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SAMPLING AND ANALYSIS PLAN FOR SOLID WASTE MANAGEMENT UNIT 28
MAINTENANCE SHOP BUILDING 1820 AREA NAS CRANE IN
5/1/2011
TETRA TECH NUS

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62470-08-D-1001



Sampling and Analysis Plan

for

SWMU 28 – MAINTENANCE SHOP BUILDING 1820 AREA

**Naval Support Activity Crane
Crane, Indiana**

Contract Task Order F273

May 2011

FINAL



201 Decatur Avenue
Building IA, Code EV
Great Lakes, Illinois 60088

SAP WORKSHEET NO. 1 -- TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

**FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
May 2011**

**RESOURCE CONSERVATION AND RECOVERY ACT
FACILITY INVESTIGATION**

**SWMU 28 – MAINTENANCE SHOP BUILDING 1820 AREA
NAVAL SUPPORT ACTIVITY CRANE
CRANE, INDIANA**

Prepared for:

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

Prepared by:

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Prepared under:

Contract No. N62470-08-D-1001
Contract Task Order F273

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NSA Crane Environmental Restoration Site Manager

Doug Griffin/RPM/Date
Indiana Department of Environmental Management

Navy Chemist /Date

Project-Specific SAP
Site Name/Project Name: NSA Crane
Site Location: Crane, Indiana

Title: SAP for SWMU 28 RFI
Date: April 2011 Revision Number: BA
Revision Date: March/February 2011

SAP WORKSHEET NO. 1 -- TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

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DRAFT FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
April/March 2011

RESOURCE CONSERVATION AND RECOVERY ACT
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Indiana Department of Environmental Management

Jonathan Tucker

Digitally signed by
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Date: 2011.04.27 15:15:40 -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) 28 – Maintenance Shop Building 1820 Area at Naval Support Activity (NSA) Crane, Indiana under Contract Task Order (CTO) F273, Contract N62470-08-D-1001, Comprehensive Long-Term Environmental Action Navy (CLEAN). This SAP was prepared under CTO F273; the field sampling and RFI report will be prepared under CTO F27G.

SWMU 28 – Maintenance Shop Building 1820 Area is located in the central area of NSA Crane, in the Garage Area and covers approximately seven acres. The site contains the location of two former maintenance buildings – Building 1820 and Building 1818. Building 1820 was the Heavy Equipment Repair Shop, and Building 1818 was the Automotive Repair Shop. In 2002, the operations and functions of both buildings were relocated to another building that is also within the Garage Area, and is currently in active use. Building 1818 was completely demolished in 2002. The above ground structure and components of Building 1820 were demolished in 2005, although the building floor slab and foundation remain on-site and are currently used for storage.

Historical reports indicate that various underground storage tanks used for waste oil were present at SWMU 28. Environmental concern was generated by the observation that various underground storage tanks (USTs) were present at SWMU 28, as well as open-top solvent tanks, various carwash cleaners, and steam cleaners (A. T. Kearney, 1987). The USTs, which were used for waste oil and waste solvents, were reportedly in poor condition upon their removal. A historical report also indicates that SWMU 28 may have been used as a disposal area for automotive batteries for an unknown period of time (Naval Energy and Environmental Support Activity [NEESA], 1983). As a result, degreasing solvents, fuels, oils, and greases associated with vehicle maintenance and use, hydraulic fluids, and various metals may have leaked onto the ground and leached into the subsurface and/or groundwater at SWMU 28.

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 delineate the nature and extent of those contaminants. The RFI will include the collection and analysis of surface and subsurface soil, sediment, groundwater, and surface water samples. These samples will be collected to provide data for use in human health and ecological risk screening and potentially for risk assessment, if necessary. This information will also be used in the remedial decision making process.

Surface and subsurface soil, sediment, surface water, and groundwater samples will be analyzed for volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), total petroleum hydrocarbons (TPH), and metals. 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 28 – Maintenance Shop Building 1820 Area. 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 and sampling are scheduled to begin in April 2011. A complete schedule is detailed in SAP Worksheet No. 16.

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

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ACRONYMS AND ABBREVIATIONS

AR	Administrative Record
BFB	Bromofluorobenzene
bgs	below ground surface
°C	degrees Celsius
CA	Corrective Action
CAS	Chemical Abstracts Service
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
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	Chemical of Potential Concern
COPEC	Constituent of Potential Ecological Concern
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
DFTPP	decafluorotriphenylphosphine
DL	Detection Limits
DO	dissolved oxygen
DON	Department of the Navy
DoD	Department of Defense
DPT	direct-push technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DRO	Diesel Range Organics
DVM	Data Validation Manager
Eco-SSL	Ecological Soil Screening Level
Empirical	Empirical Laboratories, LLC
EPC	Exposure Point Concentrations
ERA	Ecological Risk Assessment
ERO	Extended Range Organics

