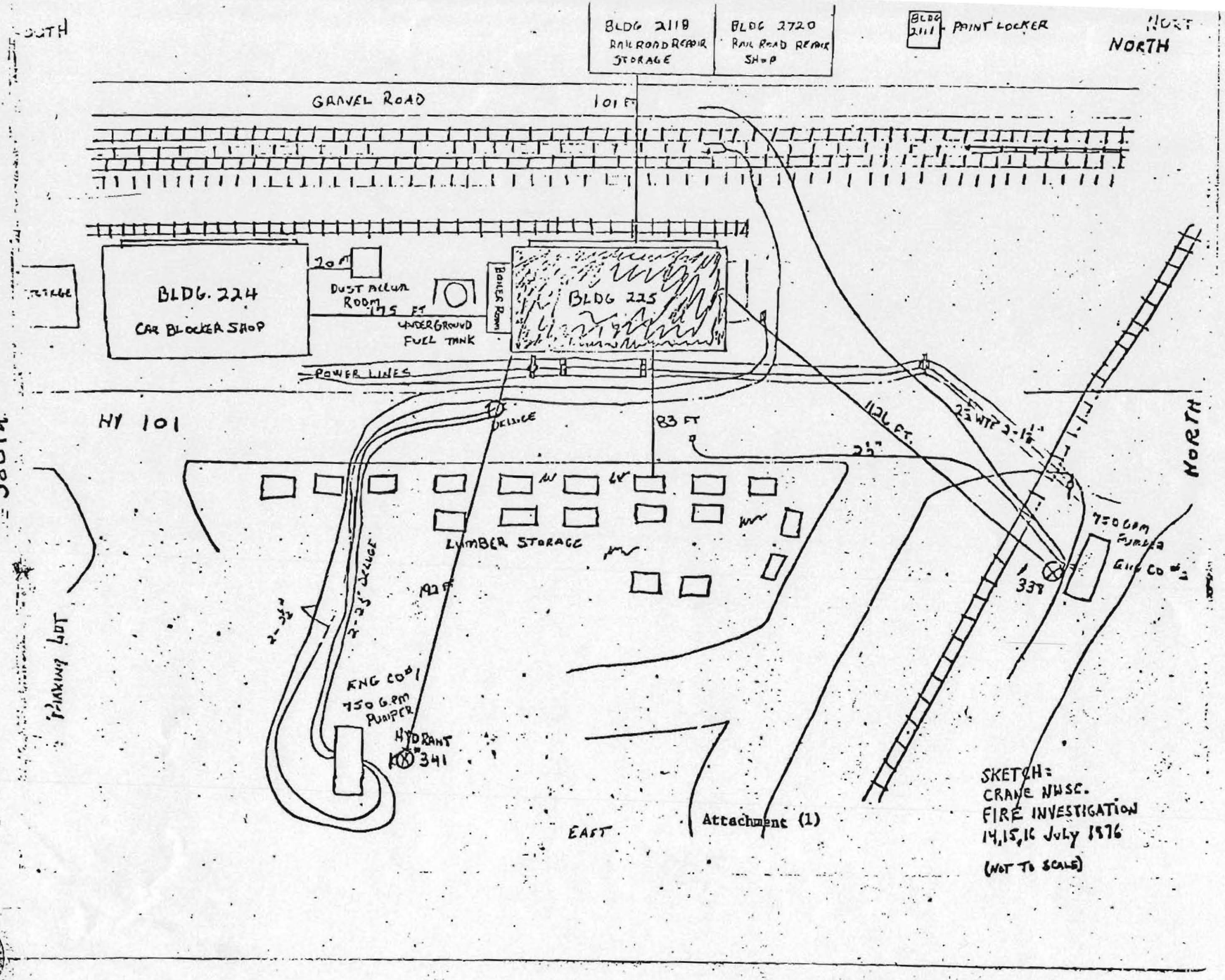

APPENDIX E

PROJECT SCREENING LEVEL BACKUP DOCUMENTATION

NSA Crane SWMU 11 Human Health Screening Criteria - Surface and Subsurface Soil Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	EPA Regional Screening Level, Migration to Groundwater [1] (mg/kg)	Adjusted EPA Regional Screening Level, Migration to Groundwater [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]			Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]		
						Residential Direct Contact	Migration to Groundwater	Residential Default Closure Level				Industrial Direct Contact	Migration to Groundwater	Industrial Default Closure Level
Volatile Organic Compounds														
1,1,1-Trichloroethane	71-55-6	8700 N	870 N	3.2	64	5000	1.9	1.9	1.9	IDEM-RDCL	38000 N	6700	280	280
1,1,2,2-Tetrachloroethane	79-34-5	0.56 C	0.56 C	0.000026	0.00052	5	0.007	0.007	0.00052	RBSSL	2.8 C	8.7	0.11	0.11
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 11	76-13-1	43000 N	4300 N	150	3000	NA	NA	NA	3000	RBSSL	180000 N	NA	NA	NA
1,1,2-Trichloroethane	79-00-5	1.1 C	1.1 C	0.000078	0.0016	9.4	0.03	0.03	0.0016	RBSSL	5.3 C	15	0.3	0.3
1,1-Dichloroethane	75-34-3	3.3 C	3.3 C	0.00069	0.014	1300	5.6	5.6	0.014	RBSSL	17 C	1700	58	58
1,1-Dichloroethene	75-35-4	240 N	24 N	0.12	2.4	310	0.058	0.058	0.058	IDEM-RDCL	1100 N	410	42	42
1,2,3- Trichlorobenzene	87-61-6	49 N	4.9 N	0.087	1.7	NA	NA	NA	1.7	RBSSL	490 N	NA	NA	NA
1,2,4-Trichlorobenzene	120-82-1	62 N[4]	6.2 N[4]	0.0068	0.14	1800	5.3	5.3	0.14	RBSSL	270 N[4]	4900	77	77
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.0054 C	0.0054 C	0.00000014	0.0000028	NA	NA	NA	0.0000028	RBSSL	0.069 C	NA	NA	NA
1,2-Dibromoethane (EDB)	106-93-4	0.034 C	0.034 C	0.0000018	0.000036	0.3	0.00034	0.00034	0.000036	RBSSL	0.17 C	0.49	0.0096	0.0096
1,2-Dichlorobenzene	95-50-1	1900 N	190 N	0.36	7.2	2800	17	17	7.2	RBSSL	9800 N	3900	270	220
1,2-Dichloroethane	107-06-2	0.43 C	0.43 C	0.000042	0.00084	3.7	0.024	0.024	0.00084	RBSSL	2.2 C	5.8	0.15	0.15
1,2-Dichloropropane	78-87-5	0.89 C	0.89 C	0.00013	0.0026	4.5	0.03	0.03	0.0026	RBSSL	4.5 C	7.2	0.25	0.25
1,3-Dichlorobenzene	541-73-1	NA	NA	NA	NA	420	2.3	2.3	2.3	IDEM-RDCL	NA	890	8.9	8.9
1,4-Dichlorobenzene	106-46-7	2.4 C	2.4 C	0.00041	0.0082	42	2.2	2.2	0.0082	RBSSL	12 C	73	3.4	3.4
1,4-Dioxane	123-91-1	4.9 C	4.9 C	0.00014	0.0028	NA	NA	NA	0.0028	RBSSL	17 C	NA	NA	NA
2-Butanone (MEK)	78-93-3	28000 N	2800 N	1.5	30	44000	35	35	30	RBSSL	200000 N	70000	250	250
2-Hexanone	591-78-6	210 N	21 N	0.011	0.22	NA	NA	NA	0.22	RBSSL	1400 N	NA	NA	NA
4-Methyl-2-pentanone (MIBK)	108-10-1	5300 N	530 N	0.45	9.0	12000	20	20	9.0	RBSSL	53000 N	29000	75	75
Acetone	67-64-1	61000 N	6100 N	4.5	90	35000	28	28	28	IDEM-RDCL	630000 N	51000	370	370
Benzene	71-43-2	1.1 C	1.1 C	0.00021	0.0042	8.4	0.034	0.034	0.0042	RBSSL	5.4 C	14	0.35	0.35
Bromochloromethane	74-97-5	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Bromodichloromethane	75-27-4	0.27 C	0.27 C	0.000032	0.00064	10	0.51	0.51	0.00064	RBSSL	1.4 C	17	0.51	0.51
Bromoform	75-25-2	61 C	61 C	0.0023	0.046	280	0.6	0.6	0.046	RBSSL	220 C	580	2.7	2.7
Bromomethane	74-83-9	7.3 N	0.73 N	0.0022	0.044	9.9	0.052	0.052	0.044	RBSSL	32 N	13	0.7	0.7
Carbon disulfide	75-15-0	820 N	82 N	0.31	6.2	900	10	10	6.2	RBSSL	3700 N	1200	82	82
Carbon tetrachloride	56-23-5	0.61 C	0.61 C	0.00017	0.0034	3.3	0.066	0.066	0.0034	RBSSL	3.0 C	5.2	0.29	0.29
Chlorobenzene	108-90-7	290 N	29 N	0.062	1.2	380	1.3	1.3	1.2	RBSSL	1400 N	510	27	27
Chloroethane	75-00-3	15000 N	1500 N	5.9	118	80	0.65	0.65	0.65	IDEM-RDCL	61000 N	120	10	10
Chloroform	67-66-3	0.29 C	0.29 C	0.000053	0.0011	3	0.47	0.47	0.0011	RBSSL	1.5 C	4.7	6	4.7
Chloromethane	74-87-3	120 N	12 N	0.049	0.98	NA	NA	NA	0.98	RBSSL	500 N	NA	NA	NA
cis-1,2-Dichloroethene	156-59-2	160 N	16 N	0.021	0.42	110	0.4	0.4	0.4	IDEM-RDCL	10000 N	140	5.8	5.8
cis-1,3-Dichloropropene	10061-01-5	1.7 C[5]	1.7 C[5]	0.00015 [5]	0.0030 [5]	NA	NA	NA	0.0030	RBSSL	8.1 C[5]	NA	NA	NA
Cyclohexane	110-82-7	7000 N	700 N	13	260	7200	330	69	69	IDEM-RDCL	29000 N	9300	1400	69
Dibromochloromethane	124-48-1	0.68 C	0.68 C	0.000039	0.00078	NA	NA	NA	0.00078	RBSSL	3.3 C	NA	NA	NA
Dichlorodifluoromethane	75-71-8	180 N	18 N	0.61	12.2	NA	NA	NA	12.2	RBSSL	780 N	NA	NA	NA
Ethylbenzene	100-41-4	5.4 C	5.4 C	0.0017	0.034	4600	13	13	0.034	RBSSL	27 C	6800	200	160
Isopropylbenzene	98-82-8	2100 N	210 N	1.1	22	1400	11	11	11	IDEM-RDCL	11000 N	1900	140	42
m,p-Xylenes	179601-23-1	620 N[6]	62 N[6]	0.20 [6]	4.0 [6]	NA	NA	NA	4.0	RBSSL	2700 [6]	NA	NA	NA
Methyl acetate	79-20-9	78000 N	7800 N	7.50	150	NA	NA	NA	150	RBSSL	1000000 N	NA	NA	NA
Methylcyclohexane	108-87-2	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Methylene chloride	75-09-2	11 C	11 C	0.0012	0.024	120	0.023	0.023	0.023	IDEM-RDCL	53 C	200	1.8	1.8
Methyl-tert-butyl ether	1634-04-4	43 C	43 C	0.0028	0.056	350	0.18	0.18	0.056	RBSSL	220 C	650	3.2	3.2
o-Xylene	95-47-6	3800 N	380 N	1.2	24	NA	NA	NA	24	RBSSL	19000 N	NA	NA	NA
Styrene	100-42-5	6300 N	630 N	1.8	36	11000	3.5	3.5	3.5	IDEM-RDCL	36000 N	16000	720	550
Tetrachloroethene	127-18-4	0.55 C	0.55 C	0.000049	0.00098	9.9	0.058	0.058	0.00098	RBSSL	2.6 C	16	0.64	0.64
Toluene	108-88-3	5000 N	500 N	1.6	32	8800	12	12	12	IDEM-RDCL	45000 N	16000	96	96
trans-1,2-Dichloroethene	156-60-5	150 N	15 N	0.031	0.62	180	0.68	0.68	0.62	RBSSL	690 N	230	14	14
trans-1,3-Dichloropropene	10061-02-6	1.7 C[5]	1.7 C[5]	0.00015 [5]	0.0030 [5]	NA	NA	NA	0.0030	RBSSL	8.1 C[5]	NA	NA	NA
Trichloroethene	79-01-6	2.8 C	2.8 C	0.00072	0.014	4.9	0.057	0.057	0.014	RBSSL	14 C	24	0.35	0.35
Trichlorofluoromethane	75-69-4	790 N	79 N	0.83	17	980	29	29	17	RBSSL	3400 N	1300	540	540
Vinyl chloride	75-01-4	0.060 C	0.060 C	0.0000056	0.00011	1.5	0.013	0.013	0.00011	RBSSL	1.7 C	6.4	0.027	0.027
Xylenes (total)	1330-20-7	630 N	63 N	0.20	4.0	690	210	170	4.0	RBSSL	2700 N	890	430	170
Semivolatile Organic Compounds														
1,1'-Biphenyl	92-52-4	3900 N	390 N	19	380	NA	NA	NA	380	RBSSL	51000 N	NA	NA	NA
1,2,4,5-Tetrachlorobenzene	95-94-3	18 N	1.8 N	0.051	1.0	NA	NA	NA	1.0	RBSSL	180 N	NA	NA	NA
2,2'-Oxybis(1-chloropropane)	108-60-1	4.6 C	4.6 C	0.00012	0.0024	30	0.027	0.027	0.0024	RBSSL	22 C	61	0.26	0.26
2,3,4,6-Tetrachlorophenol	58-90-2	1800 N	180 N	6.7	130	NA	NA	NA	130	RBSSL	18000 N	NA	NA	NA
2,4,5-Trichlorophenol	95-95-4	6100 N	610 N	14	280	18000	250	250	250	IDEM-RDCL	62000 N	49000	690	690
2,4,6-Trichlorophenol	88-06-2	61 N[4]	6.1 N[4]	0.023 [4]	0.46 [4]	18	0.07	0.07	0.07	IDEM-RDCL	620 N[4]	49	0.2	0.2
2,4-Dichlorophenol	120-83-2	180 N	18 N	0.13	2.6	550	1.1	1.1	1.1	IDEM-RDCL	1800 N	1500	3	3
2,4-Dimethylphenol	105-67-9	1200 N	120 N	0.86	17	3700	9	9	9	IDEM-RDCL	12000 N	9800	25	25
2,4-Dinitrophenol	51-28-5	120 N	12 N	0.082	1.6	370	0.29	0.29	0.29	IDEM-RDCL	1200 N	980	0.82	0.82

NSA Crane SWMU 11 Human Health Screening Criteria - Surface and Subsurface Soil Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	EPA Regional Screening Level, Migration to Groundwater [1] (mg/kg)	Adjusted EPA Regional Screening Level, Migration to Groundwater [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]			Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]		
						Residential Direct Contact	Migration to Groundwater	Residential Default Closure Level				Industrial Direct Contact	Migration to Groundwater	Industrial Default Closure Level
2,4-Dinitrotoluene	121-14-2	1.6 C	1.6 C	0.00029	0.0058	NA	NA	NA	0.0058	RBSSL	5.5 C	NA	NA	NA
2,6-Dinitrotoluene	606-20-2	61 N	6.1 N	0.050	1.0	NA	NA	NA	1.0	RBSSL	620 N	NA	NA	NA
2-Chloronaphthalene	91-58-7	6300 N	630 N	15	300	15000	42	42	42	IDEM-RDCL	82000 N	39000	560	560
2-Chlorophenol	95-57-8	390 N	39 N	0.15	3.0	360	0.75	0.75	0.75	IDEM-RDCL	5100 N	580	10	10
2-Methylphenol (o-Cresol)	95-48-7	3100 N	310 N	1.5	30	7500	14	14	14	IDEM-RDCL	31000 N	17000	39	39
2-Nitroaniline	88-74-4	610 N	61 N	0.15	3.0	550	0.67	0.67	0.67	IDEM-RDCL	6000 N	1500	1.9	1.9
2-Nitrophenol	88-75-5	120 N[7]	12 N[7]	0.08 [7]	1.6 [7]	NA	NA	NA	1.6	RBSSL	1200 N[7]	NA	NA	NA
3,3'-Dichlorobenzidine	91-94-1	1.1 C	1.1 C	0.0010	0.020	9.5	0.062	0.062	0.020	RBSSL	3.8 C	31	0.21	0.21
3-Methylphenol (m-Cresol)	108-39-4	3100 N	310 N	1.5	30	9100	9.8	9.8	9.8	IDEM-RDCL	31000 N	25000	28	28
3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	534-52-1	4.9 N	0.49 N	0.0050	0.10	NA	NA	NA	0.10	RBSSL	49 N	NA	NA	NA
4-Bromophenyl phenyl ether	101-55-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methyl phenol	59-50-7	6100 N	610 N	4.3	86	NA	NA	NA	86	RBSSL	62000 N	NA	NA	NA
4-Chloroanaline	106-47-8	2.4 C	2.40 C	0.00014	0.0028	730	0.97	0.97	0.0028	RBSSL	8.6 C	2000	2.7	2.7
4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
4-Methylphenol (p-Cresol)	106-44-5	310 N	31 N	0.15	3.0	910	1.1	1.1	1.1	IDEM-RDCL	3100 N	2500	3	3
4-Nitroaniline	100-01-6	24 C	24 C	0.0014	0.028	NA	NA	NA	0.028	RBSSL	86 C	NA	NA	NA
4-Nitrophenol	100-02-7	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Acetophenone	98-86-2	7800 N	780 N	1.1	22	NA	NA	NA	22	RBSSL	100000 N	NA	NA	NA
Atrazine	1912-24-9	2.1 C	2.1 C	0.00019	0.0038	19	0.048	0.048	0.0038	RBSSL	7.5 C	63	0.21	0.21
Benzaldehyde	100-52-7	7800 N	780 N	0.81	16.2	NA	NA	NA	16.2	RBSSL	100000 N	NA	NA	NA
Bis(2-chloroethoxy)methane	111-91-1	180 N	18 N	0.025	0.50	NA	NA	NA	0.50	RBSSL	1800 N	NA	NA	NA
Bis(2-chloroethyl)ether	111-44-4	0.21 C	0.21 C	0.0000031	0.000062	1.6	0.0007	0.0007	0.000062	RBSSL	1.0 C	3	0.012	0.012
Bis(2-ethylhexyl)phthalate	117-81-7	35 C	35 C	1.1	22	300	3600	300	22	RBSSL	120 C	980	120000	980
Butyl benzyl phthalate	85-68-7	260 C	260 C	0.51	10	37000	6200	310	10	RBSSL	910 C	98000	6200	310
Caprolactam	105-60-2	31000 N	3100 N	4.5	90	NA	NA	NA	90	RBSSL	310000 N	NA	NA	NA
Carbazole	86-74-8	NA	NA	NA	NA	210	5.9	5.9	5.9	IDEM-RDCL	NA	690	20	20
Dibenzofuran	132-64-9	78 N	7.8 N	0.68	13.6	370	4.9	4.9	4.9	IDEM-RDCL	1000 N	980	65	65
Diethyl phthalate	84-66-2	49000 N	4900 N	12	240	150000	450	450	240	RBSSL	490000 N	390000	1300	840
Dimethyl phthalate	131-11-3	49000 N[8]	4900 N[8]	12 [8]	240 [8]	1000000	2000	1100	240	RBSSL	490000 N[8]	1000000	5600	1100
Di-n-butyl phthalate	84-74-2	6100 N	610 N	9.2	184	18000	5000	760	184	RBSSL	62000 N	49000	14000	760
Di-n-octyl phthalate	117-84-0	NA	NA	NA	NA	7300	67000	2000	2000	IDEM-RDCL	NA	20000	67000	2000
Hexachlorobenzene	118-74-1	0.30 C	0.30 C	0.00053	0.011	2.7	2.2	2.2	0.011	RBSSL	1.1 C	8.6	3.9	3.9
Hexachlorobutadiene	87-68-3	61 N[4]	6.1 N[4]	0.0017	0.034	55	24	24	0.034	RBSSL	22 C	150	66	66
Hexachlorocyclopentadiene	77-47-4	370 N	37 N	0.68	14	1100	400	400	14	RBSSL	3700 N	2900	4900	720
Hexachloroethane	67-72-1	61 N[4]	6.1 N[4]	0.0029	0.058	120	2.8	2.8	0.058	RBSSL	620 N[4]	240	7.7	7.7
Isophorone	78-59-1	510 C	510 C	0.023	0.46	4500	5.3	5.3	0.46	RBSSL	1800 C	14000	18	18
Nitrobenzene	98-95-3	4.8 C	4.8 C	0.000079	0.0016	91	0.028	0.028	0.0016	RBSSL	24 C	250	0.34	0.34
N-Nitrosodi-n-propylamine	621-64-7	0.069 C	0.069 C	0.0000072	0.00014	0.61	0.0006	0.0006	0.00014	RBSSL	0.25 C	2	0.002	0.002
N-Nitrosodiphenylamine	86-30-6	99 C	99 C	0.075	1.5	870	9.7	9.7	1.5	RBSSL	350 C	2800	32	32
Pentachlorophenol	87-86-5	0.89 C	0.89 C	0.0017	0.034	20	0.028	0.028	0.028	IDEM-RDCL	9.0 C	54	0.66	0.66
Phenol	108-95-2	18000 N	1800 N	6.3	126	44000	56	56	56	IDEM-RDCL	180000 N	96000	160	160
Polycyclic Aromatic Hydrocarbons														
2-Methylnaphthalene	91-57-6	310 N	31 N	0.75	15	630	3.1	3.1	3.1	IDEM-RDCL	4100 N	1600	42	42
Acenaphthene	83-32-9	3400 N	340 N	22	440	9500	130	130	130	IDEM-RDCL	33000 N	24000	1800	1800
Acenaphthylene	208-96-8	3400 N[9]	340 N[9]	360 [9]	7200 [9]	1100	18	18	18	IDEM-RDCL	170000 N[9]	2800	180	180
Anthracene	120-12-7	17000 N	1700 N	360	7200	47000	2700	2000	1700	R-RSL	170000 N	120000	36000	2000
Benzo(a)anthracene	56-55-3	0.15 C	0.15 C	0.010	0.20	5	19	5	0.15	R-RSL	2.1 C	15	62	15
Benzo(a)pyrene	50-32-8	0.015 C	0.015 C	0.0035	0.070	0.5	8.2	0.5	0.015	R-RSL	0.21 C	1.5	16	1.5
Benzo(b)fluoranthene	205-99-2	0.15 C	0.15 C	0.035	0.70	5	57	5	0.15	R-RSL	2.1 C	15	190	15
Benzo(g,h,i)perylene	191-24-2	1700 N[10]	170 N[10]	120 [10]	2400 [10]	NA	NA	NA	170	R-RSL	17000 N[10]	NA	NA	NA
Benzo(k)fluoranthene	207-08-9	1.5 C	1.5 C	0.35	7.0	50	570	50	1.5	R-RSL	21 C	150	1900	150
Chrysene	218-01-9	15 C	15 C	1.1	22	500	1900	500	15	R-RSL	210 C	1500	6200	1500
Dibenzo(a,h)anthracene	53-70-3	0.015 C	0.015 C	0.011	0.22	0.5	18	0.5	0.015	R-RSL	0.21 C	1.5	60	1.5
Fluoranthene	206-44-0	2300 N	230 N	160	3200	6300	6300	2000	230	R-RSL	22000 N	16000	18000	2000
Fluorene	86-73-7	2300 N	230 N	27	540	6300	170	170	170	IDEM-RDCL	22000 N	16000	2300	2000
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15 C	0.15 C	0.12	2.4	5	160	5	0.15	R-RSL	2.1 C	15	540	15
Naphthalene	91-20-3	3.6 C	3.60 C	0.00047	0.0094	3200	0.7	0.7	0.0094	RBSSL	18 C	8000	170	170
Phenanthrene	85-01-8	1700 N[10]	170 N[10]	120 [10]	2400 [10]	470	13	13	13	IDEM-RDCL	17000 N[10]	1200	170	170
Pyrene	129-00-0	1700 N	170 N	120	2400	4700	4600	2000	170	R-RSL	17000 N	12000	13000	2000
Polychlorinated Biphenyls														
Aroclor-1016	12674-11-2	3.9 N	0.39 N	0.092	1.8	NA	NA	NA	0.39	R-RSL	37 N[4]	NA	NA	NA
Aroclor-1221	11104-28-2	0.14 C	0.14 C	0.00012	0.0024	NA	NA	NA	0.0024	RBSSL	0.54 C	NA	NA	NA

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Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	EPA Regional Screening Level, Migration to Groundwater [1] (mg/kg)	Adjusted EPA Regional Screening Level, Migration to Groundwater [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]			Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]		
						Residential Direct Contact	Migration to Groundwater	Residential Default Closure Level				Industrial Direct Contact	Migration to Groundwater	Industrial Default Closure Level
Aroclor-1232	11141-16-5	0.14 C	0.14 C	0.00012	0.0024	NA	NA	NA	0.0024	RBSSL	0.54 C	NA	NA	NA
Aroclor-1242	53469-21-9	0.22 C	0.22 C	0.0053	0.11	NA	NA	NA	0.11	RBSSL	0.74 C	NA	NA	NA
Aroclor-1248	12672-29-6	0.22 C	0.22 C	0.0052	0.10	NA	NA	NA	0.10	RBSSL	0.74 C	NA	NA	NA
Aroclor-1254	11097-69-1	1.1 N[4]	0.11 N[4]	0.0088	0.18	NA	NA	NA	0.11	R-RSL	0.74 C	NA	NA	NA
Aroclor-1260	11096-82-5	0.22 C	0.22 C	0.024	0.48	NA	NA	NA	0.22	R-RSL	0.74 C	NA	NA	NA
Total PCBs	1336-36-3	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Metals														
Aluminum	7429-90-5	77000 N	7700 N	55000	1100000	NA	NA	NA	7700	R-RSL	990000 NM	NA	NA	NA
Antimony	7440-36-0	31 N	3.1 N	0.66	13	140	5.4	5.4	3.1	R-RSL	410 N	620	37	37
Arsenic	7440-38-2	0.39 C	0.39 C	0.0013	0.026	3.9	5.8	3.9	0.026	RBSSL	1.6 C	20	5.8	5.8
Barium	7440-39-3	15000 N	1500 N	300	6000	63000	1600	1600	1500	R-RSL	190000 NM	230000	17000	10000
Beryllium	7440-41-7	160 N	16 N	58	1160	680	63	63	16	R-RSL	2000 N	2900	3200	2300
Cadmium	7440-43-9	70 N	7 N	1.4	28	12	7.5	7.5	7	R-RSL	800 N	990	77	77
Calcium	7440-70-2	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Chromium	7440-47-3	0.29 C[11]	0.29 C[11]	0.00083 [11]	0.017 [11]	430[11]	38[11]	38[11]	0.017	RBSSL	5.6 C[11]	650	120	120
Cobalt	7440-48-4	23 N	2.3 N	0.49	9.8	NA	NA	NA	2.3	R-RSL	300 N	NA	NA	NA
Copper	7440-50-8	3100 N	310 N	51	1020	14000	920	920	310	R-RSL	41000 N	62000	2900	2900
Iron	7439-89-6	55000 N	5500 N	640	12800	NA	NA	NA	5500	R-RSL	720000 N	NA	NA	NA
Lead	7439-92-1	400 [12]	400 [12]	14 [15]	280 [15]	400	81	81	81	IDEM-RDCL	800 [21]	1300	230	230
Magnesium	7439-95-4	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Manganese	7439-96-5	1800 N	180 N	57	1140	NA	NA	NA	180	R-RSL	23000 N	NA	NA	NA
Mercury	7439-97-6	23 N[13]	2.3 N[13]	0.57 [13]	11.4 [13]	NA	NA	NA	2.3	R-RSL	310 N[13]	NA	NA	NA
Nickel	7440-02-0	1500 N	150 N	48	960	6900	950	950	150	R-RSL	20000 N	31000	2700	2700
Potassium	7440-09-7	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Selenium	7782-49-2	390 N	39 N	0.95	19	1700	5.2	5.2	5.2	IDEM-RDCL	5100 N	7800	53	53
Silver	7440-22-4	390 N	39 N	1.6	32	1700	31	31	31	IDEM-RDCL	5100 N	7800	87	87
Sodium	7440-23-5	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Thallium	7440-28-0	NA	NA	NA	NA	24	2.8	2.8	2.8	IDEM-RDCL	NA	110	10	10
Vanadium	7440-62-2	390 N[14]	39 N[14]	180 [14]	3600 [14]	NA	NA	NA	39	R-RSL	5200 N[14]	NA	NA	NA
Zinc	7440-66-6	23000 N	2300 N	680	13600	100000	14000	10000	2300	R-RSL	310000 N	470000	38000	10000
Pesticides														
4,4'-DDD	72-54-8	2.0 C	2.0 C	0.066	1.3	28	140	28	1.3	RBSSL	7.2 C	120	480	120
4,4'-DDE	72-55-9	1.4 C	1.4 C	0.047	0.94	20	450	20	0.94	RBSSL	5.1 C	86	1500	86
4,4'-DDT	50-29-3	1.7 C	1.7 C	0.067	1.3	20	260	20	1.3	RBSSL	7.0 C	86	890	86
Aldrin	309-00-2	0.029 C	0.029 C	0.00065	0.013	0.25	4.9	0.25	0.013	RBSSL	0.10 C	0.8	16	0.8
alpha-BHC	319-84-6	0.077 C	0.077 C	0.000062	0.0012	0.99	0.0072	0.0072	0.0012	RBSSL	0.27 C	4	0.024	0.024
alpha-Chlordane	5103-71-9	1.6 C[16]	1.6 C[16]	0.013 [16]	0.26 [16]	17	9.6	9.6	0.26	RBSSL	6.5 C[16]	68	39	39
beta-BHC	319-85-7	0.27 C	0.27 C	0.00022	0.0044	3.3	0.026	0.026	0.0044	RBSSL	0.96 C	12	0.086	0.086
delta-BHC	319-86-8	0.077 C[17]	0.077 C[17]	0.000062 [17]	0.0012 [17]	0.99	0.0072	0.0072	0.0012	RBSSL	0.27 C[17]	4	0.024	0.024
Dieldrin	60-57-1	0.030 C	0.030 C	0.00017	0.0034	0.27	0.046	0.046	0.0034	RBSSL	0.11 C	0.86	0.15	0.15
Endosulfan I	959-98-8	370 N[18]	37 N[18]	3.0 [18]	60 [18]	1100	20	20	20	IDEM-RDCL	3700 N[18]	2900	46	46
Endosulfan II	33213-65-9	370 N[18]	37 N[18]	3.0 [18]	60 [18]	1100	20	20	20	IDEM-RDCL	3700 N[18]	2900	46	46
Endosulfan sulfate	1031-07-8	370 N[18]	37 N[18]	3.0 [18]	60 [18]	1100	20	20	20	IDEM-RDCL	3700 N[18]	2900	46	46
Endrin	72-20-8	18 N	1.8 N	0.44	8.8	55	0.99	0.99	0.99	IDEM-RDCL	180 N	150	15	15
Endrin aldehyde	7421-93-4	18 N[19]	1.8 N	0.44 [19]	8.8 [19]	55	0.99	0.99	0.99	IDEM-RDCL	180 N[19]	150	15	15
Endrin ketone	53494-70-5	18 N[19]	1.8 N	0.44 [19]	8.8 [19]	55	0.99	0.99	0.99	IDEM-RDCL	180 N[19]	150	15	15
gamma-BHC (Lindane)	58-89-9	0.52 C	0.52 C	0.00036	0.0072	4.8	0.0094	0.0094	0.0072	RBSSL	2.1 C	19	0.1	0.1
gamma-chlordane	5103-74-2	1.6 C[16]	1.6 C[16]	0.013 [16]	0.26 [16]	17	9.6	9.6	0.26	RBSSL	6.5 C[16]	68	39	39
Heptachlor	76-44-8	0.11 C	0.11 C	0.0012	0.024	0.93	23	0.93	0.024	RBSSL	0.38 C	2.9	36	2.9
Heptachlor epoxide	1024-57-3	0.053 C	0.053 C	0.00015	0.0030	0.47	0.67	0.47	0.0030	RBSSL	0.19 C	1.5	1	1
Methoxychlor	72-43-5	310 N	31 N	9.9	198	910	160	160	31	R-RSL	3100 N	2500	180	180
Toxaphene	8001-35-2	0.44 C	0.44 C	0.0094	0.19	3.9	31	3.9	0.19	RBSSL	1.6 C	12	31	12
Dioxins/Furans														
1,2,3,4,6,7,8,9-OCDD	3268-87-9	0.015 C[20]	0.015 C[20]	0.00087 [20]	0.017 [20]	NA	NA	NA	0.015	R-RSL	0.06 C[20]	NA	NA	NA
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.015 C[20]	0.015 C[20]	0.00087 [20]	0.017 [20]	NA	NA	NA	0.015	R-RSL	0.06 C[20]	NA	NA	NA
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.00045 C[20]	0.00045 C[20]	0.000026 [20]	0.00052 [20]	NA	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA	NA
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.00045 C[20]	0.00045 C[20]	0.000026 [20]	0.00052 [20]	NA	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA	NA
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.00045 C[20]	0.00045 C[20]	0.000026 [20]	0.00052 [20]	NA	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA	NA
1,2,3,4,7,8-HXCDD	39227-28-6	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
1,2,3,4,7,8-HXCDF	70648-26-9	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
1,2,3,6,7,8-HXCDD	57653-85-7	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
1,2,3,6,7,8-HXCDF	57117-44-9	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA

NSA Crane SWMU 11 Human Health Screening Criteria - Surface and Subsurface Soil Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	EPA Regional Screening Level, Migration to Groundwater [1] (mg/kg)	Adjusted EPA Regional Screening Level, Migration to Groundwater [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]			Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]		
						Residential Direct Contact	Migration to Groundwater	Residential Default Closure Level				Industrial Direct Contact	Migration to Groundwater	Industrial Default Closure Level
1,2,3,7,8,9-HXCDD	19408-74-3	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
1,2,3,7,8,9-HXCDF	72918-21-9	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
1,2,3,7,8-PECDD	40321-76-4	0.0000045 C[20]	4.5E-06 C[20]	0.00000026 [20]	0.0000052 [20]	NA	NA	NA	0.0000045	R-RSL	0.000018 C[20]	NA	NA	NA
1,2,3,7,8-PECDF	57117-41-6	0.00015 C[20]	0.00015 C[20]	0.0000087 [20]	0.00017 [20]	NA	NA	NA	0.00015	R-RSL	0.00060 C[20]	NA	NA	NA
2,3,4,6,7,8-HXCDF	60851-34-5	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
2,3,4,7,8-PECDF	57117-31-4	0.000015 C[20]	1.5E-05 C[20]	0.00000087 [20]	0.000017 [20]	NA	NA	NA	0.000015	R-RSL	0.000060 C[20]	NA	NA	NA
2,3,7,8-TCDD	1746-01-6	0.0000045 C	4.5E-06 C	0.00000026	0.0000052	NA	NA	NA	0.0000045	R-RSL	0.000018 C	NA	NA	NA
2,3,7,8-TCDF	51207-31-9	0.000045 C[20]	4.5E-05 C[20]	0.0000026 [20]	0.000052 [20]	NA	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA	NA
Total HpCDD	37871-00-4	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total HpCDF	38998-75-3	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total HxCDD	34465-46-8	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total HxCDF	55684-94-1	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total PeCDD	36088-22-9	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total PeCDF	30402-15-4	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total TCDD	41903-57-5	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA
Total TCDF	55722-27-5	NA	NA	NA	NA	NA	NA	NA	NA	None	NA	NA	NA	NA

Notes:

1 - The residential direct contact (R-RSL) and risk-based migration to groundwater soil screening levels (RBSSL) from the USEPA Regions 3, 6, and 9 Regional Screening Levels for Chemical Contaminants at Superfund Sites, November, 2010 available online at http://epa-prgs.ornl.gov/chemicals/index.shtml. The risk-based screening levels are based on a target hazard quotient of 1 for noncarcinogens (denoted with a "N" flag) or an incremental lifetime cancer risk (ILCR) of 1E-6 for carcinogens (denoted with a "C" flag). (Industrial criteria are also presented for information purposes.)

2 - The USEPA R-RSL (November, 2010) residential soil screening level for noncarcinogens adjusted by dividing by 10, equivalent to a target hazard quotient of 0.1. The residential soil screening level for carcinogens (not adjusted) is equivalent to an incremental lifetime cancer risk (ILCR) of 1E-6. The USEPA RBSSL (November, 2010) is adjusted for a dilution attenuation factor (DAF) of 20.

3 - Indiana Department of Environmental Management (IDEM) residential soil direct contact screening levels, migration to groundwater screening levels, and Residential Default Closure Levels from IDEM RISC Technical Guide, January 31, 2006, Appendix 1, revised May 1, 2009. (Industrial criteria are also presented for information purposes.)

4 - One tenth the noncarcinogenic value is less than the carinogenic value; therefore, the noncarcinogenic value is presented.

5 - Value is for 1,3-dichloropropene.

6 - Value is for xylene, mixture.

7 - Value is for 2,4-dinitrophenol.

8 - Value is for diethyl phthalate.

9 - Value is for acenapththene.

10 - Value is for pyrene.

11 - Value is for hexavalent chromium.

12 - Office of Solid Waste and Emergency Response soil screening level (EPA, 1994b)

13 - Value is for mercuy, inorganic salts.

14 - Value is for vanadium and compounds.

15 - Calculated from the EPA website (http://epa-prgs.ornl.gov/cgi-bin/chemicals/csl_search).

16 - Value is for chlordane.

17 - Value is for alpha-BHC.

18 - Value is for endosulfan.

19 - Value is for endrin.

20 - Value is derived by multiplying criteria for 2,3,7,8-TCDD by World Health Organization Toxicity Equivalent Factor.

21 - USEPA Technical Review Workgroup for Lead. Guidance Document. “Frequently Asked Question (FAQs) on the Adult‑Lead Model.” October 2010. http://www.epa.gov/superfund/lead/almfaq.htm

Abbreviations:

NA - Not applicable or not available
C - Carcinogen
EPA - U.S. Environmental Protection Agency
N - Noncarcinogen
NA - Not available
SSL - Soil Screening Level

Crane SWMU 11 Human Health Screening Criteria - Groundwater Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Regional Screening Level, Vapor[1] (ug/L)	Adjusted EPA Regional Screening Level, Vapor[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Volatile Organic Compounds										
1,1,1-Trichloroethane	71-55-6	9100 N	910 N	7400 N	740 N	200	3800	200	200	IDEM G-DCL
1,1,2,2-Tetrachloroethane	79-34-5	0.067 C	0.067 C	2.8 C	2.8 C	NA	0.9	0.9	0.067	T-RSL
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	59000 N	5900 N	1400 N	140 N	NA	NA	NA	140	VAPOR
1,1,2-Trichloroethane	79-00-5	0.24 C	0.24 C	4.4 C	4.4 C	5	3.2	5	0.24	T-RSL
1,1-Dichloroethane	75-34-3	2.4 C	2.4 C	6.5 C	6.5 C	NA	990	990	2.4	T-RSL
1,1-Dichloroethene	75-35-4	340 N	34 N	190 N	19 N	7	430	7	7	IDEM G-DCL
1,2,3- Trichlorobenzene	87-61-6	29 N	2.9 N	NA	NA	NA	NA	NA	2.9	T-RSL
1,2,4-Trichlorobenzene	120-82-1	4.1 N[5]	0.41 N[5]	36 N	3.6 N	70	9.5	70	0.41	T-RSL
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.00032 C	0.00032 C	0.027 C	0.027 C	0.2	NA	NA	0.00032	T-RSL
1,2-Dibromoethane (EDB)	106-93-4	0.0065 C	0.0065 C	0.15 C	0.15 C	0.05	0.086	0.05	0.0065	T-RSL
1,2-Dichlorobenzene	95-50-1	370 N	37 N	2700 N	270 N	600	480	600	37	T-RSL
1,2-Dichloroethane	107-06-2	0.15 C	0.15 C	2.0 C	2.0 C	5	2	5	0.15	T-RSL
1,2-Dichloropropane	78-87-5	0.39 C	0.39 C	2.0 C	2.0 C	5	2.6	5	0.39	T-RSL
1,3-Dichlorobenzene	541-73-1	NA	NA	NA	NA	NA	80	80	80	IDEM G-DCL
1,4-Dichlorobenzene	106-46-7	0.43 C	0.43 C	2.2 C	2.2 C	75	8	75	0.43	T-RSL
1,4-Dioxane	123-91-1	0.67 C	0.67 C	NA	NA	NA	NA	NA	0.67	T-RSL
2-Butanone (MEK)	78-93-3	7100 N	710 N	2300000 N	230000 N	NA	8400	8400	710	T-RSL
2-Hexanone	591-78-6	47 N	4.7 N	8200 N	820 N	NA	NA	NA	4.7	T-RSL
4-Methyl-2-pentanone (MIBK)	108-10-1	2000 N	200 N	550000 N	55000 N	NA	2200	2200	200	T-RSL
Acetone	67-64-1	22000 N	2200 N	23000000 N	2300000 N	NA	6900	6900	2200	T-RSL
Benzene	71-43-2	0.41 C	0.41 C	1.3 C	1.3 C	5	5.5	5	0.41	T-RSL
Bromochloromethane	74-97-5	NA	NA	NA	NA	NA	NA	NA	NA	None
Bromodichloromethane	75-27-4	0.12 C	0.12 C	0.76 C	0.76 C	80	2.9	80	0.12	T-RSL
Bromoform	75-25-2	8.5 C	8.5 C	100 C	100 C	80	110	80	8.5	T-RSL
Bromomethane	74-83-9	8.7 N	0.87 N	17 N	1.7 N	NA	11	11	0.87	T-RSL
Carbon disulfide	75-15-0	1000 N	100 N	1200 N	120 N	NA	1300	1300	100	T-RSL
Carbon tetrachloride	56-23-5	0.44 C	0.44 C	0.37 C	0.37 C	5	2.6	5	0.37	VAPOR
Chlorobenzene	108-90-7	91 N	9.1 N	400 N	40 N	100	130	100	9.1	T-RSL
Chloroethane	75-00-3	21000 N	2100 N	22000 N	2200 N	NA	62	62	62	IDEM G-DCL
Chloroform	67-66-3	0.19 C	0.19 C	0.73 C	0.73 C	80	2.8	80	0.19	T-RSL
Chloromethane	74-87-3	190 N	19 N	260 N	26 N	NA	NA	NA	19	T-RSL
cis-1,2-Dichloroethene	156-59-2	73 N	7.3 N	NA	NA	70	77	70	7.3	T-RSL
cis-1,3-Dichloropropene	10061-01-5	40 N[6]	4 N[6]	4.1 C[6]	4.1 C[6]	NA	NA	NA	4	T-RSL
Cyclohexane	110-82-7	13000 N	1300 N	1000 N	100 N	NA	13000	13000	100	VAPOR
Dibromochloromethane	124-48-1	0.15 C	0.15 C	2.8 C	2.8 C	80	NA	NA	0.15	T-RSL
Dichlorodifluoromethane	75-71-8	390 N	39 N	15 N	1.5 N	NA	NA	NA	1.5	VAPOR
Ethylbenzene	100-41-4	1.5 C	1.5 C	3.0 C	3 C	700	1600	700	1.5	T-RSL
Isopropylbenzene	98-82-8	680 N	68 N	890 N	89 N	NA	830	830	68	T-RSL
m,p-Xylenes	179601-23-1	200 N[7]	20 N[7]	480 N[7]	48 N[7]	10000	NA	NA	20	T-RSL
Methyl acetate	79-20-9	37000 N	3700 N	NA	NA	NA	NA	NA	3700	T-RSL
Methylcyclohexane	108-87-2	NA	NA	NA	NA	NA	NA	NA	NA	None
Methylene chloride	75-09-2	4.8 C	4.8 C	40 C	40 C	5	63	5	4.8	T-RSL
Methyl-tert-butyl ether	1634-04-4	12 C	12 C	390 C	390 C	NA	40	40	12	T-RSL
o-Xylene	95-47-6	1200 N	120 N	3500 N	350 N	NA	NA	NA	120	T-RSL
Styrene	100-42-5	1600 N	160 N	9100 N	910 N	100	2000	100	100	IDEM G-DCL
Tetrachloroethene	127-18-4	0.11 C	0.11 C	0.57 C	0.57 C	5	6.5	5	0.11	T-RSL
Toluene	108-88-3	2300 N	230 N	19000 N	1900 N	1000	2400	1000	230	T-RSL
trans-1,2-Dichloroethene	156-60-5	110 N	11 N	370 N	37 N	100	150	100	11	T-RSL
trans-1,3-Dichloropropene	10061-02-6	40 N[6]	4.0 N[6]	4.1 C[6]	4.1 C[6]	NA	NA	NA	4.0	T-RSL