ERSM	Environmental Restoration Site Manager
ESL	Ecological Screening Level
EU	Exposure Units
FD	Field Duplicate
FID	flame ionization detector
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
GRO	Gasoline Range Organics
HASP	Health and Safety Plan
HCl	hydrochloric acid
HDOP	horizontal dilution of precision
HHRA	Human Health Risk Assessment
HSM	Health and Safety Manager
IA	Investigative Area
ICAL	Initial Calibration
ICV	Initial Calibration Verification
IDEM	Indiana Department of Environmental Management
IDW	investigation-derived waste
ILCR	Incremental Lifetime Cancer Risk
IS	Internal Standard
IUPPS	Indiana Underground Plant Protection Services
L	liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limits of Detection
LOQ	Limits 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 Criteria
msl	Mean Sea Level
NA	Not Applicable
NACIP	Navy Assessment and Control of Installation Polutants
NAVFAC	Naval Facilities Engineering Command

NC	no criteria
NFA	No Further Action
NPDES	National Pollutant Discharge Elimination System
NSA	Naval Support Activity
NTU	Nephelometric Turbidity Unit
ORP	Oxidation-Reduction Potential
OWS	Oil-Water Separator
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PDF	Portable Document Format
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PPE	personal protective equipment
ppm	parts per million
PQLG	Project Quantitation Limit Goal
PQO	project quality objective
PSL	Project Screening Level
PWD	Public Works Department
QA	quality assurance
QAO	Quality Assurance Officer
QAM	Quality Assurance Manager
QC	quality control
QSM	Quality Systems Manual
%R	percent recovery
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFI	Resource Conservation and Recovery Act Facility Investigation
RI	Remedial 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
SAP	Sampling and Analysis Plan
SDG	sample delivery group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language

SQuiRT	Screening Quick Reference Table
SSL	Soil Screening Level
SSO	Site Safety Officer
SVOC	semivolatile organic compound
SWMU	Solid Waste Management Unit
TBD	To Be Determined
Test America	Test America/Seattle
TOC	total organic carbon
TPH	total petroleum hydrocarbon
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
UFP-SAP	Uniform Federal Policy for Sampling Analysis Plan
µg/kg	microgram per kilogram
µg/L	microgram per liter
USEPA	United States Environmental Protection Agency
UST	underground storage tank
VCT	Vitrified Clay Tile
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) 28 – Maintenance Shop Building 1820 Area
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) F273

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 Plans (UFP-QAPP)* (USEPA, 2005) and *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5, QAMS (USEPA, 2002).

2. Identify regulatory program: Resource Conservation and Recovery Act of 1976, as amended by the Hazardous and Solid Waste Amendments of 1984.

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
<u>Data Quality Objectives (DQO) Meeting</u>	<u>November 4, 2010</u>

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

Title	Date
<u>"Initial Assessment Study of Naval Weapons Support System" (IAS), (Naval Energy and Environmental Support Activity [NEESA] 13-003)</u>	<u>May 1983</u>
<u>"Preliminary Review/Visual Site Inspection Report" (VSI), (A.T. Kearney)</u>	<u>March 1987</u>
<u>"CPP Special Project C-302, Construct Heavy Equipment Maintenance Facility" (Naval Sea Systems Command [NAVSEA])</u>	<u>September 2, 2002</u>

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

Indiana Department of Environmental Management (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@tetrattech.com
Garth Glenn (distribution letter only)	Deputy Program Manager/ Manages Program Activities	Tetra Tech	757-461-3926	garth.glenn@tetrattech.com
Tony Klimek, P.E.	Project Manager (PM)/ Manages Project Activities	Tetra Tech	513-557-5057	tony.klimek@tetrattech.com
Ralph Basinski	Crane Activity Coordinator/ Coordinates Tetra Tech Activities at NSA Crane	Tetra Tech	412-921-8308	ralph.basinski@tetrattech.com
Tom Johnston, PhD (electronic copy only)	Quality Assurance Manager (QAM))/ Manages Corporate	Tetra Tech	412-921-8615	tom.johnston@tetrattech.com

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address
	Quality Assurance (QA) Program and Implementation			
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@tetrattech.com
Joe Samchuck (electronic copy only)	Data Validation Manager (DVM) / Manages Data Validation	Tetra Tech	412-921-8510	joseph.samchuck@tetrattech.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@tetrattech.com
Mark Traxler (electronic copy only)	Project Chemist/ Provides Coordination with Laboratory	Tetra Tech	610-382-1171	mark.traxler@tetrattech.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
Curtis Armstrong (electronic copy only)	Laboratory PM – Representative for Laboratory and Analytical Issues	Test America/Seattle (Test America)	253-922-2310	curtis.armstrong@testamericainc.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	