Crane SWMU 11 Human Health Screening Criteria - Groundwater Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Regional Screening Level, Vapor[1] (ug/L)	Adjusted EPA Regional Screening Level, Vapor[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Trichloroethene	79-01-6	2.0 C	2.0 C	3.0 C	3.0 C	5	2.8	5	2.0	T-RSL
Trichlorofluoromethane	75-69-4	1300 N	130 N	180 N	18 N	NA	1700	1700	18	VAPOR
Vinyl chloride	75-01-4	0.016 C	0.016 C	0.15 C	0.15 C	2	0.53	2	0.016	T-RSL
Xylenes (total)	1330-20-7	200 N	20 N	480 N	48 N	10000	270	10000	20	T-RSL
Semivolatile Organic Compounds										
1,1'-Biphenyl	92-52-4	1800 N	180 N	NA	NA	NA	NA	NA	180	T-RSL
1,2,4,5-Tetrachlorobenzene	95-94-3	11 N	1.1 N	NA	NA	NA	NA	NA	1.1	T-RSL
2,2'-Oxybis(1-chloropropane)	108-60-1	0.32 C	0.32 C	80 C	80 C	NA	4.2	4.2	0.32	T-RSL
2,3,4,6-Tetrachlorophenol	58-90-2	1100 N	110 N	NA	NA	NA	NA	NA	110	T-RSL
2,4,5-Trichlorophenol	95-95-4	3700 N	370 N	NA	NA	NA	3600	3600	370	T-RSL
2,4,6-Trichlorophenol	88-06-2	37 N[5]	3.7 N[5]	NA	NA	NA	3.6	3.6	3.6	IDEM G-DCL
2,4-Dichlorophenol	120-83-2	110 N	11 N	NA	NA	NA	110	110	11	T-RSL
2,4-Dimethylphenol	105-67-9	730 N	73 N	NA	NA	NA	730	730	73	T-RSL
2,4-Dinitrophenol	51-28-5	73 N	7.3 N	NA	NA	NA	73	73	7.3	T-RSL
2,4-Dinitrotoluene	121-14-2	0.22 C	0.22 C	NA	NA	NA	NA	NA	0.22	T-RSL
2,6-Dinitrotoluene	606-20-2	37 N	3.7 N	NA	NA	NA	NA	NA	3.7	T-RSL
2-Chloronaphthalene	91-58-7	2900 N	290 N	NA	NA	NA	610	610	290	T-RSL
2-Chlorophenol	95-57-8	180 N	18 N	NA	NA	NA	38	38	18	T-RSL
2-Methylphenol (o-Cresol)	95-48-7	1800 N	180 N	NA	NA	NA	1800	1800	180	T-RSL
2-Nitroaniline	88-74-4	370 N	37 N	NA	NA	NA	110	110	37	T-RSL
2-Nitrophenol	88-75-5	73 N[8]	7.3 N[8]	NA	NA	NA	NA	NA	7.3	T-RSL
3,3'-Dichlorobenzidine	91-94-1	0.15 C	0.15 C	NA	NA	NA	1.9	1.9	0.15	T-RSL
3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	NA	NA	NA	None
4,6-Dinitro-2-methylphenol	534-52-1	2.9 N	0.29 N	NA	NA	NA	NA	NA	0.29	T-RSL
4-Bromophenyl phenyl ether	101-55-3	NA	NA	NA	NA	NA	NA	NA	NA	None
4-Chloro-3-methyl phenol	59-50-7	3700 N	370 N	NA	NA	NA	NA	NA	370	T-RSL
4-Chloroanaline	106-47-8	0.34 C	0.34 C	NA	NA	NA	150	150	0.34	T-RSL
4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	NA	NA	NA	NA	NA	NA	None
4-Methylphenol (p-Cresol)	106-44-5	180 N	18 N	NA	NA	NA	180	180	18	T-RSL
4-Nitroaniline	100-01-6	3.4 C	3.4 C	NA	NA	NA	NA	NA	3.4	T-RSL
4-Nitrophenol	100-02-7	NA	NA	NA	NA	NA	NA	NA	NA	None
Acetophenone	98-86-2	3700 N	370 N	NA	NA	NA	NA	NA	370	T-RSL
Atrazine	1912-24-9	0.29 C	0.29 C	NA	NA	3	3.9	3	0.29	T-RSL
Benzaldehyde	100-52-7	3700 N	370 N	NA	NA	NA	NA	NA	370	T-RSL
Bis(2-chloroethoxy)methane	111-91-1	110 N	11 N	NA	NA	NA	NA	NA	11	T-RSL
Bis(2-chloroethyl)ether	111-44-4	0.012 C	0.012 C	11 C	11 C	NA	0.15	0.15	0.012	T-RSL
Bis(2-ethylhexyl)phthalate	117-81-7	4.8 C	4.8 C	NA	NA	6	61	6	4.8	T-RSL
Butyl benzyl phthalate	85-68-7	35 C	35 C	NA	NA	NA	7300	2700	35	T-RSL
Caprolactam	105-60-2	18000 N	1800 N	NA	NA	NA	NA	NA	1800	T-RSL
Carbazole	86-74-8	NA	NA	NA	NA	NA	43	43	43	IDEM G-DCL
Dibenzofuran	132-64-9	37 N	3.7 N	NA	NA	NA	15	15	3.7	T-RSL
Diethyl phthalate	84-66-2	29000 N	2900 N	NA	NA	NA	29000	29000	2900	T-RSL
Dimethyl phthalate	131-11-3	29000 N[9]	2900 N[9]	NA	NA	NA	360000	360000	2900	T-RSL
Di-n-butyl phthalate	84-74-2	3700 N	370 N	NA	NA	NA	3600	3600	370	T-RSL
Di-n-octyl phthalate	117-84-0	NA	NA	NA	NA	NA	1500	20	20	IDEM G-DCL
Hexachlorobenzene	118-74-1	0.042 C	0.042 C	0.076 C	0.076 C	1	0.53	1	0.042	T-RSL
Hexachlorobutadiene	87-68-3	0.86 C	0.86 C	NA	NA	NA	11	11	0.86	T-RSL
Hexachlorocyclopentadiene	77-47-4	220 N	22 N	0.19 N	0.019 N	50	220	50	0.019	VAPOR
Hexachloroethane	67-72-1	37 N[5]	3.7 N[5]	3.8 C	3.8 C	NA	36	36	3.7	T-RSL
Isophorone	78-59-1	71 C	71 C	NA	NA	NA	900	900	71	T-RSL

Crane SWMU 11 Human Health Screening Criteria - Groundwater Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Regional Screening Level, Vapor[1] (ug/L)	Adjusted EPA Regional Screening Level, Vapor[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Nitrobenzene	98-95-3	0.12 C	0.12 C	62 C	62 C	NA	4.3	4.3	0.12	T-RSL
N-Nitrosodi-n-propylamine	621-64-7	0.0096 C	0.0096 C	NA	NA	NA	0.12	0.12	0.0096	T-RSL
N-Nitrosodiphenylamine	86-30-6	14 C	14 C	NA	NA	NA	170	170	14	T-RSL
Pentachlorophenol	87-86-5	0.17 C	0.17 C	NA	NA	1	7.1	1	0.17	T-RSL
Phenol	108-95-2	11000 N	1100 N	NA	NA	NA	11000	11000	1100	T-RSL
Polycyclic Aromatic Hydrocarbons										
2-Methylnaphthalene	91-57-6	150 N	15 N	NA	NA	NA	31	31	15	T-RSL
Acenaphthene	83-32-9	2200 N	220 N	NA	NA	NA	460	460	220	T-RSL
Acenaphthylene	208-96-8	2200 N[10]	220 N[10]	NA	NA	NA	71	71	71	IDEM G-DCL
Anthracene	120-12-7	11000 N	1100 N	NA	NA	NA	2300	2300	1100	T-RSL
Benzo(a)anthracene	56-55-3	0.029 C	0.029 C	NA	NA	NA	1.2	1.2	0.029	T-RSL
Benzo(a)pyrene	50-32-8	0.0029 C	0.0029 C	NA	NA	0.2	0.12	0.2	0.0029	T-RSL
Benzo(b)fluoranthene	205-99-2	0.029 C	0.029 C	320 C	320 C	NA	1.2	1.2	0.029	T-RSL
Benzo(g,h,i)perylene	191-24-2	1100 N[11]	110 N[11]	NA	NA	NA	NA	NA	110	T-RSL
Benzo(k)fluoranthene	207-08-9	0.29 C	0.29 C	NA	NA	NA	12	12	0.29	T-RSL
Chrysene	218-01-9	2.9 C	2.9 C	410 C	410 C	NA	120	120	2.9	T-RSL
Dibenzo(a,h)anthracene	53-70-3	0.0029 C	0.0029 C	NA	NA	NA	0.12	0.12	0.0029	T-RSL
Fluoranthene	206-44-0	1500 N	150 N	NA	NA	NA	1500	1500	150	T-RSL
Fluorene	86-73-7	1500 N	150 N	NA	NA	NA	310	310	150	T-RSL
Indeno(1,2,3-c,d)pyrene	193-39-5	0.029 C	0.029 C	NA	NA	NA	1.2	1.2	0.029	T-RSL
Naphthalene	91-20-3	0.14 C	0.14 C	4.0 C	4.0 C	NA	8.3	8.3	0.14	T-RSL
Phenanthrene	85-01-8	1100 N[11]	110 N[11]	NA	NA	NA	23	23	23	IDEM G-DCL
Pyrene	129-00-0	1100 N	110 N	NA	NA	NA	1100	1100	110	T-RSL
Polychlorinated Biphenyls										
Aroclor-1016	12674-11-2	0.96 C	0.96 C	NA	NA	0.5	0.5	0.43	0.43	IDEM GW
Aroclor-1221	11104-28-2	0.0068 C	0.0068 C	NA	NA	0.5	0.5	0.43	0.0068	T-RSL
Aroclor-1232	11141-16-5	0.0068 C	0.0068 C	NA	NA	0.5	0.5	0.43	0.0068	T-RSL
Aroclor-1242	53469-21-9	0.034 C	0.034 C	NA	NA	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1248	12672-29-6	0.034 C	0.034 C	NA	NA	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1254	11097-69-1	0.034 C	0.034 C	NA	NA	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1260	11096-82-5	0.034 C	0.034 C	NA	NA	0.5	0.5	0.43	0.034	T-RSL
Total PCBs	1336-36-3	NA	NA	NA	NA	0.5	0.5	0.43	0.43	IDEM GW
Metals										
Aluminum	7429-90-5	37000 N	3700 N	NA	NA	NA	NA	NA	3700	T-RSL
Antimony	7440-36-0	15 N	1.5 N	NA	NA	6	15	6	1.5	T-RSL
Arsenic	7440-38-2	0.045 C	0.045 C	NA	NA	10	0.57	10	0.045	T-RSL
Barium	7440-39-3	7300 N	730 N	NA	NA	2000	7300	2000	730	T-RSL
Beryllium	7440-41-7	73 N	7.3 N	NA	NA	4	73	4	4	IDEM G-DCL
Cadmium	7440-43-9	18 N	1.8 N	NA	NA	5	18	5	1.8	T-RSL
Calcium	7440-70-2	NA	NA	NA	NA	NA	NA	NA	NA	None
Chromium	7440-47-3	0.043 C[12]	0.043 C[12]	NA	NA	100	NA	NA	0.043	T-RSL
Cobalt	7440-48-4	11 N	1.1 N	NA	NA	NA	NA	NA	1.1	T-RSL
Copper	7440-50-8	1500 N	150 N	NA	NA	1300	1500	1300	150	T-RSL
Iron	7439-89-6	26000 N	2600 N	NA	NA	NA	NA	NA	2600	T-RSL
Lead	7439-92-1	15 [13]	15 [13]	NA	NA	15	15	15	15	T-RSL
Magnesium	7439-95-4	NA	NA	NA	NA	NA	NA	NA	NA	None
Manganese	7439-96-5	880 N	88 N	NA	NA	NA	NA	NA	88	T-RSL
Mercury	7439-97-6	11 N[14]	1.1 N[14]	NA	NA	2	NA	NA	1.1	T-RSL
Nickel	7440-02-0	730 N	73 N	NA	NA	NA	730	730	73	T-RSL
Potassium	7440-09-7	NA	NA	NA	NA	NA	NA	NA	NA	None

Crane SWMU 11 Human Health Screening Criteria - Groundwater Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Regional Screening Level, Vapor[1] (ug/L)	Adjusted EPA Regional Screening Level, Vapor[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Selenium	7782-49-2	180 N	18 N	NA	NA	50	180	50	18	T-RSL
Silver	7440-22-4	180 N	18 N	NA	NA	NA	180	180	18	T-RSL
Sodium	7440-23-5	NA	NA	NA	NA	NA	NA	NA	NA	None
Thallium	7440-28-0	NA	NA	NA	NA	2	2.6	2	2	IDEM G-DCL
Vanadium	7440-62-2	180 N[15]	18 N[15]	NA	NA	NA	NA	NA	18	T-RSL
Zinc	7440-66-6	11000 N	1100 N	NA	NA	NA	11000	11000	1100	T-RSL
Pesticides										
4,4'-DDD	72-54-8	0.28 C	0.28 C	NA	NA	NA	3.5	3.5	0.28	T-RSL
4,4'-DDE	72-55-9	0.20 C	0.20 C	NA	NA	NA	2.5	2.5	0.20	T-RSL
4,4'-DDT	50-29-3	0.20 C	0.20 C	NA	NA	NA	2.5	2.5	0.20	T-RSL
Aldrin	309-00-2	0.0040 C	0.0040 C	NA	NA	NA	0.05	0.05	0.0040	T-RSL
alpha-BHC	319-84-6	0.011 C	0.011 C	NA	NA	NA	0.14	0.14	0.011	T-RSL
alpha-Chlordane	5103-71-9	0.19 C[16]	0.19 C[16]	NA	NA	NA	2.4	2	0.19	T-RSL
beta-BHC	319-85-7	0.037 C	0.037 C	NA	NA	NA	0.47	0.47	0.037	T-RSL
delta-BHC	319-86-8	0.011 C[17]	0.011 C[17]	NA	NA	NA	0.14	0.14	0.011	T-RSL
Dieldrin	60-57-1	0.0042 C	0.0042 C	NA	NA	NA	0.053	0.053	0.0042	T-RSL
Endosulfan I	959-98-8	220 N[18]	22 N[18]	NA	NA	NA	220	220	22	T-RSL
Endosulfan II	33213-65-9	220 N[18]	22 N[18]	NA	NA	NA	220	220	22	T-RSL
Endosulfan sulfate	1031-07-8	220 N[18]	22 N[18]	NA	NA	NA	220	220	22	T-RSL
Endrin	72-20-8	11 N	1.1 N	NA	NA	2	11	2	1.1	T-RSL
Endrin aldehyde	7421-93-4	11 N[19]	1.1 N[19]	NA	NA	NA	11	2	1.1	T-RSL
Endrin ketone	53494-70-5	11 N[19]	1.1 N[19]	NA	NA	NA	11	2	1.1	T-RSL
gamma-BHC (Lindane)	58-89-9	0.061 C	0.061 C	NA	NA	0.2	0.66	0.2	0.061	T-RSL
gamma-chlordane	5103-74-2	0.19 C[16]	0.19 C[16]	NA	NA	NA	2.4	2	0.19	T-RSL
Heptachlor	76-44-8	0.015 C	0.015 C	NA	NA	0.4	0.19	0.4	0.015	T-RSL
Heptachlor epoxide	1024-57-3	0.0074 C	0.0074 C	NA	NA	0.2	0.094	0.2	0.0074	T-RSL
Methoxychlor	72-43-5	180 N	18 N	NA	NA	40	180	40	18	T-RSL
Toxaphene	8001-35-2	0.061 C	0.061 C	NA	NA	3	0.77	3	0.061	T-RSL
Dioxins/Furans										
1,2,3,4,6,7,8,9-OCDD	3268-87-9	0.0017 C[20]	0.0017 C[20]	NA	NA	NA	NA	NA	0.0017	T-RSL
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.0017 C[20]	0.0017 C[20]	NA	NA	NA	NA	NA	0.0017	T-RSL
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	NA	NA	0.000052	T-RSL
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	NA	NA	0.000052	T-RSL
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	NA	NA	0.000052	T-RSL
1,2,3,4,7,8-HXCDD	39227-28-6	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,4,7,8-HXCDF	70648-26-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,6,7,8-HXCDD	57653-85-7	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,6,7,8-HXCDF	57117-44-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8,9-HXCDD	19408-74-3	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8,9-HXCDF	72918-21-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8-PECDD	40321-76-4	0.00000052 C[20]	0.00000052 C[20]	NA	NA	NA	NA	NA	0.00000052	T-RSL
1,2,3,7,8-PECDF	57117-41-6	0.000017 C[20]	0.000017 C[20]	NA	NA	NA	NA	NA	0.000017	T-RSL
2,3,4,6,7,8-HXCDF	60851-34-5	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
2,3,4,7,8-PECDF	57117-31-4	0.0000017 C[20]	0.0000017 C[20]	NA	NA	NA	NA	NA	0.0000017	T-RSL
2,3,7,8-TCDD	1746-01-6	0.00000052 C	0.00000052 C	NA	NA	0.00003	NA	NA	0.00000052	T-RSL
2,3,7,8-TCDF	51207-31-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	NA	NA	0.0000052	T-RSL
Total HpCDD	37871-00-4	NA	NA	NA	NA	NA	NA	NA	NA	None
Total HpCDF	38998-75-3	NA	NA	NA	NA	NA	NA	NA	NA	None
Total HxCDD	34465-46-8	NA	NA	NA	NA	NA	NA	NA	NA	None

Crane SWMU 11 Human Health Screening Criteria - Groundwater Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Regional Screening Level, Vapor[1] (ug/L)	Adjusted EPA Regional Screening Level, Vapor[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Total HxCDF	55684-94-1	NA	NA	NA	NA	NA	NA	NA	NA	None
Total PeCDD	36088-22-9	NA	NA	NA	NA	NA	NA	NA	NA	None
Total PeCDF	30402-15-4	NA	NA	NA	NA	NA	NA	NA	NA	None
Total TCDD	41903-57-5	NA	NA	NA	NA	NA	NA	NA	NA	None
Total TCDF	55722-27-5	NA	NA	NA	NA	NA	NA	NA	NA	None

Notes:

1 - The tapwater screening levels from the USEPA Regions 3, 6, and 9 Regional Screening Levels (T-RSLs) for Chemical Contaminants at Superfund Sites, November, 2010 available online at http://epa-prgs.ornl.gov/chemicals/index.shtml. The vapor screening levels (VAPOR) were calculated using USEPA guidance (November, 2002) and toxicity values from USEPA (November, 2010). The risk-based screening levels are based on a target hazard quotient of 1 for noncarcinogens (denoted with a "N" flag) or an incremental lifetime cancer risk (ILCR) of 1E-6 for carcinogens (denoted with a "C" flag).

2 - The USEPA T-RSL (November, 2010) and VAPOR (calculated using USEPA guidance [November, 2002] and toxicity criteria [November, 2010]) adjusted values from the risk-based screening level for non-carcinogens adjusted by dividing by 10, equivalent to a target hazard quotient of 0.1. The risk-based screening level for carcinogens is equivalent to an incremental lifetime cancer risk (ILCR) of 1E-6.

3 - USEPA Maximum Contaminant Levels (MCLs) from the 2009 Edition of the Drinking Water Standards and Health Advisories (USEPA, October 2009).

4 - Indiana Department of Environmental Management (IDEM) groundwater screening levels for direct contact and groundwater Default Closure Levels from IDEM RISC Technical Guide, January 31, 2006, Appendix 1, revised May 1, 2009.

5 - One tenth the noncarcinogenic value is less than the carinogenic value; therefore, the noncarcinogenic value is presented.

6 - Value is for 1,3-dichloropropene.

7 - Value is for xylene, mixture.

8 - Value is for 2,4-dinitrophenol.

9 - Value is for diethyl phthalate.

10 - Value is for acenapththene.

11 - Value is for pyrene.

12 - Value is for hexavalent chromium.

13 - Action level under Safe Drinking Water Act.

14 - Value is for mercuy, inorganic salts.

15 - Value is for vanadium and compounds.

16 - Value is for chlordane.

17 - Value is for alpha-BHC.

18 - Value is for endosulfan.

19 - Value is for endrin.

20 - Value is derived by multiplying criteria for 2,3,7,8-TCDD by World Health Organization Toxicity Equivalent Factor.

Abbreviations:

NA - Not available or not applicable

N - Noncarcinogen

C - carcinogen

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level
Volatile Organic Compounds										
1,1,1-Trichloroethane	71-55-6	8700 N	870 N	5000	1.9	1.9	IDEM-RDCL	38000 N	6700	280
1,1,2,2-Tetrachloroethane	79-34-5	0.56 C	0.56 C	5	0.007	0.007	IDEM-RDCL	2.8 C	8.7	0.11
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 11	76-13-1	43000 N	4300 N	NA	NA	4300	R-RSL	180000 N	NA	NA
1,1,2-Trichloroethane	79-00-5	1.1 C	1.1 C	9.4	0.03	0.03	IDEM-RDCL	5.3 C	15	0.3
1,1-Dichloroethane	75-34-3	3.3 C	3.3 C	1300	5.6	3.3	R-RSL	17 C	1700	58
1,1-Dichloroethene	75-35-4	240 N	24 N	310	0.058	0.058	IDEM-RDCL	1100 N	410	42
1,2,3- Trichlorobenzene	87-61-6	49 N	4.9 N	NA	NA	4.9	R-RSL	490 N	NA	NA
1,2,4-Trichlorobenzene	120-82-1	62 N[4]	6.2 N[4]	1800	5.3	5.3	IDEM-RDCL	270 N[4]	4900	77
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.0054 C	0.0054 C	NA	NA	0.0054	R-RSL	0.069 C	NA	NA
1,2-Dibromoethane (EDB)	106-93-4	0.034 C	0.034 C	0.3	0.00034	0.00034	IDEM-RDCL	0.17 C	0.49	0.0096
1,2-Dichlorobenzene	95-50-1	1900 N	190 N	2800	17	17	IDEM-RDCL	9800 N	3900	220
1,2-Dichloroethane	107-06-2	0.43 C	0.43 C	3.7	0.024	0.024	IDEM-RDCL	2.2 C	5.8	0.15
1,2-Dichloropropane	78-87-5	0.89 C	0.89 C	4.5	0.03	0.03	IDEM-RDCL	4.5 C	7.2	0.25
1,3-Dichlorobenzene	541-73-1	NA	NA	420	2.3	2.3	IDEM-RDCL	NA	890	8.9
1,4-Dichlorobenzene	106-46-7	2.4 C	2.4 C	42	2.2	2.2	IDEM-RDCL	12 C	73	3.4
1,4-Dioxane	123-91-1	4.9 C	4.9 C	NA	NA	4.9	R-RSL	160 C	NA	NA
2-Butanone (MEK)	78-93-3	28000 N	2800 N	44000	35	35	IDEM-RDCL	200000 N	70000	250
2-Hexanone	591-78-6	210 N	21 N	NA	NA	21	R-RSL	1400 N	NA	NA
4-Methyl-2-pentanone (MIBK)	108-10-1	5300 N	530 N	12000	20	20	IDEM-RDCL	53000 N	29000	75
Acetone	67-64-1	61000 N	6100 N	35000	28	28	IDEM-RDCL	630000 N	51000	370
Benzene	71-43-2	1.1 C	1.1 C	8.4	0.034	0.034	IDEM-RDCL	5.4 C	14	0.35
Bromochloromethane	74-97-5	NA	NA	NA	NA	NA	None	NA	NA	NA
Bromodichloromethane	75-27-4	0.27 C	0.27 C	10	0.51	0.27	R-RSL	1.4 C	17	0.51
Bromoform	75-25-2	61 C	61 C	280	0.6	0.6	IDEM-RDCL	220 C	580	2.7
Bromomethane	74-83-9	7.3 N	0.73 N	9.9	0.052	0.052	IDEM-RDCL	32 N	13	0.7
Carbon disulfide	75-15-0	820 N	82 N	900	10	10	IDEM-RDCL	3700 N	1200	82
Carbon tetrachloride	56-23-5	0.61 C	0.61 C	3.3	0.066	0.066	IDEM-RDCL	3.0 C	5.2	0.29
Chlorobenzene	108-90-7	290 N	29 N	380	1.3	1.3	IDEM-RDCL	1400 N	510	27
Chloroethane	75-00-3	15000 N	1500 N	80	0.65	0.65	IDEM-RDCL	61000 N	120	10
Chloroform	67-66-3	0.29 C	0.29 C	3	0.47	0.29	R-RSL	1.5 C	4.7	4.7
Chloromethane	74-87-3	120 N	12 N	NA	NA	12	R-RSL	500 N	NA	NA
cis-1,2-Dichloroethene	156-59-2	160 N	16 N	110	0.4	0.4	IDEM-RDCL	10000 N	140	5.8
cis-1,3-Dichloropropene	10061-01-5	1.7 C[5]	1.7 C[5]	NA	NA	1.7	R-RSL	8.1 C[5]	NA	NA
Cyclohexane	110-82-7	7000 N	700 N	7200	69	69	IDEM-RDCL	29000 N	9300	69
Dibromochloromethane	124-48-1	0.68 C	0.68 C	NA	NA	0.68	R-RSL	3.3 C	NA	NA
Dichlorodifluoromethane	75-71-8	180 N	18 N	NA	NA	18	R-RSL	780 N	NA	NA
Ethylbenzene	100-41-4	5.4 C	5.4 C	4600	13	5.4	R-RSL	27 C	6800	160
Isopropylbenzene	98-82-8	2100 N	210 N	1400	11	11	IDEM-RDCL	11000 N	1900	42
m,p-Xylenes	179601-23-1	620 N[6]	62 N[6]	NA	NA	62	R-RSL	2700 [6]	NA	NA
Methyl acetate	79-20-9	78000 N	7800 N	NA	NA	7800	R-RSL	1000000 N	NA	NA
Methylcyclohexane	108-87-2	NA	NA	NA	NA	NA	None	NA	NA	NA
Methylene chloride	75-09-2	11 C	11 C	120	0.023	0.023	IDEM-RDCL	53 C	200	1.8
Methyl-tert-butyl ether	1634-04-4	43 C	43 C	350	0.18	0.18	IDEM-RDCL	220 C	650	3.2
o-Xylene	95-47-6	3800 N	380 N	NA	NA	380	R-RSL	19000 N	NA	NA
Styrene	100-42-5	6300 N	630 N	11000	3.5	3.5	IDEM-RDCL	36000 N	16000	550

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level
Tetrachloroethene	127-18-4	0.55 C	0.55 C	9.9	0.058	0.058	IDEM-RDCL	2.6 C	16	0.64
Toluene	108-88-3	5000 N	500 N	8800	12	12	IDEM-RDCL	45000 N	16000	96
trans-1,2-Dichloroethene	156-60-5	150 N	15 N	180	0.68	0.68	IDEM-RDCL	690 N	230	14
trans-1,3-Dichloropropene	10061-02-6	1.7 C[5]	1.7 C[5]	NA	NA	1.7	R-RSL	8.1 C[5]	NA	NA
Trichloroethene	79-01-6	2.8 C	2.8 C	4.9	0.057	0.057	IDEM-RDCL	14 C	24	0.35
Trichlorofluoromethane	75-69-4	790 N	79 N	980	29	29	IDEM-RDCL	3400 N	1300	540
Vinyl chloride	75-01-4	0.060 C	0.060 C	1.5	0.013	0.013	IDEM-RDCL	1.7 C	6.4	0.027
Xylenes (total)	1330-20-7	630 N	63 N	690	170	63.0	R-RSL	2700 N	890	170
Semivolatile Organic Compounds										
1,1'-Biphenyl	92-52-4	3900 N	390 N	NA	NA	390	R-RSL	51000 N	NA	NA
1,2,4,5-Tetrachlorobenzene	95-94-3	18 N	1.8 N	NA	NA	1.8	R-RSL	180 N	NA	NA
2,2'-Oxybis(1-chloropropane)	108-60-1	4.6 C	4.6 C	30	0.027	0.027	IDEM-RDCL	22 C	61	0.26
2,3,4,6-Tetrachlorophenol	58-90-2	1800 N	180 N	NA	NA	180	R-RSL	18000 N	NA	NA
2,4,5-Trichlorophenol	95-95-4	6100 N	610 N	18000	250	250	IDEM-RDCL	62000 N	49000	690
2,4,6-Trichlorophenol	88-06-2	61 N[4]	6.1 N[4]	18	0.07	0.07	IDEM-RDCL	620 N[4]	49	0.2
2,4-Dichlorophenol	120-83-2	180 N	18 N	550	1.1	1.1	IDEM-RDCL	1800 N	1500	3
2,4-Dimethylphenol	105-67-9	1200 N	120 N	3700	9	9	IDEM-RDCL	12000 N	9800	25
2,4-Dinitrophenol	51-28-5	120 N	12 N	370	0.29	0.29	IDEM-RDCL	1200 N	980	0.82
2,4-Dinitrotoluene	121-14-2	1.6 C	1.6 C	NA	NA	1.6	R-RSL	5.5 C	NA	NA
2,6-Dinitrotoluene	606-20-2	61 N	6.1 N	NA	NA	6.1	R-RSL	620 N	NA	NA
2-Chloronaphthalene	91-58-7	6300 N	630 N	15000	42	42	IDEM-RDCL	82000 N	39000	560
2-Chlorophenol	95-57-8	390 N	39 N	360	0.75	0.75	IDEM-RDCL	5100 N	580	10
2-Methylphenol (o-Cresol)	95-48-7	3100 N	310 N	7500	14	14	IDEM-RDCL	31000 N	17000	39
2-Nitroaniline	88-74-4	610 N	61 N	550	0.67	0.67	IDEM-RDCL	6000 N	1500	1.9
2-Nitrophenol	88-75-5	120 N[7]	12 N[7]	NA	NA	12	R-RSL	1200 N[7]	NA	NA
3,3'-Dichlorobenzidine	91-94-1	1.1 C	1.1 C	9.5	0.062	0.062	IDEM-RDCL	3.8 C	31	0.21
3-Methylphenol (m-Cresol)	108-39-4	3100 N	310 N	9100	9.8	9.8	IDEM-RDCL	31000 N	25000	28
3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	None	NA	NA	NA
4,6-Dinitro-2-methylphenol	534-52-1	4.9 N	0.49 N	NA	NA	0.49	R-RSL	49 N	NA	NA
4-Bromophenyl phenyl ether	101-55-3	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methyl phenol	59-50-7	6100 N	610 N	NA	NA	610	R-RSL	62000 N	NA	NA
4-Chloroanaline	106-47-8	2.4 C	2.40 C	730	0.97	0.97	IDEM-RDCL	8.6 C	2000	2.7
4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	NA	NA	NA	None	NA	NA	NA
4-Methylphenol (p-Cresol)	106-44-5	310 N	31 N	910	1.1	1.1	IDEM-RDCL	3100 N	2500	3
4-Nitroaniline	100-01-6	24 C	24 C	NA	NA	24	R-RSL	86 C	NA	NA
4-Nitrophenol	100-02-7	NA	NA	NA	NA	NA	None	NA	NA	NA
Acetophenone	98-86-2	7800 N	780 N	NA	NA	780	R-RSL	100000 N	NA	NA
Atrazine	1912-24-9	2.1 C	2.1 C	19	0.048	0.048	IDEM-RDCL	7.5 C	63	0.21
Benzaldehyde	100-52-7	7800 N	780 N	NA	NA	780	R-RSL	100000 N	NA	NA
Bis(2-chloroethoxy)methane	111-91-1	180 N	18 N	NA	NA	18	R-RSL	1800 N	NA	NA
Bis(2-chloroethyl)ether	111-44-4	0.21 C	0.21 C	1.6	0.0007	0.0007	IDEM-RDCL	1.0 C	3	0.012
Bis(2-ethylhexyl)phthalate	117-81-7	35 C	35 C	300	300	35	R-RSL	120 C	980	980
Butyl benzyl phthalate	85-68-7	260 C	260 C	37000	310	260	R-RSL	910 C	98000	310
Caprolactam	105-60-2	31000 N	3100 N	NA	NA	3100	R-RSL	310000 N	NA	NA
Carbazole	86-74-8	NA	NA	210	5.9	5.9	IDEM-RDCL	NA	690	20

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level
Dibenzofuran	132-64-9	78 N	7.8 N	370	4.9	4.9	IDEM-RDCL	1000 N	980	65
Diethyl phthalate	84-66-2	49000 N	4900 N	150000	450	450	IDEM-RDCL	490000 N	390000	840
Dimethyl phthalate	131-11-3	49000 N[8]	4900 N[8]	1000000	1100	1100	IDEM-RDCL	490000 N[8]	1000000	1100
Di-n-butyl phthalate	84-74-2	6100 N	610 N	18000	760	610	R-RSL	62000 N	49000	760
Di-n-octyl phthalate	117-84-0	NA	NA	7300	2000	2000	IDEM-RDCL	NA	20000	2000
Hexachlorobenzene	118-74-1	0.30 C	0.30 C	2.7	2.2	0.30	R-RSL	1.1 C	8.6	3.9
Hexachlorobutadiene	87-68-3	61 N[4]	6.1 N[4]	55	24	6.1	R-RSL	22 C	150	66
Hexachlorocyclopentadiene	77-47-4	370 N	37 N	1100	400	37	R-RSL	3700 N	2900	720
Hexachloroethane	67-72-1	61 N[4]	6.1 N[4]	120	2.8	2.8	IDEM-RDCL	620 N[4]	240	7.7
Isophorone	78-59-1	510 C	510 C	4500	5.3	5.3	IDEM-RDCL	1800 C	14000	18
Nitrobenzene	98-95-3	4.8 C	4.8 C	91	0.028	0.028	IDEM-RDCL	24 C	250	0.34
N-Nitrosodi-n-propylamine	621-64-7	0.069 C	0.069 C	0.61	0.0006	0.0006	IDEM-RDCL	0.25 C	2	0.002
N-Nitrosodiphenylamine	86-30-6	99 C	99 C	870	9.7	9.7	IDEM-RDCL	350 C	2800	32
Pentachlorophenol	87-86-5	0.89 C	0.89 C	20	0.028	0.028	IDEM-RDCL	9.0 C	54	0.66
Phenol	108-95-2	18000 N	1800 N	44000	56	56	IDEM-RDCL	180000 N	96000	160
Polycyclic Aromatic Hydrocarbons										
2-Methylnaphthalene	91-57-6	310 N	31 N	630	3.1	3.1	IDEM-RDCL	4100 N	1600	42
Acenaphthene	83-32-9	3400 N	340 N	9500	130	130	IDEM-RDCL	33000 N	24000	1800
Acenaphthylene	208-96-8	3400 N[9]	340 N[9]	1100	18	18	IDEM-RDCL	170000 N[9]	2800	180
Anthracene	120-12-7	17000 N	1700 N	47000	2000	1700	R-RSL	170000 N	120000	2000
Benzo(a)anthracene	56-55-3	0.15 C	0.15 C	5	5	0.15	R-RSL	2.1 C	15	15
Benzo(a)pyrene	50-32-8	0.015 C	0.015 C	0.5	0.5	0.015	R-RSL	0.21 C	1.5	1.5
Benzo(b)fluoranthene	205-99-2	0.15 C	0.15 C	5	5	0.15	R-RSL	2.1 C	15	15
Benzo(g,h,i)perylene	191-24-2	1700 N[10]	170 N[10]	NA	NA	170	R-RSL	17000 N[10]	NA	NA
Benzo(k)fluoranthene	207-08-9	1.5 C	1.5 C	50	50	1.5	R-RSL	21 C	150	150
Chrysene	218-01-9	15 C	15 C	500	500	15	R-RSL	210 C	1500	1500
Dibenzo(a,h)anthracene	53-70-3	0.015 C	0.015 C	0.5	0.5	0.015	R-RSL	0.21 C	1.5	1.5
Fluoranthene	206-44-0	2300 N	230 N	6300	2000	230	R-RSL	22000 N	16000	2000
Fluorene	86-73-7	2300 N	230 N	6300	170	170	IDEM-RDCL	22000 N	16000	2000
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15 C	0.15 C	5	5	0.15	R-RSL	2.1 C	15	15
Naphthalene	91-20-3	3.6 C	3.60 C	3200	0.7	0.7	IDEM-RDCL	18 C	8000	170
Phenanthrene	85-01-8	1700 N[10]	170 N[10]	470	13	13	IDEM-RDCL	17000 N[10]	1200	170
Pyrene	129-00-0	1700 N	170 N	4700	2000	170	R-RSL	17000 N	12000	2000
Polychlorinated Biphenyls										
Aroclor-1016	12674-11-2	3.9 N	0.39 N	NA	NA	0.39	R-RSL	37 N[4]	NA	NA
Aroclor-1221	11104-28-2	0.14 C	0.14 C	NA	NA	0.14	R-RSL	0.54 C	NA	NA
Aroclor-1232	11141-16-5	0.14 C	0.14 C	NA	NA	0.14	R-RSL	0.54 C	NA	NA
Aroclor-1242	53469-21-9	0.22 C	0.22 C	NA	NA	0.22	R-RSL	0.74 C	NA	NA
Aroclor-1248	12672-29-6	0.22 C	0.22 C	NA	NA	0.22	R-RSL	0.74 C	NA	NA
Aroclor-1254	11097-69-1	1.1 N[4]	0.11 N[4]	NA	NA	0.11	R-RSL	0.74 C	NA	NA
Aroclor-1260	11096-82-5	0.22 C	0.22 C	NA	NA	0.22	R-RSL	0.74 C	NA	NA
Total PCBs	1336-36-3	NA	NA	NA	NA	NA	None	NA	NA	NA
Metals										
Aluminum	7429-90-5	77000 N	7700 N	NA	NA	7700	R-RSL	990000 NM	NA	NA
Antimony	7440-36-0	31 N	3.1 N	140	5.4	3.1	R-RSL	410 N	620	37

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level
Arsenic	7440-38-2	0.39 C	0.39 C	3.9	3.9	0.39	R-RSL	1.6 C	20	5.8
Barium	7440-39-3	15000 N	1500 N	63000	1600	1500	R-RSL	190000 NM	230000	10000
Beryllium	7440-41-7	160 N	16 N	680	63	16	R-RSL	2000 N	2900	2300
Cadmium	7440-43-9	70 N	7 N	12	7.5	7	R-RSL	800 N	990	77
Calcium	7440-70-2	NA	NA	NA	NA	NA	None	NA	NA	NA
Chromium	7440-47-3	0.29 C[11]	0.29 C[11]	430	38	0.29	R-RSL	5.6 C[11]	650	120
Cobalt	7440-48-4	23 N	2.3 N	NA	NA	2.3	R-RSL	300 N	NA	NA
Copper	7440-50-8	3100 N	310 N	14000	920	310	R-RSL	41000 N	62000	2900
Iron	7439-89-6	55000 N	5500 N	NA	NA	5500	R-RSL	720000 N	NA	NA
Lead	7439-92-1	400 [12]	400 [12]	400	81	81	IDEM-RDCL	800 [21]	1300	230
Magnesium	7439-95-4	NA	NA	NA	NA	NA	None	NA	NA	NA
Manganese	7439-96-5	1800 N	180 N	NA	NA	180	R-RSL	23000 N	NA	NA
Mercury	7439-97-6	23 N[13]	2.3 N[13]	NA	NA	2.3	R-RSL	310 N[13]	NA	NA
Nickel	7440-02-0	1500 N	150 N	6900	950	150	R-RSL	20000 N	31000	2700
Potassium	7440-09-7	NA	NA	NA	NA	NA	None	NA	NA	NA
Selenium	7782-49-2	390 N	39 N	1700	5.2	5.2	IDEM-RDCL	5100 N	7800	53
Silver	7440-22-4	390 N	39 N	1700	31	31	IDEM-RDCL	5100 N	7800	87
Sodium	7440-23-5	NA	NA	NA	NA	NA	None	NA	NA	NA
Thallium	7440-28-0	NA	NA	24	2.8	2.8	IDEM-RDCL	NA	110	10
Vanadium	7440-62-2	390 N[14]	39 N[14]	NA	NA	39	R-RSL	5200 N[14]	NA	NA
Zinc	7440-66-6	23000 N	2300 N	100000	10000	2300	R-RSL	310000 N	470000	10000
Pesticides										
4,4'-DDD	72-54-8	2.0 C	2.0 C	28	28	2.0	R-RSL	7.2 C	120	120
4,4'-DDE	72-55-9	1.4 C	1.4 C	20	20	1.4	R-RSL	5.1 C	86	86
4,4'-DDT	50-29-3	1.7 C	1.7 C	20	20	1.7	R-RSL	7.0 C	86	86
Aldrin	309-00-2	0.029 C	0.029 C	0.25	0.25	0.029	R-RSL	0.10 C	0.8	0.8
alpha-BHC	319-84-6	0.077 C	0.077 C	0.99	0.0072	0.0072	IDEM-RDCL	0.27 C	4	0.024
alpha-Chlordane	5103-71-9	1.6 C[16]	1.6 C[16]	17	9.6	1.6	R-RSL	6.5 C[16]	68	39
beta-BHC	319-85-7	0.27 C	0.27 C	3.3	0.026	0.026	IDEM-RDCL	0.96 C	12	0.086
delta-BHC	319-86-8	0.077 C[17]	0.077 C[17]	0.99	0.0072	0.0072	IDEM-RDCL	0.27 C[17]	4	0.024
Dieldrin	60-57-1	0.030 C	0.030 C	0.27	0.046	0.03	R-RSL	0.11 C	0.86	0.15
Endosulfan I	959-98-8	370 N[18]	37 N[18]	1100	20	20	IDEM-RDCL	3700 N[18]	2900	46
Endosulfan II	33213-65-9	370 N[18]	37 N[18]	1100	20	20	IDEM-RDCL	3700 N[18]	2900	46
Endosulfan sulfate	1031-07-8	370 N[18]	37 N[18]	1100	20	20	IDEM-RDCL	3700 N[18]	2900	46
Endrin	72-20-8	18 N	1.8 N	55	0.99	0.99	IDEM-RDCL	180 N	150	15
Endrin aldehyde	7421-93-4	18 N[19]	1.8 N	55	0.99	0.99	IDEM-RDCL	180 N[19]	150	15
Endrin ketone	53494-70-5	18 N[19]	1.8 N	55	0.99	0.99	IDEM-RDCL	180 N[19]	150	15
gamma-BHC (Lindane)	58-89-9	0.52 C	0.52 C	4.8	0.0094	0.0094	IDEM-RDCL	2.1 C	19	0.1
gamma-chlordane	5103-74-2	1.6 C[16]	1.6 C[16]	17	9.6	1.6	R-RSL	6.5 C[16]	68	39
Heptachlor	76-44-8	0.11 C	0.11 C	0.93	0.93	0.11	R-RSL	0.38 C	2.9	2.9
Heptachlor epoxide	1024-57-3	0.053 C	0.053 C	0.47	0.47	0.053	R-RSL	0.19 C	1.5	1
Methoxychlor	72-43-5	310 N	31 N	910	160	31	R-RSL	3100 N	2500	180
Toxaphene	8001-35-2	0.44 C	0.44 C	3.9	3.9	0.44	R-RSL	1.6 C	12	12
Dioxins/Furans										

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level
1,2,3,4,6,7,8,9-OCDD	3268-87-9	0.015 C[20]	0.015 C[20]	NA	NA	0.015	R-RSL	0.06 C[20]	NA	NA
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.015 C[20]	0.015 C[20]	NA	NA	0.015	R-RSL	0.06 C[20]	NA	NA
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.00045 C[20]	0.00045 C[20]	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.00045 C[20]	0.00045 C[20]	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.00045 C[20]	0.00045 C[20]	NA	NA	0.00045	R-RSL	0.0018 C[20]	NA	NA
1,2,3,4,7,8-HXCDD	39227-28-6	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,4,7,8-HXCDF	70648-26-9	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,6,7,8-HXCDD	57653-85-7	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,6,7,8-HXCDF	57117-44-9	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,7,8,9-HXCDD	19408-74-3	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,7,8,9-HXCDF	72918-21-9	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
1,2,3,7,8-PECDD	40321-76-4	0.0000045 C[20]	4.5E-06 C[20]	NA	NA	0.0000045	R-RSL	0.000018 C[20]	NA	NA
1,2,3,7,8-PECDF	57117-41-6	0.00015 C[20]	0.00015 C[20]	NA	NA	0.00015	R-RSL	0.00060 C[20]	NA	NA
2,3,4,6,7,8-HXCDF	60851-34-5	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
2,3,4,7,8-PECDF	57117-31-4	0.000015 C[20]	1.5E-05 C[20]	NA	NA	0.000015	R-RSL	0.000060 C[20]	NA	NA
2,3,7,8-TCDD	1746-01-6	0.0000045 C	4.5E-06 C	NA	NA	0.0000045	R-RSL	0.000018 C	NA	NA
2,3,7,8-TCDF	51207-31-9	0.000045 C[20]	4.5E-05 C[20]	NA	NA	0.000045	R-RSL	0.00018 C[20]	NA	NA
Total HpCDD	37871-00-4	NA	NA	NA	NA	NA	None	NA	NA	NA
Total HpCDF	38998-75-3	NA	NA	NA	NA	NA	None	NA	NA	NA
Total HxCDD	34465-46-8	NA	NA	NA	NA	NA	None	NA	NA	NA
Total HxCDF	55684-94-1	NA	NA	NA	NA	NA	None	NA	NA	NA
Total PeCDD	36088-22-9	NA	NA	NA	NA	NA	None	NA	NA	NA
Total PeCDF	30402-15-4	NA	NA	NA	NA	NA	None	NA	NA	NA
Total TCDD	41903-57-5	NA	NA	NA	NA	NA	None	NA	NA	NA
Total TCDF	55722-27-5	NA	NA	NA	NA	NA	None	NA	NA	NA

Notes:

1 - The residential direct contact (R-RSL) and risk-based migration to groundwater soil screening levels (RBSSL) from the USEPA Regions 3, 6, and 9 Regional Screening Levels for Chemical Contaminants at Superfund Sites, November, 2010 available online at http://epa-prgs.ornl.gov/chemicals/index.shtml. The risk-based screening levels are based on a target hazard quotient of 1 for noncarcinogens (denoted with a "N" flag) or an incremental lifetime cancer risk (ILCR) of 1E-6 for carcinogens (denoted with a "C" flag). (Industrial criteria are also presented for information purposes.)

2 - The USEPA R-RSL (November, 2010) residential soil screening level for noncarcinogens adjusted by dividing by 10, equivalent to a target hazard quotient of 0.1. The residential soil screening level for carcinogens (not adjusted) is equivalent to an incremental lifetime cancer risk (ILCR) of 1E-6. The USEPA RBSSL (November, 2010) is adjusted for a dilution attenuation factor (DAF) of 20.

3 - Indiana Department of Environmental Management (IDEM) residential soil direct contact screening levels, migration to groundwater screening levels, and Residential Default Closure Levels from IDEM RISC Technical Guide, January 31, 2006, Appendix 1, revised May 1, 2009. (Industrial criteria are also presented for information purposes.)

4 - One tenth the noncarcinogenic value is less than the carinogenic value; therefore, the noncarcinogenic value is presented.

5 - Value is for 1,3-dichloropropene.

6 - Value is for xylene, mixture.

7 - Value is for 2,4-dinitrophenol.

8 - Value is for diethyl phthalate.

9 - Value is for acenapththene.

10 - Value is for pyrene.

NSA Crane SWMU 11 Human Health Screening Criteria - Sediment Samples

Analyte	CAS Number	EPA Regional Screening Level, Residential Soil [1] (mg/kg)	Adjusted EPA Regional Screening Level, Residential Soil [2] (mg/kg)	2009 IDEM RISC Residential Closure Levels for Soil (mg/kg) [3]		Lowest Human Health Criterion	Lowest Human Health Criterion Reference	EPA Regional Screening Level, Industrial Soil [1] (mg/kg)	2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) [3]	
				Residential Direct Contact	Residential Default Closure Level				Industrial Direct Contact	Industrial Default Closure Level

11 - Value is for hexavalent chromium.
12 - Office of Solid Waste and Emergency Response soil screening level (EPA, 1994b)
13 - Value is for mercuy, inorganic salts.
14 - Value is for vanadium and compounds.
15 - Calculated from the EPA website (http://epa-prgs.ornl.gov/cgi-bin/chemicals/csl_search).
16 - Value is for chlordane.
17 - Value is for alpha-BHC.
18 - Value is for endosulfan.
19 - Value is for endrin.
20 - Value is derived by multiplying criteria for 2,3,7,8-TCDD by World Health Organization Toxicity Equivalent Factor.
21 - USEPA Technical Review Workgroup for Lead. Guidance Document. “Frequently Asked Question (FAQs) on the Adult‑Lead Model.” October 2010. http://www.epa.gov/superfund/lead/almfaq.htm

Abbreviations:
NA - Not applicable or not available
C - Carcinogen
EPA - U.S. Environmental Protection Agency
N - Noncarcinogen
NA - Not available
SSL - Soil Screening Level

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	9100 N	910 N	200	3800	200	200	IDEM G-DCL
1,1,2,2-Tetrachloroethane	79-34-5	0.067 C	0.067 C	NA	0.9	0.9	0.067	T-RSL
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	59000 N	5900 N	NA	NA	NA	5900	T-RSL
1,1,2-Trichloroethane	79-00-5	0.24 C	0.24 C	5	3.2	5	0.24	T-RSL
1,1-Dichloroethane	75-34-3	2.4 C	2.4 C	NA	990	990	2.4	T-RSL
1,1-Dichloroethene	75-35-4	340 N	34 N	7	430	7	7	IDEM G-DCL
1,2,3- Trichlorobenzene	87-61-6	29 N	2.9 N	NA	NA	NA	2.9	T-RSL
1,2,4-Trichlorobenzene	120-82-1	4.1 N[5]	0.41 N[5]	70	9.5	70	0.41	T-RSL
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.00032 C	0.00032 C	0.2	NA	NA	0.00032	T-RSL
1,2-Dibromoethane (EDB)	106-93-4	0.0065 C	0.0065 C	0.05	0.086	0.05	0.0065	T-RSL
1,2-Dichlorobenzene	95-50-1	370 N	37 N	600	480	600	37	T-RSL
1,2-Dichloroethane	107-06-2	0.15 C	0.15 C	5	2	5	0.15	T-RSL
1,2-Dichloropropane	78-87-5	0.39 C	0.39 C	5	2.6	5	0.39	T-RSL
1,3-Dichlorobenzene	541-73-1	NA	NA	NA	80	80	80	IDEM G-DCL
1,4-Dichlorobenzene	106-46-7	0.43 C	0.43 C	75	8	75	0.43	T-RSL
1,4-Dioxane	123-91-1	0.67 C	0.67 C	NA	NA	NA	0.67	T-RSL
2-Butanone (MEK)	78-93-3	7100 N	710 N	NA	8400	8400	710	T-RSL
2-Hexanone	591-78-6	47 N	4.7 N	NA	NA	NA	4.7	T-RSL
4-Methyl-2-pentanone (MIBK)	108-10-1	2000 N	200 N	NA	2200	2200	200	T-RSL
Acetone	67-64-1	22000 N	2200 N	NA	6900	6900	2200	T-RSL
Benzene	71-43-2	0.41 C	0.41 C	5	5.5	5	0.41	T-RSL
Bromochloromethane	74-97-5	NA	NA	NA	NA	NA	NA	None
Bromodichloromethane	75-27-4	0.12 C	0.12 C	80	2.9	80	0.12	T-RSL
Bromoform	75-25-2	8.5 C	8.5 C	80	110	80	8.5	T-RSL
Bromomethane	74-83-9	8.7 N	0.87 N	NA	11	11	0.87	T-RSL
Carbon disulfide	75-15-0	1000 N	100 N	NA	1300	1300	100	T-RSL
Carbon tetrachloride	56-23-5	0.44 C	0.44 C	5	2.6	5	0.44	T-RSL
Chlorobenzene	108-90-7	91 N	9.1 N	100	130	100	9.1	T-RSL
Chloroethane	75-00-3	21000 N	2100 N	NA	62	62	62	IDEM G-DCL
Chloroform	67-66-3	0.19 C	0.19 C	80	2.8	80	0.19	T-RSL
Chloromethane	74-87-3	190 N	19 N	NA	NA	NA	19	T-RSL
cis-1,2-Dichloroethene	156-59-2	73 N	7.3 N	70	77	70	7.3	T-RSL
cis-1,3-Dichloropropene	10061-01-5	40 N[6]	4 N[6]	NA	NA	NA	4	T-RSL
Cyclohexane	110-82-7	13000 N	1300 N	NA	13000	13000	1300	T-RSL
Dibromochloromethane	124-48-1	0.15 C	0.15 C	80	NA	NA	0.15	T-RSL
Dichlorodifluoromethane	75-71-8	390 N	39 N	NA	NA	NA	39	T-RSL
Ethylbenzene	100-41-4	1.5 C	1.5 C	700	1600	700	1.5	T-RSL
Isopropylbenzene	98-82-8	680 N	68 N	NA	830	830	68	T-RSL

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
m,p-Xylenes	179601-23-1	200 N[7]	20 N[7]	10000	NA	NA	20	T-RSL
Methyl acetate	79-20-9	37000 N	3700 N	NA	NA	NA	3700	T-RSL
Methylcyclohexane	108-87-2	NA	NA	NA	NA	NA	NA	None
Methylene chloride	75-09-2	4.8 C	4.8 C	5	63	5	4.8	T-RSL
Methyl-tert-butyl ether	1634-04-4	12 C	12 C	NA	40	40	12	T-RSL
o-Xylene	95-47-6	1200 N	120 N	NA	NA	NA	120	T-RSL
Styrene	100-42-5	1600 N	160 N	100	2000	100	100	IDEM G-DCL
Tetrachloroethene	127-18-4	0.11 C	0.11 C	5	6.5	5	0.11	T-RSL
Toluene	108-88-3	2300 N	230 N	1000	2400	1000	230	T-RSL
trans-1,2-Dichloroethene	156-60-5	110 N	11 N	100	150	100	11	T-RSL
trans-1,3-Dichloropropene	10061-02-6	40 N[6]	4.0 N[6]	NA	NA	NA	4.0	T-RSL
Trichloroethene	79-01-6	2.0 C	2.0 C	5	2.8	5	2.0	T-RSL
Trichlorofluoromethane	75-69-4	1300 N	130 N	NA	1700	1700	130	T-RSL
Vinyl chloride	75-01-4	0.016 C	0.016 C	2	0.53	2	0.016	T-RSL
Xylenes (total)	1330-20-7	200 N	20 N	10000	270	10000	20	T-RSL
Semivolatile Organic Compounds								
1,1'-Biphenyl	92-52-4	1800 N	180 N	NA	NA	NA	180	T-RSL
1,2,4,5-Tetrachlorobenzene	95-94-3	11 N	1.1 N	NA	NA	NA	1.1	T-RSL
2,2'-Oxybis(1-chloropropane)	108-60-1	0.32 C	0.32 C	NA	4.2	4.2	0.32	T-RSL
2,3,4,6-Tetrachlorophenol	58-90-2	1100 N	110 N	NA	NA	NA	110	T-RSL
2,4,5-Trichlorophenol	95-95-4	3700 N	370 N	NA	3600	3600	370	T-RSL
2,4,6-Trichlorophenol	88-06-2	37 N[5]	3.7 N[5]	NA	3.6	3.6	3.6	IDEM G-DCL
2,4-Dichlorophenol	120-83-2	110 N	11 N	NA	110	110	11	T-RSL
2,4-Dimethylphenol	105-67-9	730 N	73 N	NA	730	730	73	T-RSL
2,4-Dinitrophenol	51-28-5	73 N	7.3 N	NA	73	73	7.3	T-RSL
2,4-Dinitrotoluene	121-14-2	0.22 C	0.22 C	NA	NA	NA	0.22	T-RSL
2,6-Dinitrotoluene	606-20-2	37 N	3.7 N	NA	NA	NA	3.7	T-RSL
2-Chloronaphthalene	91-58-7	2900 N	290 N	NA	610	610	290	T-RSL
2-Chlorophenol	95-57-8	180 N	18 N	NA	38	38	18	T-RSL
2-Methylphenol (o-Cresol)	95-48-7	1800 N	180 N	NA	1800	1800	180	T-RSL
2-Nitroaniline	88-74-4	370 N	37 N	NA	110	110	37	T-RSL
2-Nitrophenol	88-75-5	73 N[8]	7.3 N[8]	NA	NA	NA	7.3	T-RSL
3,3'-Dichlorobenzidine	91-94-1	0.15 C	0.15 C	NA	1.9	1.9	0.15	T-RSL
3-Nitroaniline	99-09-2	NA	NA	NA	NA	NA	NA	None
4,6-Dinitro-2-methylphenol	534-52-1	2.9 N	0.29 N	NA	NA	NA	0.29	T-RSL
4-Bromophenyl phenyl ether	101-55-3	NA	NA	NA	NA	NA	NA	None
4-Chloro-3-methyl phenol	59-50-7	3700 N	370 N	NA	NA	NA	370	T-RSL
4-Chloroanaline	106-47-8	0.34 C	0.34 C	NA	150	150	0.34	T-RSL
4-Chlorophenyl phenyl ether	7005-72-3	NA	NA	NA	NA	NA	NA	None

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
4-Methylphenol (p-Cresol)	106-44-5	180 N	18 N	NA	180	180	18	T-RSL
4-Nitroaniline	100-01-6	3.4 C	3.4 C	NA	NA	NA	3.4	T-RSL
4-Nitrophenol	100-02-7	NA	NA	NA	NA	NA	NA	None
Acetophenone	98-86-2	3700 N	370 N	NA	NA	NA	370	T-RSL
Atrazine	1912-24-9	0.29 C	0.29 C	3	3.9	3	0.29	T-RSL
Benzaldehyde	100-52-7	3700 N	370 N	NA	NA	NA	370	T-RSL
Bis(2-chloroethoxy)methane	111-91-1	110 N	11 N	NA	NA	NA	11	T-RSL
Bis(2-chloroethyl)ether	111-44-4	0.012 C	0.012 C	NA	0.15	0.15	0.012	T-RSL
Bis(2-ethylhexyl)phthalate	117-81-7	4.8 C	4.8 C	6	61	6	4.8	T-RSL
Butyl benzyl phthalate	85-68-7	35 C	35 C	NA	7300	2700	35	T-RSL
Caprolactam	105-60-2	18000 N	1800 N	NA	NA	NA	1800	T-RSL
Carbazole	86-74-8	NA	NA	NA	43	43	43	IDEM G-DCL
Dibenzofuran	132-64-9	37 N	3.7 N	NA	15	15	3.7	T-RSL
Diethyl phthalate	84-66-2	29000 N	2900 N	NA	29000	29000	2900	T-RSL
Dimethyl phthalate	131-11-3	29000 N[9]	2900 N[9]	NA	360000	360000	2900	T-RSL
Di-n-butyl phthalate	84-74-2	3700 N	370 N	NA	3600	3600	370	T-RSL
Di-n-octyl phthalate	117-84-0	NA	NA	NA	1500	20	20	IDEM G-DCL
Hexachlorobenzene	118-74-1	0.042 C	0.042 C	1	0.53	1	0.042	T-RSL
Hexachlorobutadiene	87-68-3	0.86 C	0.86 C	NA	11	11	0.86	T-RSL
Hexachlorocyclopentadiene	77-47-4	220 N	22 N	50	220	50	22	T-RSL
Hexachloroethane	67-72-1	37 N[5]	3.7 N[5]	NA	36	36	3.7	T-RSL
Isophorone	78-59-1	71 C	71 C	NA	900	900	71	T-RSL
Nitrobenzene	98-95-3	0.12 C	0.12 C	NA	4.3	4.3	0.12	T-RSL
N-Nitrosodi-n-propylamine	621-64-7	0.0096 C	0.0096 C	NA	0.12	0.12	0.0096	T-RSL
N-Nitrosodiphenylamine	86-30-6	14 C	14 C	NA	170	170	14	T-RSL
Pentachlorophenol	87-86-5	0.17 C	0.17 C	1	7.1	1	0.17	T-RSL
Phenol	108-95-2	11000 N	1100 N	NA	11000	11000	1100	T-RSL
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	150 N	15 N	NA	31	31	15	T-RSL
Acenaphthene	83-32-9	2200 N	220 N	NA	460	460	220	T-RSL
Acenaphthylene	208-96-8	2200 N[10]	220 N[10]	NA	71	71	71	IDEM G-DCL
Anthracene	120-12-7	11000 N	1100 N	NA	2300	2300	1100	T-RSL
Benzo(a)anthracene	56-55-3	0.029 C	0.029 C	NA	1.2	1.2	0.029	T-RSL
Benzo(a)pyrene	50-32-8	0.0029 C	0.0029 C	0.2	0.12	0.2	0.0029	T-RSL
Benzo(b)fluoranthene	205-99-2	0.029 C	0.029 C	NA	1.2	1.2	0.029	T-RSL
Benzo(g,h,i)perylene	191-24-2	1100 N[11]	110 N[11]	NA	NA	NA	110	T-RSL
Benzo(k)fluoranthene	207-08-9	0.29 C	0.29 C	NA	12	12	0.29	T-RSL
Chrysene	218-01-9	2.9 C	2.9 C	NA	120	120	2.9	T-RSL
Dibenzo(a,h)anthracene	53-70-3	0.0029 C	0.0029 C	NA	0.12	0.12	0.0029	T-RSL

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Fluoranthene	206-44-0	1500 N	150 N	NA	1500	1500	150	T-RSL
Fluorene	86-73-7	1500 N	150 N	NA	310	310	150	T-RSL
Indeno(1,2,3-c,d)pyrene	193-39-5	0.029 C	0.029 C	NA	1.2	1.2	0.029	T-RSL
Naphthalene	91-20-3	0.14 C	0.14 C	NA	8.3	8.3	0.14	T-RSL
Phenanthrene	85-01-8	1100 N[11]	110 N[11]	NA	23	23	23	IDEM G-DCL
Pyrene	129-00-0	1100 N	110 N	NA	1100	1100	110	T-RSL
Metals								
Aluminum	7429-90-5	37000 N	3700 N	NA	NA	NA	3700	T-RSL
Antimony	7440-36-0	15 N	1.5 N	6	15	6	1.5	T-RSL
Arsenic	7440-38-2	0.045 C	0.045 C	10	0.57	10	0.045	T-RSL
Barium	7440-39-3	7300 N	730 N	2000	7300	2000	730	T-RSL
Beryllium	7440-41-7	73 N	7.3 N	4	73	4	4	IDEM G-DCL
Cadmium	7440-43-9	18 N	1.8 N	5	18	5	1.8	T-RSL
Calcium	7440-70-2	NA	NA	NA	NA	NA	NA	None
Chromium	7440-47-3	0.043 C[12]	0.043 C[12]	100	NA	NA	0.043	T-RSL
Cobalt	7440-48-4	11 N	1.1 N	NA	NA	NA	1.1	T-RSL
Copper	7440-50-8	1500 N	150 N	1300	1500	1300	150	T-RSL
Iron	7439-89-6	26000 N	2600 N	NA	NA	NA	2600	T-RSL
Lead	7439-92-1	15 [13]	15 [13]	15	15	15	15	T-RSL
Magnesium	7439-95-4	NA	NA	NA	NA	NA	NA	None
Manganese	7439-96-5	880 N	88 N	NA	NA	NA	88	T-RSL
Mercury	7439-97-6	11 N[14]	1.1 N[14]	2	NA	NA	1.1	T-RSL
Nickel	7440-02-0	730 N	73 N	NA	730	730	73	T-RSL
Potassium	7440-09-7	NA	NA	NA	NA	NA	NA	None
Selenium	7782-49-2	180 N	18 N	50	180	50	18	T-RSL
Silver	7440-22-4	180 N	18 N	NA	180	180	18	T-RSL
Sodium	7440-23-5	NA	NA	NA	NA	NA	NA	None
Thallium	7440-28-0	NA	NA	2	2.6	2	2	IDEM G-DCL
Vanadium	7440-62-2	180 N[15]	18 N[15]	NA	NA	NA	18	T-RSL
Zinc	7440-66-6	11000 N	1100 N	NA	11000	11000	1100	T-RSL
Pesticides								
4,4'-DDD	72-54-8	0.28 C	0.28 C	NA	3.5	3.5	0.28	T-RSL
4,4'-DDE	72-55-9	0.20 C	0.20 C	NA	2.5	2.5	0.20	T-RSL
4,4'-DDT	50-29-3	0.20 C	0.20 C	NA	2.5	2.5	0.20	T-RSL
Aldrin	309-00-2	0.0040 C	0.0040 C	NA	0.05	0.05	0.0040	T-RSL
alpha-BHC	319-84-6	0.011 C	0.011 C	NA	0.14	0.14	0.011	T-RSL
alpha-Chlordane	5103-71-9	0.19 C[16]	0.19 C[16]	NA	2.4	2	0.19	T-RSL
beta-BHC	319-85-7	0.037 C	0.037 C	NA	0.47	0.47	0.037	T-RSL
delta-BHC	319-86-8	0.011 C[17]	0.011 C[17]	NA	0.14	0.14	0.011	T-RSL

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[1] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[2] (ug/L)	EPA Maximum Contaminant Level (ug/L)[3]	IDEM GW[4] (ug/L)	IDEM GW Default Closure Level[4] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Dieldrin	60-57-1	0.0042 C	0.0042 C	NA	0.053	0.053	0.0042	T-RSL
Endosulfan I	959-98-8	220 N[18]	22 N[18]	NA	220	220	22	T-RSL
Endosulfan II	33213-65-9	220 N[18]	22 N[18]	NA	220	220	22	T-RSL
Endosulfan sulfate	1031-07-8	220 N[18]	22 N[18]	NA	220	220	22	T-RSL
Endrin	72-20-8	11 N	1.1 N	2	11	2	1.1	T-RSL
Endrin aldehyde	7421-93-4	11 N[19]	1.1 N[19]	NA	11	2	1.1	T-RSL
Endrin ketone	53494-70-5	11 N[19]	1.1 N[19]	NA	11	2	1.1	T-RSL
gamma-BHC (Lindane)	58-89-9	0.061 C	0.061 C	0.2	0.66	0.2	0.061	T-RSL
gamma-chlordane	5103-74-2	0.19 C[16]	0.19 C[16]	NA	2.4	2	0.19	T-RSL
Heptachlor	76-44-8	0.015 C	0.015 C	0.4	0.19	0.4	0.015	T-RSL
Heptachlor epoxide	1024-57-3	0.0074 C	0.0074 C	0.2	0.094	0.2	0.0074	T-RSL
Methoxychlor	72-43-5	180 N	18 N	40	180	40	18	T-RSL
Toxaphene	8001-35-2	0.061 C	0.061 C	3	0.77	3	0.061	T-RSL
Dioxins/Furans								
1,2,3,4,6,7,8,9-OCDD	3268-87-9	0.0017 C[20]	0.0017 C[20]	NA	NA	NA	0.0017	T-RSL
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.0017 C[20]	0.0017 C[20]	NA	NA	NA	0.0017	T-RSL
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	0.000052	T-RSL
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	0.000052	T-RSL
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.000052 C[20]	0.000052 C[20]	NA	NA	NA	0.000052	T-RSL
1,2,3,4,7,8-HXCDD	39227-28-6	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,4,7,8-HXCDF	70648-26-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,6,7,8-HXCDD	57653-85-7	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,6,7,8-HXCDF	57117-44-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8,9-HXCDD	19408-74-3	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8,9-HXCDF	72918-21-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
1,2,3,7,8-PECDD	40321-76-4	0.00000052 C[20]	0.00000052 C[20]	NA	NA	NA	0.00000052	T-RSL
1,2,3,7,8-PECDF	57117-41-6	0.000017 C[20]	0.000017 C[20]	NA	NA	NA	0.000017	T-RSL
2,3,4,6,7,8-HXCDF	60851-34-5	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
2,3,4,7,8-PECDF	57117-31-4	0.0000017 C[20]	0.0000017 C[20]	NA	NA	NA	0.0000017	T-RSL
2,3,7,8-TCDD	1746-01-6	0.00000052 C	0.00000052 C	0.00003	NA	NA	0.00000052	T-RSL
2,3,7,8-TCDF	51207-31-9	0.0000052 C[20]	0.0000052 C[20]	NA	NA	NA	0.0000052	T-RSL
Total HpCDD	37871-00-4	NA	NA	NA	NA	NA	NA	None
Total HpCDF	38998-75-3	NA	NA	NA	NA	NA	NA	None
Total HxCDD	34465-46-8	NA	NA	NA	NA	NA	NA	None
Total HxCDF	55684-94-1	NA	NA	NA	NA	NA	NA	None
Total PeCDD	36088-22-9	NA	NA	NA	NA	NA	NA	None
Total PeCDF	30402-15-4	NA	NA	NA	NA	NA	NA	None
Total TCDD	41903-57-5	NA	NA	NA	NA	NA	NA	None

Crane SWMU 11 Human Health Screening Criteria - Surface Water Samples

Analyte	CAS Number	EPA Regional Screening Level, Tap Water[(1)] (ug/L)	Adjusted EPA Regional Screening Level, Tap Water[(2)] (ug/L)	EPA Maximum Contaminant Level (ug/L)[(3)]	IDEM GW[(4)] (ug/L)	IDEM GW Default Closure Level[(4)] (ug/L)	Minimum Criteria (ug/L)	Minimum Criterion Reference
Total TCDF	55722-27-5	NA	NA	NA	NA	NA	NA	None

Notes:

1 - The surface water screening levels from the USEPA Regions 3, 6, and 9 Tapwater Regional Screening Levels (T-RSLs) for Chemical Contaminants at Superfund Sites, November, 2010 available online at http://epa-prgs.ornl.gov/chemicals/index.shtml. The risk-based screening levels are based on a target hazard quotient of 1 for noncarcinogens (denoted with a "N" flag) or an incremental lifetime cancer risk (ILCR) of 1E-6 for carcinogens (denoted with a "C" flag).

2 - The USEPA T-RSL (November, 2010) adjusted values from the risk-based screening level for noncarcinogens adjusted by dividing by 10, equivalent to a target hazard quotient of 0.1. The risk-based screening level for carcinogens is equivalent to an incremental lifetime cancer risk (ILCR) of 1E-6.

3 - USEPA Maximum Contaminant Levels (MCLs) from the 2009 Edition of the Drinking Water Standards and Health Advisories (USEPA, October 2009).

4 - Indiana Department of Environmental Management (IDEM) groundwater screening levels for direct contact and groundwater Default Closure Levels from IDEM RISC Technical Guide, January 31, 2006, Appendix 1, revised May 1, 2009.

5 - One tenth the noncarcinogenic value is less than the carinogenic value; therefore, the noncarcinogenic value is presented.

6 - Value is for 1,3-dichloropropene.

7 - Value is for xylene, mixture.

8 - Value is for 2,4-dinitrophenol.

9 - Value is for diethyl phthalate.

10 - Value is for acenapththene.

11 - Value is for pyrene.

12 - Value is for hexavalent chromium.

13 - Action level under Safe Drinking Water Act.

14 - Value is for mercuy, inorganic salts.

15 - Value is for vanadium and compounds.

16 - Value is for chlordane.

17 - Value is for alpha-BHC.

18 - Value is for endosulfan.

19 - Value is for endrin.

20 - Value is derived by multiplying criteria for 2,3,7,8-TCDD by World Health Organization Toxicity Equivalent Factor.

Abbreviations:

NA - Not available or not applicable

N - Noncarcinogen

C - carcinogen

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Soil Samples

Analyte	CAS Number	Ecological Soil Screening Level [1] (mg/kg)	Source of Ecological Soil Screening Level	EPA Eco SSL (mg/kg)	EPA R5 Eco Soil (mg/kg)	NOAA SQUIRT (mg/kg)
Volatile Organic Compounds						
1,1,1-Trichloroethane	71-55-6	29.8	R5 ECOS	NA	**29.8**	**29.8**
1,1,2,2-Tetrachloroethane	79-34-5	0.127	R5 ECOS	NA	**0.127**	**0.127**
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	--	--	NA	NA	NA
1,1,2-Trichloroethane	79-00-5	28.6	R5 ECOS	NA	**28.6**	**28.6**
1,1-Dichloroethane	75-34-3	20.1	R5 ECOS	NA	**20.1**	**20.1**
1,1-Dichloroethene	75-35-4	8.28	R5 ECOS	NA	**8.28**	**8.28**
1,2,3- Trichlorobenzene	87-61-6	11.1	R5 ECOS	NA	**11.1**	**11.1**
1,2,4-Trichlorobenzene	120-82-1	11.1	R5 ECOS	NA	**11.1**	**11.1**
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.0352	R5 ECOS	NA	**0.0352**	**0.0352**
1,2-Dibromoethane (EDB)	106-93-4	1.23	R5 ECOS	NA	**1.23**	**1.23**
1,2-Dichlorobenzene	95-50-1	2.96	R5 ECOS	NA	**2.96**	**2.96**
1,2-Dichloroethane	107-06-2	21.2	R5 ECOS	NA	**21.2**	**21.2**
1,2-Dichloropropane	78-87-5	32.7	R5 ECOS	NA	**32.7**	**32.7**
1,3-Dichlorobenzene	541-73-1	37.7	R5 ECOS	NA	**37.7**	**37.7**
1,4-Dichlorobenzene	106-46-7	0.546	R5 ECOS	NA	**0.546**	**0.546**
1,4-Dioxane	123-91-1	2.05	R5 ECOS	NA	**2.05**	**2.05**
2-Butanone (MEK)	78-93-3	89.6	R5 ECOS	NA	**89.6**	**89.6**
2-Hexanone	591-78-6	12.6	R5 ECOS	NA	**12.6**	**12.6**
4-Methyl-2-pentanone (MIBK)	108-10-1	443	R5 ECOS	NA	**443**	**443**
Acetone	67-64-1	2.5	R5 ECOS	NA	**2.5**	**2.5**
Benzene	71-43-2	0.255	R5 ECOS	NA	**0.255**	**0.255**
Bromochloromethane	74-97-5	--	--	NA	NA	NA
Bromodichloromethane	75-27-4	0.54	R5 ECOS	NA	**0.54**	**0.54**
Bromoform	75-25-2	15.9	R5 ECOS	NA	**15.9**	**15.9**
Bromomethane	74-83-9	0.235	R5 ECOS	NA	**0.235**	**0.235**
Carbon disulfide	75-15-0	0.0941	R5 ECOS	NA	**0.0941**	**0.0941**
Carbon tetrachloride	56-23-5	2.98	R5 ECOS	NA	**2.98**	**2.98**
Chlorobenzene	108-90-7	13.1	R5 ECOS	NA	**13.1**	**13.1**
Chloroethane	75-00-3	--	--	NA	NA	NA
Chloroform	67-66-3	1.19	R5 ECOS	NA	**1.19**	**1.19**
Chloromethane	74-87-3	10.4	R5 ECOS	NA	**10.4**	**10.4**
cis-1,2-Dichloroethene	156-59-2	0.78373	R5 ECOS	NA	**0.784**	**0.784**
cis-1,3-Dichloropropene	10061-01-5	0.398	R5 ECOS	NA	**0.398**	**0.398**
Cyclohexane	110-82-7	--	--	NA	NA	NA
Dibromochloromethane	124-48-1	--	--	NA	NA	NA
Dichlorodifluoromethane	75-71-8	39.5	R5 ECOS	NA	**39.5**	**39.5**
Ethylbenzene	100-41-4	5.16	R5 ECOS	NA	**5.16**	**5.16**
Isopropylbenzene	98-82-8	--	--	NA	NA	NA
m,p-Xylenes	179601-23-1	10	R5 ECOS	NA	**10**	**10**
Methyl acetate	79-20-9	--	--	NA	NA	NA
Methylcyclohexane	108-87-2	--	--	NA	NA	NA
Methylene chloride	75-09-2	4.05	R5 ECOS	NA	**4.05**	**4.05**
Methyl-tert-butyl ether	1634-04-4	--	--	NA	NA	NA
o-Xylene	95-47-6	10	R5 ECOS	NA	**10**	**10**
Styrene	100-42-5	4.69	R5 ECOS	NA	**4.69**	**4.69**
Tetrachloroethene	127-18-4	9.92	R5 ECOS	NA	**9.92**	**9.92**
Toluene	108-88-3	5.45	R5 ECOS	NA	**5.45**	**5.45**
trans-1,2-Dichloroethene	156-60-5	0.784	R5 ECOS	NA	**0.784**	**0.784**
trans-1,3-Dichloropropene	10061-02-6	0.398	R5 ECOS	NA	**0.398**	**0.398**
Trichloroethene	79-01-6	12.4	R5 ECOS	NA	**12.4**	**12.4**
Trichlorofluoromethane	75-69-4	16.4	R5 ECOS	NA	**16.4**	**16.4**
Vinyl chloride	75-01-4	0.646	R5 ECOS	NA	**0.646**	**0.646**
Xylenes (total)	1330-20-7	10	R5 ECOS	NA	**10**	**10**
Semivolatile Organic Compounds						
1,1'-Biphenyl	92-52-4	60	R5 ECOS	NA	**60**	**60**
1,2,4,5-Tetrachlorobenzene	95-94-3	2.02	R5 ECOS	NA	**2.02**	**2.02**
2,2'-Oxybis(1-chloropropane)	108-60-1	19.9	R5 ECOS	NA	**19.9**	**19.9**
2,3,4,6-Tetrachlorophenol	58-90-2	0.199	R5 ECOS	NA	**0.199**	**0.199**
2,4,5-Trichlorophenol	95-95-4	14.1	R5 ECOS	NA	**14.1**	**14.1**
2,4,6-Trichlorophenol	88-06-2	9.94	R5 ECOS	NA	**9.94**	**9.94**
2,4-Dichlorophenol	120-83-2	87.5	R5 ECOS	NA	**87.5**	**87.5**
2,4-Dimethylphenol	105-67-9	0.01	R5 ECOS	NA	**0.01**	**0.01**
2,4-Dinitrophenol	51-28-5	0.0609	R5 ECOS	NA	**0.0609**	**0.0609**
2,4-Dinitrotoluene	121-14-2	1.28	R5 ECOS	NA	**1.28**	**1.28**
2,6-Dinitrotoluene	606-20-2	0.0328	R5 ECOS	NA	**0.0328**	**0.0328**
2-Chloronaphthalene	91-58-7	0.0122	R5 ECOS	NA	**0.0122**	**0.0122**
2-Chlorophenol	95-57-8	0.243	R5 ECOS	NA	**0.243**	**0.243**
2-Methylphenol (o-Cresol)	95-48-7	40.4	R5 ECOS	NA	**40.4**	**40.4**
2-Nitroaniline	88-74-4	74.1	R5 ECOS	NA	**74.1**	**74.1**
2-Nitrophenol	88-75-5	1.6	R5 ECOS	NA	**1.6**	**1.6**
3,3'-Dichlorobenzidine	91-94-1	0.646	R5 ECOS	NA	**0.646**	**0.646**
3-Nitroaniline	99-09-2	3.16	R5 ECOS	NA	**3.16**	**3.16**
4,6-Dinitro-2-methylphenol	534-52-1	0.144	R5 ECOS	NA	**0.144**	**0.144**
4-Bromophenyl phenyl ether	101-55-3	--	--	NA	NA	NA
4-Chloro-3-methyl phenol	59-50-7	7.95	R5 ECOS	NA	**7.95**	**7.95**
4-Chloroanaline	106-47-8	1.1	R5 ECOS	NA	**1.1**	**1.1**
4-Chlorophenyl phenyl ether	7005-72-3	--	--	NA	NA	NA
4-Methylphenol (p-Cresol)	106-44-5	163	R5 ECOS	NA	**163**	**163**

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Soil Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (mg/kg)	Source of Ecological Soil Screening Level	EPA Eco SSL (mg/kg)	EPA R5 Eco Soil (mg/kg)	NOAA SQUIRT (mg/kg)
4-Nitroaniline	100-01-6	21.9	R5 ECOS	NA	**21.9**	**21.9**
4-Nitrophenol	100-02-7	5.12	R5 ECOS	NA	**5.12**	**5.12**
Acetophenone	98-86-2	300	R5 ECOS	NA	**300**	**300**
Atrazine	1912-24-9	--	--	NA	NA	NA
Benzaldehyde	100-52-7	--	--	NA	NA	NA
Bis(2-chloroethoxy)methane	111-91-1	0.302	R5 ECOS	NA	**0.302**	**0.302**
Bis(2-chloroethyl)ether	111-44-4	23.7	R5 ECOS	NA	**23.7**	**23.7**
Bis(2-ethylhexyl)phthalate	117-81-7	0.925	R5 ECOS	NA	**0.925**	**0.925**
Butyl benzyl phthalate	85-68-7	0.239	R5 ECOS	NA	**0.239**	**0.239**
Caprolactam	105-60-2	--	--	NA	NA	NA
Carbazole	86-74-8	--	--	NA	NA	NA
Dibenzofuran	132-64-9	--	--	NA	NA	NA
Diethyl phthalate	84-66-2	24.8	R5 ECOS	NA	**24.8**	**24.8**
Dimethyl phthalate	131-11-3	734	R5 ECOS	NA	**734**	**734**
Di-n-butyl phthalate	84-74-2	0.15	R5 ECOS	NA	**0.15**	**0.15**
Di-n-octyl phthalate	117-84-0	709	R5 ECOS	NA	**709**	**709**
Hexachlorobenzene	118-74-1	0.199	R5 ECOS	NA	**0.199**	**0.199**
Hexachlorobutadiene	87-68-3	0.0398	R5 ECOS	NA	**0.0398**	**0.0398**
Hexachlorocyclopentadiene	77-47-4	0.755	R5 ECOS	NA	**0.755**	**0.755**
Hexachloroethane	67-72-1	0.596	R5 ECOS	NA	**0.596**	**0.596**
Isophorone	78-59-1	139	R5 ECOS	NA	**139**	**139**
Nitrobenzene	98-95-3	1.31	R5 ECOS	NA	**1.31**	**1.31**
N-Nitrosodi-n-propylamine	621-64-7	0.544	R5 ECOS	NA	**0.544**	**0.544**
N-Nitrosodiphenylamine	86-30-6	0.545	R5 ECOS	NA	**0.545**	**0.545**
Pentachlorophenol	87-86-5	0.119	R5 ECOS	NA	**0.119**	**0.119**
Phenol	108-95-2	120	R5 ECOS	NA	**120**	**120**
Polycyclic Aromatic Hydrocarbons						
2-Methylnaphthalene	91-57-6	29	Eco SSL	**29**	3.24	3.24
Acenaphthene	83-32-9	29	Eco SSL	**29**	682	682
Acenaphthylene	208-96-8	29	Eco SSL	**29**	682	682
Anthracene	120-12-7	29	Eco SSL	**29**	1480	1480
Benzo(a)anthracene	56-55-3	1.1	Eco SSL	**1.1**	5.21	5.21
Benzo(a)pyrene	50-32-8	1.1	Eco SSL	**1.1**	1.52	1.52
Benzo(b)fluoranthene	205-99-2	1.1	Eco SSL	**1.1**	59.8	59.8
Benzo(g,h,i)perylene	191-24-2	1.1	Eco SSL	**1.1**	119	119
Benzo(k)fluoranthene	207-08-9	1.1	Eco SSL	**1.1**	148	148
Chrysene	218-01-9	1.1	Eco SSL	**1.1**	4.73	4.73
Dibenzo(a,h)anthracene	53-70-3	1.1	Eco SSL	**1.1**	18.4	18.4
Fluoranthene	206-44-0	29	Eco SSL	**29**	122	122
Fluorene	86-73-7	29	Eco SSL	**29**	122	122
Indeno(1,2,3-c,d)pyrene	193-39-5	1.1	Eco SSL	**1.1**	109	109
Naphthalene	91-20-3	29	Eco SSL	**29**	0.0994	0.0994
Phenanthrene	85-01-8	29	Eco SSL	**29**	45.7	45.7
Pyrene	129-00-0	1.1	Eco SSL	**1.1**	78.5	78.5
Polychlorinated Biphenyls						
Aroclor-1016	12674-11-2	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1221	11104-28-2	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1232	11141-16-5	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1242	53469-21-9	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1248	12672-29-6	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1254	11097-69-1	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Aroclor-1260	11096-82-5	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Total PCBs	1336-36-3	0.000332	R5 ECOS	NA	**0.000332**	**0.000332**
Metals						
Aluminum	7429-90-5	NA	Eco SSL	**NA**	**NA**	**NA**
Antimony	7440-36-0	0.27	Eco SSL	**0.27**	0.142	0.142
Arsenic	7440-38-2	18	Eco SSL	**18**	5.7	5.7
Barium	7440-39-3	330	Eco SSL	**330**	1.04	1.04
Beryllium	7440-41-7	21	Eco SSL	**21**	1.06	1.06
Cadmium	7440-43-9	0.36	Eco SSL	**0.36**	0.00222	0.00222
Calcium	7440-70-2	--	--	NA	NA	NA
Chromium	7440-47-3	26	Eco SSL	**26**	0.4	0.4
Cobalt	7440-48-4	13	Eco SSL	**13**	0.14	0.14
Copper	7440-50-8	28	Eco SSL	**28**	5.4	5.4
Iron	7439-89-6	NA	Eco SSL	**NA**	**NA**	**NA**
Lead	7439-92-1	11	Eco SSL	**11**	0.0537	0.0537
Magnesium	7439-95-4	--	--	NA	NA	NA
Manganese	7439-96-5	220	Eco SSL	**220**	**220**	**220**
Mercury	7439-97-6	0.1	R5 ECOS	NA	**0.1**	**0.1**
Nickel	7440-02-0	38	Eco SSL	**38**	13.6	13.6
Potassium	7440-09-7	--	--	NA	NA	NA
Selenium	7782-49-2	0.52	Eco SSL	**0.52**	**0.52**	**0.52**
Silver	7440-22-4	4.2	Eco SSL	**4.2**	2	2
Sodium	7440-23-5	--	--	NA	NA	NA
Thallium	7440-28-0	0.0569	R5 ECOS	NA	**0.0569**	**0.0569**
Vanadium	7440-62-2	7.8	Eco SSL	**7.8**	1.59	1.59
Zinc	7440-66-6	46	Eco SSL	**46**	6.62	6.62
Pesticides						
4,4'-DDD	72-54-8	0.758	R5 ECOS	NA	**0.758**	

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Soil Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (mg/kg)	Source of Ecological Soil Screening Level	EPA Eco SSL (mg/kg)	EPA R5 Eco Soil (mg/kg)	NOAA SQUIRT (mg/kg)
4,4'-DDE	72-55-9	0.596	R5 ECOS	NA	**0.596**	
4,4'-DDT	50-29-3	0.021	Eco SSL	**0.021**	0.0035	
Aldrin	309-00-2	0.00332	R5 ECOS	NA	**0.00332**	
alpha-BHC	319-84-6	0.0994	R5 ECOS	NA	**0.0994**	
alpha-Chlordane	5103-71-9	0.224	R5 ECOS	NA	**0.224**	
beta-BHC	319-85-7	0.00398	R5 ECOS	NA	**0.00398**	
delta-BHC	319-86-8	9.94	R5 ECOS	NA	**9.94**	
Dieldrin	60-57-1	0.0049	Eco SSL	**0.0049**	0.00238	
Endosulfan I	959-98-8	0.119	R5 ECOS	NA	**0.119**	
Endosulfan II	33213-65-9	0.119	R5 ECOS	NA	**0.119**	
Endosulfan sulfate	1031-07-8	0.0358	R5 ECOS	NA	**0.0358**	
Endrin	72-20-8	0.0101	R5 ECOS	NA	**0.0101**	
Endrin aldehyde	7421-93-4	0.0105	R5 ECOS	NA	**0.0105**	
Endrin ketone	53494-70-5	--	--	NA	NA	
gamma-BHC (Lindane)	58-89-9	0.005	R5 ECOS	NA	**0.005**	
gamma-chlordane	5103-74-2	0.224	R5 ECOS	NA	**0.224**	
Heptachlor	76-44-8	0.00598	R5 ECOS	NA	**0.00598**	
Heptachlor epoxide	1024-57-3	0.152	R5 ECOS	NA	**0.152**	
Methoxychlor	72-43-5	0.0199	R5 ECOS	NA	**0.0199**	
Toxaphene	8001-35-2	0.119	R5 ECOS	NA	**0.119**	
Dioxins/Furans						
1,2,3,4,6,7,8,9-OCDD	3268-87-9	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,4,6,7,8,9-OCDF	39001-02-0	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,4,6,7,8-HPCDD	35822-46-9	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,4,6,7,8-HPCDF	67562-39-4	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,4,7,8,9-HPCDF	55673-89-7	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,4,7,8-HXCDD	39227-28-6	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,4,7,8-HXCDF	70648-26-9	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,6,7,8-HXCDD	57653-85-7	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,6,7,8-HXCDF	57117-44-9	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,7,8,9-HXCDD	19408-74-3	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,7,8,9-HXCDF	72918-21-9	**0.0000386**	R5 ECOS	NA	**0.0000386**	
1,2,3,7,8-PECDD	40321-76-4	**0.000000199**	R5 ECOS	NA	**0.000000199**	
1,2,3,7,8-PECDF	57117-41-6	**0.0000386**	R5 ECOS	NA	**0.0000386**	
2,3,4,6,7,8-HXCDF	60851-34-5	**0.0000386**	R5 ECOS	NA	**0.0000386**	
2,3,4,7,8-PECDF	57117-31-4	**0.0000386**	R5 ECOS	NA	**0.0000386**	
2,3,7,8-TCDD	1746-01-6	**0.000000199**	R5 ECOS	NA	**0.000000199**	
2,3,7,8-TCDF	51207-31-9	**0.0000386**	R5 ECOS	NA	**0.0000386**	
Total HpCDD	37871-00-4	--	NA	NA	NA	
Total HpCDF	38998-75-3	--	NA	NA	NA	
Total HxCDD	34465-46-8	--	NA	NA	NA	
Total HxCDF	55684-94-1	--	NA	NA	NA	
Total PeCDD	36088-22-9	--	NA	NA	NA	
Total PeCDF	30402-15-4	--	NA	NA	NA	
Total TCDD	41903-57-5	--	NA	NA	NA	
Total TCDF	55722-27-5	--	NA	NA	NA	

1- The following hierarchy was used for selecting the Ecological Screening level in order of preference:
USEPA Ecological Soil Screening Levels (Eco SSL) (EPA, 2003, 2005, 2006, 2007). The lower of the plant, invertebrate, or wildlife Eco SSL is selected as the screening level.
The lower of the Oak Ridge National Laboratory (ORNL) plant or invertebrate benchmarks (Efroymson, et al., 1997a, 1997b).
USEPA Region 5 Ecological Soil Screening Levels (R5 ECOS) (USEPA, 2005).
Lowest of National Oceanographic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQUIRT) surface soil benchmarks (Buchman, 2008).

Shaded cells are values that were selected as the overall ecological soil screening level.

NSA Crane SWMU 11 Ecological Screening Criteria - Sediment Samples

Analyte	CAS Number	Ecological Soil Screening Level [1] (mg/kg)	Source of Ecological Soil Screening Level	EPA R5 Eco SED (mg/kg)	EPA R3 BTAG SED (mg/kg)	NOAA SQUIRT (mg/kg)
Volatile Organic Compounds						
1,1,1-Trichloroethane	71-55-6	0.213	R5 ECOSED	0.213	0.0302	
1,1,2,2-Tetrachloroethane	79-34-5	0.85	R5 ECOSED	0.85	1.36	
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	--	None	NA	NA	
1,1,2-Trichloroethane	79-00-5	0.518	R5 ECOSED	0.518	1.24	
1,1-Dichloroethane	75-34-3	0.000575	R5 ECOSED	0.000575	NA	
1,1-Dichloroethene	75-35-4	0.0194	R5 ECOSED	0.0194	0.031	
1,2,3- Trichlorobenzene	87-61-6	0.858	R3 BTAG SED	NA	0.858	
1,2,4-Trichlorobenzene	120-82-1	5.062	R5 ECOSED	5.062	2.1	
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	--	None	NA	NA	
1,2-Dibromoethane (EDB)	106-93-4	--	None	NA	NA	
1,2-Dichlorobenzene	95-50-1	0.294	R5 ECOSED	0.294	0.0165	
1,2-Dichloroethane	107-06-2	0.26	R5 ECOSED	0.26	NA	
1,2-Dichloropropane	78-87-5	0.333	R5 ECOSED	0.333	NA	
1,3-Dichlorobenzene	541-73-1	1.315	R5 ECOSED	1.315	4.43	
1,4-Dichlorobenzene	106-46-7	0.318	R5 ECOSED	0.318	0.599	
1,4-Dioxane	123-91-1	0.119	R5 ECOSED	0.119	NA	
2-Butanone (MEK)	78-93-3	0.0424	R5 ECOSED	0.0424	NA	
2-Hexanone	591-78-6	0.0582	R5 ECOSED	0.0582	NA	
4-Methyl-2-pentanone (MIBK)	108-10-1	0.0251	R5 ECOSED	0.0251	NA	
Acetone	67-64-1	0.0099	R5 ECOSED	0.0099	NA	
Benzene	71-43-2	0.142	R5 ECOSED	0.142	NA	
Bromochloromethane	74-97-5	--	None	NA	NA	
Bromodichloromethane	75-27-4	--	None	NA	NA	
Bromoform	75-25-2	0.492	R5 ECOSED	0.492	0.654	
Bromomethane	74-83-9	0.00137	R5 ECOSED	0.00137	NA	
Carbon disulfide	75-15-0	0.0239	R5 ECOSED	0.0239	0.000851	
Carbon tetrachloride	56-23-5	1.45	R5 ECOSED	1.45	0.0642	
Chlorobenzene	108-90-7	0.291	R5 ECOSED	0.291	0.00842	
Chloroethane	75-00-3	--	None	NA	NA	
Chloroform	67-66-3	0.121	R5 ECOSED	0.121	NA	
Chloromethane	74-87-3	--	None	NA	NA	
cis-1,2-Dichloroethene	156-59-2	0.20894	R5 ECOSED	0.20894	NA	
cis-1,3-Dichloropropene	10061-01-5	0.00296	R5 ECOSED	0.00296	NA	
Cyclohexane	110-82-7	--	None	NA	NA	
Dibromochloromethane	124-48-1	--	None	NA	NA	
Dichlorodifluoromethane	75-71-8	--	None	NA	NA	
Ethylbenzene	100-41-4	0.175	R5 ECOSED	0.175	1.1	
Isopropylbenzene	98-82-8	--	None	NA	0.086	
m,p-Xylenes	179601-23-1	0.433	R5 ECOSED	**0.433**	65	
Methyl acetate	79-20-9	--	None	NA	NA	
Methylcyclohexane	108-87-2	--	None	NA	NA	
Methylene chloride	75-09-2	0.159	R5 ECOSED	0.159	NA	
Methyl-tert-butyl ether	1634-04-4	--	None	NA	NA	
o-Xylene	95-47-6	0.433	R5 ECOSED	0.433	NA	
Styrene	100-42-5	0.254	R5 ECOSED	0.254	0.559	
Tetrachloroethene	127-18-4	0.99	R5 ECOSED	0.99	0.468	
Toluene	108-88-3	1.22	R5 ECOSED	1.22	NA	
trans-1,2-Dichloroethene	156-60-5	0.654	R5 ECOSED	0.654	1.05	
trans-1,3-Dichloropropene	10061-02-6	0.00296	R5 ECOSED	0.00296	NA	
Trichloroethene	79-01-6	0.112	R5 ECOSED	0.112	0.0969	
Trichlorofluoromethane	75-69-4	--	None	NA	NA	
Vinyl chloride	75-01-4	0.202	R5 ECOSED	0.202	NA	
Xylenes (total)	1330-20-7	0.433	R5 ECOSED	**0.433**	65	
Semivolatile Organic Compounds						
1,1'-Biphenyl	92-52-4	**1.22**	R3 BTAG SED	NA	**1.22**	
1,2,4,5-Tetrachlorobenzene	95-94-3	1.252	R5 ECOSED	1.252	1.09	
2,2'-Oxybis(1-chloropropane)	108-60-1	--	None	NA	NA	
2,3,4,6-Tetrachlorophenol	58-90-2	0.129	R5 ECOSED	0.129	0.284	
2,4,5-Trichlorophenol	95-95-4	--	None	NA	NA	
2,4,6-Trichlorophenol	88-06-2	0.208	R5 ECOSED	0.208	0.213	
2,4-Dichlorophenol	120-83-2	0.0817	R5 ECOSED	0.0817	0.117	
2,4-Dimethylphenol	105-67-9	0.304	R5 ECOSED	0.304	0.029	
2,4-Dinitrophenol	51-28-5	0.00621	R5 ECOSED	0.00621	NA	
2,4-Dinitrotoluene	121-14-2	0.0144	R5 ECOSED	0.0144	0.0416	
2,6-Dinitrotoluene	606-20-2	0.0398	R5 ECOSED	0.0398	NA	
2-Chloronaphthalene	91-58-7	0.417	R5 ECOSED	0.417	NA	
2-Chlorophenol	95-57-8	0.0319	R5 ECOSED	0.0319	0.0312	
2-Methylphenol (o-Cresol)	95-48-7	0.0554	R5 ECOSED	0.0554	NA	
2-Nitroaniline	88-74-4	--	None	NA	NA	
2-Nitrophenol	88-75-5	--	None	NA	NA	
3,3'-Dichlorobenzidine	91-94-1	0.127	R5 ECOSED	0.127	0.127	
3-Nitroaniline	99-09-2	--	None	NA	NA	
4,6-Dinitro-2-methylphenol	534-52-1	0.104	R5 ECOSED	0.104	NA	
4-Bromophenyl phenyl ether	101-55-3	1.55	R5 ECOSED	1.55	1.23	

NSA Crane SWMU 11 Ecological Screening Criteria - Sediment Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (mg/kg)	Source of Ecological Soil Screening Level	EPA R5 Eco SED (mg/kg)	EPA R3 BTAG SED (mg/kg)	NOAA SQUIRT (mg/kg)
4-Chloro-3-methyl phenol	59-50-7	0.388	R5 ECOSED	0.388	NA	
4-Chloroanaline	106-47-8	0.146	R5 ECOSED	0.146	NA	
4-Chlorophenyl phenyl ether	7005-72-3	--	None	NA	NA	
4-Methylphenol (p-Cresol)	106-44-5	0.0202	R5 ECOSED	0.0202	0.67	
4-Nitroaniline	100-01-6	--	None	NA	NA	
4-Nitrophenol	100-02-7	0.0133	R5 ECOSED	0.0133	NA	
Acetophenone	98-86-2	--	None	NA	NA	
Atrazine	1912-24-9	0.00662	R3 BTAG SED	NA	0.00662	
Benzaldehyde	100-52-7	--	None	NA	NA	
Bis(2-chloroethoxy)methane	111-91-1	--	None	NA	NA	
Bis(2-chloroethyl)ether	111-44-4	3.52	R5 ECOSED	3.52	NA	
Bis(2-ethylhexyl)phthalate	117-81-7	0.182	R5 ECOSED	0.182	0.18	
Butyl benzyl phthalate	85-68-7	1.97	R5 ECOSED	1.97	10.9	
Caprolactam	105-60-2	--	None	NA	NA	
Carbazole	86-74-8	--	None	NA	NA	
Dibenzofuran	132-64-9	0.449	R5 ECOSED	0.449	0.415	
Diethyl phthalate	84-66-2	0.295	R5 ECOSED	0.295	0.603	
Dimethyl phthalate	131-11-3	--	None	NA	NA	
Di-n-butyl phthalate	84-74-2	1.114	R5 ECOSED	1.114	6.47	
Di-n-octyl phthalate	117-84-0	40.6	R5 ECOSED	40.6	NA	
Hexachlorobenzene	118-74-1	0.02	R5 ECOSED	0.02	0.02	
Hexachlorobutadiene	87-68-3	0.0265	R5 ECOSED	0.0265	NA	
Hexachlorocyclopentadiene	77-47-4	0.901	R5 ECOSED	0.901	NA	
Hexachloroethane	67-72-1	0.584	R5 ECOSED	0.584	1.027	
Isophorone	78-59-1	0.432	R5 ECOSED	0.432	NA	
Nitrobenzene	98-95-3	0.145	R5 ECOSED	0.145	NA	
N-Nitrosodi-n-propylamine	621-64-7	--	None	NA	NA	
N-Nitrosodiphenylamine	86-30-6	2.68	R3 BTAG SED	NA	2.68	
Pentachlorophenol	87-86-5	23	R5 ECOSED	23	0.504	
Phenol	108-95-2	0.0491	R5 ECOSED	0.0491	0.42	
Polycyclic Aromatic Hydrocarbons						
2-Methylnaphthalene	91-57-6	0.0202	R5 ECOSED	**0.0202**	**0.0202**	
Acenaphthene	83-32-9	0.00671	R5 ECOSED	**0.00671**	0.0067	
Acenaphthylene	208-96-8	0.00587	R5 ECOSED	**0.00587**	0.0059	
Anthracene	120-12-7	0.0572	R5 ECOSED	**0.0572**	**0.0572**	
Benzo(a)anthracene	56-55-3	0.108	R5 ECOSED	**0.108**	**0.108**	
Benzo(a)pyrene	50-32-8	0.15	R5 ECOSED	**0.15**	**0.15**	
Benzo(b)fluoranthene	205-99-2	10.4	R5 ECOSED	**10.4**	NA	
Benzo(g,h,i)perylene	191-24-2	0.17	R5 ECOSED	**0.17**	**0.17**	
Benzo(k)fluoranthene	207-08-9	0.24	R5 ECOSED	**0.24**	**0.24**	
Chrysene	218-01-9	0.166	R5 ECOSED	**0.166**	**0.166**	
Dibenzo(a,h)anthracene	53-70-3	0.033	R5 ECOSED	**0.033**	**0.033**	
Fluoranthene	206-44-0	0.423	R5 ECOSED	**0.423**	**0.423**	
Fluorene	86-73-7	0.0774	R5 ECOSED	**0.0774**	**0.0774**	
Indeno(1,2,3-c,d)pyrene	193-39-5	0.2	R5 ECOSED	**0.2**	0.017	
Naphthalene	91-20-3	0.176	R5 ECOSED	**0.176**	**0.176**	
Phenanthrene	85-01-8	0.204	R5 ECOSED	**0.204**	**0.204**	
Pyrene	129-00-0	0.195	R5 ECOSED	**0.195**	**0.195**	
Polychlorinated Biphenyls						
Aroclor-1016	12674-11-2	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1221	11104-28-2	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1232	11141-16-5	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1242	53469-21-9	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1248	12672-29-6	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1254	11097-69-1	0.0598	R5 ECOSED	**0.0598**	NA	
Aroclor-1260	11096-82-5	0.0598	R5 ECOSED	**0.0598**	NA	
Total PCBs	1336-36-3	0.0598	R5 ECOSED	**0.0598**	**0.0598**	
Metals						
Aluminum	7429-90-5	--	None	NA	NA	
Antimony	7440-36-0	2	R3 BTAG SED	NA	2	
Arsenic	7440-38-2	9.79	R5 ECOSED	9.79	9.8	
Barium	7440-39-3	--	None	NA	NA	
Beryllium	7440-41-7	--	None	NA	NA	
Cadmium	7440-43-9	0.99	R5 ECOSED	0.99	0.99	
Calcium	7440-70-2	--	None	NA	NA	
Chromium	7440-47-3	43.4	R5 ECOSED	43.4	43.4	
Cobalt	7440-48-4	50	R5 ECOSED	50	50	
Copper	7440-50-8	31.6	R5 ECOSED	31.6	31.6	
Iron	7439-89-6	20000	R3 BTAG SED	NA	20000	
Lead	7439-92-1	35.8	R5 ECOSED	35.8	35.8	
Magnesium	7439-95-4	--	None	NA	NA	
Manganese	7439-96-5	460	R3 BTAG SED	NA	460	
Mercury	7439-97-6	0.174	R5 ECOSED	0.174	0.18	
Nickel	7440-02-0	22.7	R5 ECOSED	22.7	22.7	
Potassium	7440-09-7	--	None	NA	NA	

NSA Crane SWMU 11 Ecological Screening Criteria - Sediment Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (mg/kg)	Source of Ecological Soil Screening Level	EPA R5 Eco SED (mg/kg)	EPA R3 BTAG SED (mg/kg)	NOAA SQUIRT (mg/kg)
Selenium	7782-49-2	2	R3 BTAG SED	NA	2	
Silver	7440-22-4	0.5	R5 ECOSED	0.5	1	
Sodium	7440-23-5	--	None	NA	NA	
Thallium	7440-28-0	--	None	NA	NA	
Vanadium	7440-62-2	--	None	NA	NA	
Zinc	7440-66-6	121	R5 ECOSED	121	121	
Pesticides						
4,4'-DDD	72-54-8	**0.00488**	R5 ECOSED	**0.00488**	**0.00488**	
4,4'-DDE	72-55-9	**0.00316**	R5 ECOSED	**0.00316**	**0.00316**	
4,4'-DDT	50-29-3	**0.00416**	R5 ECOSED	**0.00416**	NA	
Aldrin	309-00-2	**0.002**	R5 ECOSED	**0.002**	**0.002**	
alpha-BHC	319-84-6	**0.006**	R5 ECOSED	**0.006**	**0.006**	
alpha-Chlordane	5103-71-9	**0.0045**	R5 ECOSED	**0.0045**	NA	
beta-BHC	319-85-7	**0.005**	R5 ECOSED	**0.005**	**0.005**	
delta-BHC	319-86-8	**71.5**	R5 ECOSED	**71.5**	6.4	
Dieldrin	60-57-1	**0.0019**	R5 ECOSED	**0.0019**	**0.0019**	
Endosulfan I	959-98-8	**0.00326**	R5 ECOSED	**0.00326**	0.0029	
Endosulfan II	33213-65-9	**0.00194**	R5 ECOSED	**0.00194**	0.014	
Endosulfan sulfate	1031-07-8	**0.0346**	R5 ECOSED	**0.0346**	0.0054	
Endrin	72-20-8	**0.00222**	R5 ECOSED	**0.00222**	**0.00222**	
Endrin aldehyde	7421-93-4	**0.48**	R5 ECOSED	**0.48**	NA	
Endrin ketone	53494-70-5	--	None	NA	NA	
gamma-BHC (Lindane)	58-89-9	**0.00237**	R5 ECOSED	**0.00237**	**0.00237**	
gamma-chlordane	5103-74-2	**0.0045**	R5 ECOSED	**0.0045**	NA	
Heptachlor	76-44-8	**0.0006**	R5 ECOSED	**0.0006**	0.068	
Heptachlor epoxide	1024-57-3	**0.00247**	R5 ECOSED	**0.00247**	**0.00247**	
Methoxychlor	72-43-5	**0.0136**	R5 ECOSED	**0.0136**	0.0187	
Toxaphene	8001-35-2	**7.70E-05**	R5 ECOSED	**7.70E-05**	0.0001	
Dioxins/Furans						
1,2,3,4,6,7,8,9-OCDD	3268-87-9	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,4,6,7,8,9-OCDF	39001-02-0	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,4,6,7,8-HPCDD	35822-46-9	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,4,6,7,8-HPCDF	67562-39-4	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,4,7,8,9-HPCDF	55673-89-7	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,4,7,8-HXCDD	39227-28-6	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,4,7,8-HXCDF	70648-26-9	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,6,7,8-HXCDD	57653-85-7	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,6,7,8-HXCDF	57117-44-9	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,7,8,9-HXCDD	19408-74-3	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,7,8,9-HXCDF	72918-21-9	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
1,2,3,7,8-PECDD	40321-76-4	**0.0000033**	R5 ECOSED	**0.0000033**	NA	
1,2,3,7,8-PECDF	57117-41-6	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
2,3,4,6,7,8-HXCDF	60851-34-5	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
2,3,4,7,8-PECDF	57117-31-4	**1.29E-08**	R5 ECOSED	**1.29E-08**	NA	
2,3,7,8-TCDD	1746-01-6	**1.20E-07**	R5 ECOSED	**1.20E-07**	0.00000085	
2,3,7,8-TCDF	51207-31-9	--	None	NA	NA	
Total HpCDD	37871-00-4	--	None	NA	NA	
Total HpCDF	38998-75-3	--	None	NA	NA	
Total HxCDD	34465-46-8	--	None	NA	NA	
Total HxCDF	55684-94-1	--	None	NA	NA	
Total PeCDD	36088-22-9	--	None	NA	NA	
Total PeCDF	30402-15-4	--	None	NA	NA	
Total TCDD	41903-57-5	--	None	NA	NA	
Total TCDF	55722-27-5	--	None	NA	NA	

1- The following hierarchy was used for selecting the Ecological Screening level in order of preference:
USEPA Region 5 Ecological Sediment Screening Levels (R5 ECOSED) (USEPA, 2005).
USEPA Region 3 BTAG Ecological Sediment Screening Levels (R3 BTAG SED) (USEPA, 2005).
Lowest of National Oceanographic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQUIRT) surface water benchmarks (Buchman, 2008).

Shaded cells are values that were selected as the overall ecological sediment screening level.

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Water Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (ug/L)	Source of Ecological Soil Screening Level	EPA R5 Eco SW (ug/L)	EPA R3 BTAG FW SW (ug/L)	EPA T-RSL Adjusted (ug/L)	IDEM SW (ug/L)	NOAA SQUIRT (ug/L)
Volatile Organic Compounds								
1,1,1-Trichloroethane	71-55-6	76	R5 ECO SW	76	11	910	NA	
1,1,2,2-Tetrachloroethane	79-34-5	380	R5 ECO SW	380	610	0.067	NA	
1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)	76-13-1	5900	T-RSL	NA	NA	5900	NA	
1,1,2-Trichloroethane	79-00-5	500	R5 ECO SW	500	1200	0.24	NA	
1,1-Dichloroethane	75-34-3	47	R5 ECO SW	47	47	2.4	NA	
1,1-Dichloroethene	75-35-4	65	R5 ECO SW	65	25	34	NA	
1,2,3- Trichlorobenzene	87-61-6	8	R3 BTAG SW	NA	8	2.9	NA	
1,2,4-Trichlorobenzene	120-82-1	30	R5 ECO SW	30	24	0.41	NA	
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8	0.00032	T-RSL	NA	NA	0.00032	NA	
1,2-Dibromoethane (EDB)	106-93-4	0.0065	T-RSL	NA	NA	0.0065	NA	
1,2-Dichlorobenzene	95-50-1	14	R5 ECO SW	14	0.7	37	NA	
1,2-Dichloroethane	107-06-2	910	R5 ECO SW	910	100	0.15	NA	
1,2-Dichloropropane	78-87-5	360	R5 ECO SW	360	NA	0.39	NA	
1,3-Dichlorobenzene	541-73-1	38	R5 ECO SW	38	150	NA	NA	
1,4-Dichlorobenzene	106-46-7	9.4	R5 ECO SW	9.4	26	0.43	NA	
1,4-Dioxane	123-91-1	22000	R5 ECO SW	22000	NA	6.1	NA	
2-Butanone (MEK)	78-93-3	2200	R5 ECO SW	2200	14000	710	NA	
2-Hexanone	591-78-6	99	R5 ECO SW	99	99	NA	NA	
4-Methyl-2-pentanone (MIBK)	108-10-1	170	R5 ECO SW	170	170	200	NA	
Acetone	67-64-1	1700	R5 ECO SW	1700	1500	2200	NA	
Benzene	71-43-2	114	R5 ECO SW	114	370	0.41	NA	
Bromochloromethane	74-97-5	--	None	NA	NA	NA	NA	
Bromodichloromethane	75-27-4	0.12	T-RSL	NA	NA	0.12	NA	
Bromoform	75-25-2	230	R5 ECO SW	230	320	8.5	NA	
Bromomethane	74-83-9	16	R5 ECO SW	16	NA	0.87	NA	
Carbon disulfide	75-15-0	15	R5 ECO SW	15	0.92	100	NA	
Carbon tetrachloride	56-23-5	240	R5 ECO SW	240	13.3	0.44	NA	
Chlorobenzene	108-90-7	47	R5 ECO SW	47	1.3	9.1	NA	
Chloroethane	75-00-3	2100	T-RSL	NA	NA	2100	NA	
Chloroform	67-66-3	140	R5 ECO SW	140	1.8	0.19	NA	
Chloromethane	74-87-3	19	T-RSL	NA	NA	19	NA	
cis-1,2-Dichloroethene	156-59-2	37	T-RSL	NA	NA	37	NA	
cis-1,3-Dichloropropene	10061-01-5	7.9	R5 ECO SW	7.9	NA	4	NA	
Cyclohexane	110-82-7	1300	T-RSL	NA	NA	1300	NA	
Dibromochloromethane	124-48-1	0.15	T-RSL	NA	NA	0.15	NA	
Dichlorodifluoromethane	75-71-8	39	T-RSL	NA	NA	39	NA	
Ethylbenzene	100-41-4	14	R5 ECO SW	14	90	1.5	NA	
Isopropylbenzene	98-82-8	2.6	R3 BTAG SW	NA	2.6	68	NA	
m,p-Xylenes	179601-23-1	27	T-RSL	27	NA	20	NA	
Methyl acetate	79-20-9	3700	T-RSL	NA	NA	3700	NA	
Methylcyclohexane	108-87-2	--	None	NA	NA	NA	NA	
Methylene chloride	75-09-2	0.94	R5 ECO SW	0.94	98.1	4.8	NA	
Methyl-tert-butyl ether	1634-04-4	11070	R3 BTAG SW	NA	11070	12	NA	
o-Xylene	95-47-6	27	R5 ECO SW	27	NA	120	NA	
Styrene	100-42-5	32	R5 ECO SW	32	72	160	NA	
Tetrachloroethene	127-18-4	45	R5 ECO SW	45	111	0.11	NA	
Toluene	108-88-3	253	R5 ECO SW	253	2	230	NA	
trans-1,2-Dichloroethene	156-60-5	970	R5 ECO SW	970	970	11	NA	
trans-1,3-Dichloropropene	10061-02-6	7.9	R5 ECO SW	7.9	NA	NA	NA	
Trichloroethene	79-01-6	47	R5 ECO SW	47	21	2	NA	
Trichlorofluoromethane	75-69-4	130	T-RSL	NA	NA	130	NA	
Vinyl chloride	75-01-4	930	R5 ECO SW	930	930	0.016	NA	
Xylenes (total)	1330-20-7	27	R5 ECO SW	27	13	20	NA	
Semivolatile Organic Compounds								
1,1'-Biphenyl	92-52-4	14	R3 BTAG SW	NA	14	180	NA	
1,2,4,5-Tetrachlorobenzene	95-94-3	3	R5 ECO SW	3	3	1.1	NA	
2,2'-Oxybis(1-chloropropane)	108-60-1	0.32	T-RSL	NA	NA	0.32	NA	
2,3,4,6-Tetrachlorophenol	58-90-2	1.2	R5 ECO SW	1.2	1.2	110	NA	
2,4,5-Trichlorophenol	95-95-4	370	T-RSL	NA	NA	370	NA	
2,4,6-Trichlorophenol	88-06-2	4.9	R5 ECO SW	4.9	4.9	3.7	NA	
2,4-Dichlorophenol	120-83-2	11	R5 ECO SW	11	11	11	NA	
2,4-Dimethylphenol	105-67-9	100	R5 ECO SW	100	NA	73	NA	
2,4-Dinitrophenol	51-28-5	19	R5 ECO SW	19	NA	73	NA	
2,4-Dinitrotoluene	121-14-2	44	R5 ECO SW	44	44	0.22	NA	
2,6-Dinitrotoluene	606-20-2	81	R5 ECO SW	81	81	3.7	NA	
2-Chloronaphthalene	91-58-7	0.396	R5 ECO SW	0.396	NA	290	NA	
2-Chlorophenol	95-57-8	24	R5 ECO SW	24	24	18	NA	
2-Methylphenol (o-Cresol)	95-48-7	67	R5 ECO SW	67	13	180	NA	
2-Nitroaniline	88-74-4	37	T-RSL	NA	NA	37	NA	
2-Nitrophenol	88-75-5	1920	R3 BTAG SW	NA	1920	7.3	NA	
3,3'-Dichlorobenzidine	91-94-1	4.5	R5 ECO SW	4.5	4.5	0.15	NA	
3-Nitroaniline	99-09-2	--	None	NA	NA	NA	NA	
4,6-Dinitro-2-methylphenol	534-52-1	23	R5 ECO SW	23	NA	0.29	NA	
4-Bromophenyl phenyl ether	101-55-3	1.5	R5 ECO SW	1.5	1.5	NA	NA	
4-Chloro-3-methyl phenol	59-50-7	34.8	R5 ECO SW	34.8	NA	370	NA	
4-Chloroanaline	106-47-8	232	R5 ECO SW	232	232	0.34	NA	
4-Chlorophenyl phenyl ether	7005-72-3	--	None	NA	NA	NA	NA	
4-Methylphenol (p-Cresol)	106-44-5	25	R5 ECO SW	25	543	18	NA	
4-Nitroaniline	100-01-6	3.4	T-RSL	NA	NA	3.4	NA	
4-Nitrophenol	100-02-7	60	R5 ECO SW	60	60	NA	NA	
Acetophenone	98-86-2	370	T-RSL	NA	NA	370	NA	
Atrazine	1912-24-9	1.8	R3 BTAG SW	NA	1.8	0.29	NA	

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Water Samples

Analyte	CAS Number	Ecological Soil Screening Level [1] (ug/L)	Source of Ecological Soil Screening Level	EPA R5 Eco SW (ug/L)	EPA R3 BTAG FW SW (ug/L)	EPA T-RSL Adjusted (ug/L)	IDEM SW (ug/L)	NOAA SQUIRT (ug/L)
Benzaldehyde	100-52-7	370	T-RSL	NA	NA	370	NA	
Bis(2-chloroethoxy)methane	111-91-1	11	T-RSL	NA	NA	11	NA	
Bis(2-chloroethyl)ether	111-44-4	19000	R5 ECO SW	19000	NA	0.012	NA	
Bis(2-ethylhexyl)phthalate	117-81-7	0.3	R5 ECO SW	0.3	16	4.8	NA	
Butyl benzyl phthalate	85-68-7	23	R5 ECO SW	23	19	35	NA	
Caprolactam	105-60-2	1800	T-RSL	NA	NA	1800	NA	
Carbazole	86-74-8	--	None	NA	NA	NA	NA	
Dibenzofuran	132-64-9	4	R5 ECO SW	4	3.7	3.7	NA	
Diethyl phthalate	84-66-2	110	R5 ECO SW	110	210	2900	NA	
Dimethyl phthalate	131-11-3	--	None	NA	NA	NA	NA	
Di-n-butyl phthalate	84-74-2	9.7	R5 ECO SW	9.7	19	370	NA	
Di-n-octyl phthalate	117-84-0	30	R5 ECO SW	30	22	NA	NA	
Hexachlorobenzene	118-74-1	0.0003	R5 ECO SW	0.0003	0.0003	0.042	NA	
Hexachlorobutadiene	87-68-3	0.053	R5 ECO SW	0.053	1.3	0.86	NA	
Hexachlorocyclopentadiene	77-47-4	77	R5 ECO SW	77	NA	22	NA	
Hexachloroethane	67-72-1	8	R5 ECO SW	8	12	3.7	NA	
Isophorone	78-59-1	920	R5 ECO SW	920	NA	71	NA	
Nitrobenzene	98-95-3	220	R5 ECO SW	220	NA	0.12	NA	
N-Nitrosodi-n-propylamine	621-64-7	0.0096	T-RSL	NA	NA	0.0096	NA	
N-Nitrosodiphenylamine	86-30-6	210	R3 BTAG SW	NA	210	14	NA	
Pentachlorophenol	87-86-5	4	R5 ECO SW	4	0.5	0.56	19	
Phenol	108-95-2	180	R5 ECO SW	180	4	1100	NA	
Polycyclic Aromatic Hydrocarbons								
2-Methylnaphthalene	91-57-6	330	R5 ECO SW	330	4.7	15	NA	
Acenaphthene	83-32-9	38	R5 ECO SW	38	5.8	220	NA	
Acenaphthylene	208-96-8	4840	R5 ECO SW	4840	NA	220	NA	
Anthracene	120-12-7	0.035	R5 ECO SW	0.035	0.012	1100	NA	
Benzo(a)anthracene	56-55-3	0.025	R5 ECO SW	0.025	0.018	0.029	NA	
Benzo(a)pyrene	50-32-8	0.014	R5 ECO SW	0.014	0.015	0.0029	NA	
Benzo(b)fluoranthene	205-99-2	9.07	R5 ECO SW	9.07	NA	0.029	NA	
Benzo(g,h,i)perylene	191-24-2	7.64	R5 ECO SW	7.64	NA	110	NA	
Benzo(k)fluoranthene	207-08-9	0.29	T-RSL	NA	NA	0.29	NA	
Chrysene	218-01-9	2.9	T-RSL	NA	NA	2.9	NA	
Dibenzo(a,h)anthracene	53-70-3	0.0029	T-RSL	NA	NA	0.0029	NA	
Fluoranthene	206-44-0	1.9	R5 ECO SW	1.9	0.04	150	NA	
Fluorene	86-73-7	19	R5 ECO SW	19	3	150	NA	
Indeno(1,2,3-c,d)pyrene	193-39-5	4.31	R5 ECO SW	4.31	NA	0.029	NA	
Naphthalene	91-20-3	13	R5 ECO SW	13	1.1	0.14	NA	
Phenanthrene	85-01-8	3.6	R5 ECO SW	3.6	0.4	110	NA	
Pyrene	129-00-0	0.3	R5 ECO SW	0.3	0.025	110	NA	
Polychlorinated Biphenyls								
Aroclor-1016	12674-11-2	0.00012	R5 ECO SW	0.00012	0.000074	0.96	0.014	0.000332
Aroclor-1221	11104-28-2	0.00012	R5 ECO SW	0.00012	0.000074	0.0068	0.014	0.000332
Aroclor-1232	11141-16-5	0.00012	R5 ECO SW	0.00012	0.000074	0.0068	0.014	0.000332
Aroclor-1242	53469-21-9	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	0.000332
Aroclor-1248	12672-29-6	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	0.000332
Aroclor-1254	11097-69-1	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	0.000332
Aroclor-1260	11096-82-5	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	0.000332
Total PCBs	1336-36-3	0.00012	R5 ECO SW	0.00012	0.000074	NA	0.014	0.000332
Metals								
Aluminum	7429-90-5	87	R3 BTAG SW	NA	87	3700	750	
Antimony	7440-36-0	80	R5 ECO SW	80	30	1.5	NA	
Arsenic	7440-38-2	148	R5 ECO SW	148	5	0.045	340	
Barium	7440-39-3	220	R5 ECO SW	220	4	730	NA	
Beryllium	7440-41-7	3.6	R5 ECO SW	3.6	0.66	7.3	NA	
Cadmium	7440-43-9	0.15	R5 ECO SW	0.15	0.25	1.8	2	
Calcium	7440-70-2	116000	R3 BTAG SW	NA	116000	NA	NA	
Chromium	7440-47-3	42	R5 ECO SW	42	85	0.043	16	
Cobalt	7440-48-4	24	R5 ECO SW	24	23	1.1	NA	
Copper	7440-50-8	1.58	R5 ECO SW	1.58	9	150	13	
Iron	7439-89-6	300	R3 BTAG SW	NA	300	2600	1000	
Lead	7439-92-1	1.17	R5 ECO SW	1.17	2.5	15	65	
Magnesium	7439-95-4	82000	R3 BTAG SW	NA	82000	NA	NA	
Manganese	7439-96-5	120	R3 BTAG SW	NA	120	88	NA	
Mercury	7439-97-6	0.0013	R5 ECO SW	0.0013	0.026	1.1	1.4	
Nickel	7440-02-0	28.9	R5 ECO SW	28.9	52	73	NA	
Potassium	7440-09-7	53000	R3 BTAG SW	NA	53000	NA	470	
Selenium	7782-49-2	5	R5 ECO SW	5	1	18	5	
Silver	7440-22-4	0.12	R5 ECO SW	0.12	3.2	18	3.2	
Sodium	7440-23-5	680000	R3 BTAG SW	NA	680000	NA	NA	
Thallium	7440-28-0	10	R5 ECO SW	10	0.8	NA	NA	
Vanadium	7440-62-2	12	R5 ECO SW	12	20	18	NA	
Zinc	7440-66-6	65.7	R5 ECO SW	65.7	120	1100	120	
Pesticides								
4,4'-DDD	72-54-8	0.011	R3 BTAG SW	NA	0.011	0.28	NA	
4,4'-DDE	72-55-9	4.51E-09	R5 ECO SW	4.51E-09	NA	0.2	NA	
4,4'-DDT	50-29-3	1.10E-05	R5 ECO SW	1.10E-05	0.0005	0.2	1.1	
Aldrin	309-00-2	0.017	R5 ECO SW	0.017	3	0.004	3	
alpha-BHC	319-84-6	12.4	R5 ECO SW	12.4	NA	0.011	NA	
alpha-Chlordane	5103-71-9	0.00029	R5 ECO SW	0.00029	NA	0.19	2.4	
beta-BHC	319-85-7	0.495	R5 ECO SW	0.495	2.2	0.037	NA	
delta-BHC	319-86-8	667	R5 ECO SW	667	141	0.011	NA	
Dieldrin	60-57-1	7.10E-05	R5 ECO SW	7.10E-05	0.056	0.0042	0.24	

NSA Crane SWMU 11 Ecological Screening Criteria - Surface Water Samples

Analyte	CAS Number	Ecological Soil Screening Level [(1)] (ug/L)	Source of Ecological Soil Screening Level	EPA R5 Eco SW (ug/L)	EPA R3 BTAG FW SW (ug/L)	EPA T-RSL Adjusted (ug/L)	IDEM SW (ug/L)	NOAA SQUIRT (ug/L)
Endosulfan I	959-98-8	0.056	R5 ECO SW	0.056	0.051	22	0.22	
Endosulfan II	33213-65-9	0.056	R5 ECO SW	0.056	0.051	22	0.22	
Endosulfan sulfate	1031-07-8	2.22	R5 ECO SW	2.22	NA	22	NA	
Endrin	72-20-8	0.036	R5 ECO SW	0.036	0.036	1.1	0.086	
Endrin aldehyde	7421-93-4	0.15	R5 ECO SW	0.15	NA	1.1	NA	
Endrin ketone	53494-70-5	11	T-RSL	NA	NA	1.1	NA	
gamma-BHC (Lindane)	58-89-9	0.026	R5 ECO SW	0.026	0.01	0.061	0.95	
gamma-chlordane	5103-74-2	0.00029	R5 ECO SW	0.00029	NA	0.19	2.4	
Heptachlor	76-44-8	0.0038	R5 ECO SW	0.0038	0.0019	0.015	0.52	
Heptachlor epoxide	1024-57-3	0.0038	R5 ECO SW	0.0038	0.0019	0.0074	0.52	
Methoxychlor	72-43-5	0.019	R5 ECO SW	0.019	0.019	18	0.03	
Toxaphene	8001-35-2	0.00014	R5 ECO SW	0.00014	0.0002	0.061	0.73	
Dioxins/Furans								
1,2,3,4,6,7,8,9-OCDD	3268-87-9	0.000000278	R5 ECO SW	2.78E-07	NA	0.0017	NA	
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.00129	R5 ECO SW	0.00129	NA	0.0017	NA	
1,2,3,4,6,7,8-HPCDD	35822-46-9	0.000000278	R5 ECO SW	2.78E-07	NA	0.000052	NA	
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.00129	R5 ECO SW	0.00129	NA	0.000052	NA	
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.00129	R5 ECO SW	0.00129	NA	0.000052	NA	
1,2,3,4,7,8-HXCDD	39227-28-6	0.000000278	R5 ECO SW	2.78E-07	NA	0.0000052	NA	
1,2,3,4,7,8-HXCDF	70648-26-9	0.00129	R5 ECO SW	0.00129	NA	0.0000052	NA	
1,2,3,6,7,8-HXCDD	57653-85-7	0.000000278	R5 ECO SW	2.78E-07	NA	0.0000052	NA	
1,2,3,6,7,8-HXCDF	57117-44-9	0.00129	R5 ECO SW	0.00129	NA	0.0000052	NA	
1,2,3,7,8,9-HXCDD	19408-74-3	0.000000278	R5 ECO SW	2.78E-07	NA	0.0000052	NA	
1,2,3,7,8,9-HXCDF	72918-21-9	0.00129	R5 ECO SW	0.00129	NA	0.0000052	NA	
1,2,3,7,8-PECDD	40321-76-4	0.000000278	R5 ECO SW	2.78E-07	NA	0.00000052	NA	
1,2,3,7,8-PECDF	57117-41-6	0.00129	R5 ECO SW	0.00129	NA	0.000017	NA	
2,3,4,6,7,8-HXCDF	60851-34-5	0.00129	R5 ECO SW	0.00129	NA	0.0000052	NA	
2,3,4,7,8-PECDF	57117-31-4	0.00129	R5 ECO SW	0.00129	NA	0.0000017	NA	
2,3,7,8-TCDD	1746-01-6	3.00E-09	R5 ECO SW	3.00E-09	3.1E-09	0.00000052	NA	
2,3,7,8-TCDF	51207-31-9	0.0000052	T-RSL	NA	NA	0.0000052	NA	
Total HpCDD	37871-00-4	NA	None	NA	NA	NA	NA	
Total HpCDF	38998-75-3	NA	None	NA	NA	NA	NA	
Total HxCDD	34465-46-8	NA	None	NA	NA	NA	NA	
Total HxCDF	55684-94-1	NA	None	NA	NA	NA	NA	
Total PeCDD	36088-22-9	NA	None	NA	NA	NA	NA	
Total PeCDF	30402-15-4	NA	None	NA	NA	NA	NA	
Total TCDD	41903-57-5	NA	None	NA	NA	NA	NA	
Total TCDF	55722-27-5	NA	None	NA	NA	NA	NA	

1- The following hierarchy was used for selecting the Ecological Screening level in order of preference:
USEPA Region 5 Ecological Surface Water Screening Levels (R5 ECO SW) (USEPA, 2005).
USEPA Region 3 BTAG Ecological Surface Water Screening Levels (R3 BTAG SW) (USEPA, 2005).
USEPA Regions 3, 6, and 9 Tapwater Regional Screening Levels (T-RSL), adjusted to 1/10 of value for non-carcinogens (USEPA, May 2010).
Lowest of National Oceanographic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQUIRT) surface water benchmarks (Buchman, 2008).

Shaded cells are values that were selected as the overall ecological surface water screening level.