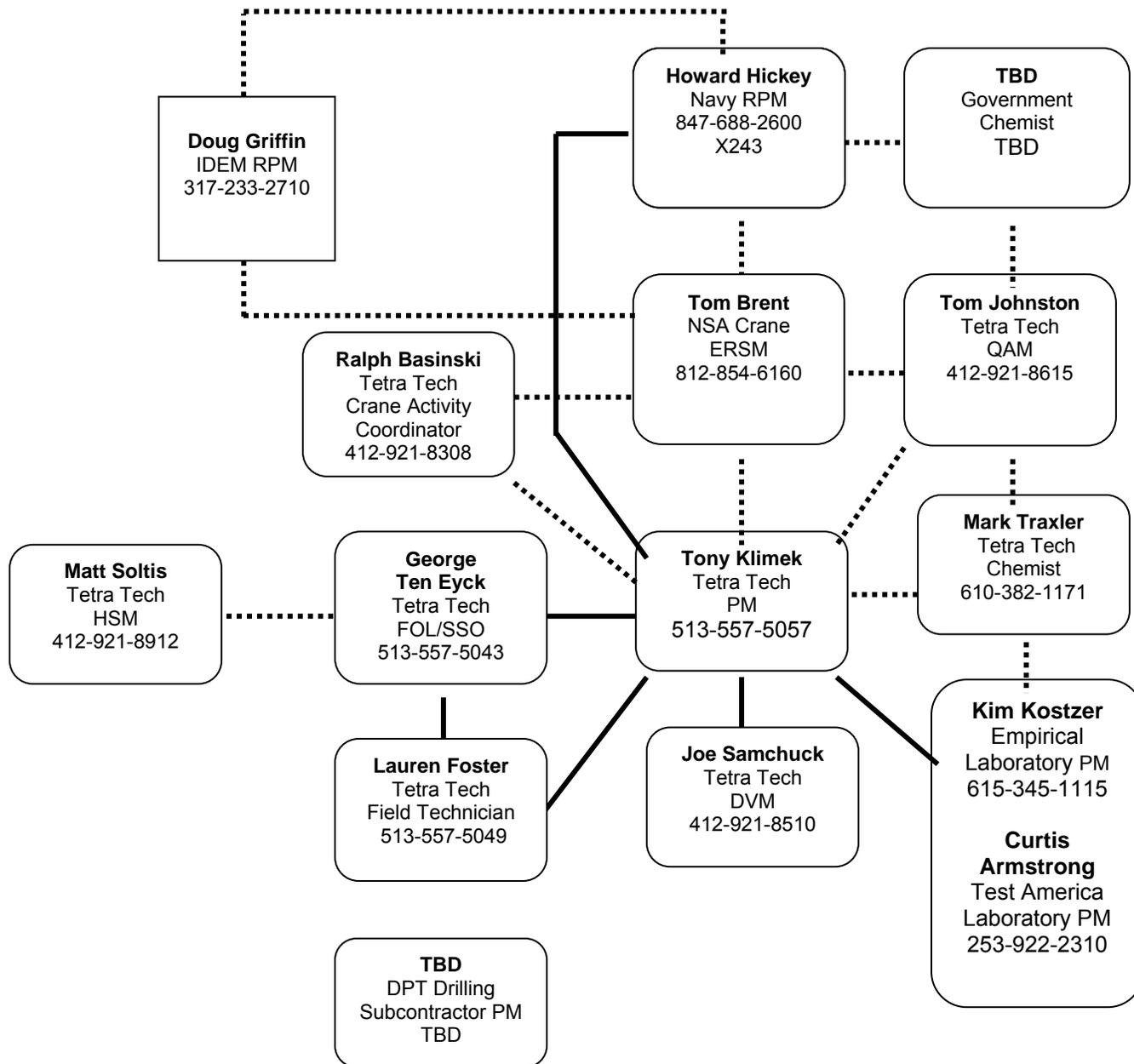
Name ⁽¹⁾	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Joe Samchuck	Tetra Tech/ DVM/ Manages Data Validation	412-921-8510		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, and 34-37	
Mark Traxler	Tetra Tech/ Project Chemist/ Provides Coordination with 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	
Curtis Armstrong	Test America/ Laboratory PM/ Representative for Laboratory and Analytical Issues	253-922-2310		Worksheet Nos. 6, 12, 14, 15, 19, 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 five days and send the Navy RPM a concurrence letter within seven 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 one 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 one 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 one 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 two days of identifying the need for change on a FTMR form and obtain required approvals within five 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 one 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 Test America Laboratory PM Tetra Tech Project Chemist Tetra Tech DVM Tetra Tech PM NSA Crane ERSM	Kim Kostzer Curtis Armstrong Mark Traxler Joe Samchuck Tony Klimek Tom Brent	615-345-1115 253-922-2310 610-382-1171 412-921-8510 513-557-5057 812-854-6160	<p>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.</p> <p>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.</p> <p>The Tetra Tech DVM or Project Chemist notifies the Tetra Tech PM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech PM verbally advises the NSA Crane ERSM within 24 hours of notification from the Tetra Tech Project Chemist or DVM. The NSA Crane ERSM 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 Navy Chemist.</p>

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.
Peter Ramanauskas	RPM/ Provides regulator Input	USEPA Region 5	Oversees project implementation, including scoping, and data review and evaluation.
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, data review, and evaluation.
Ralph Basinski	Crane Activity Coordinator/ Coordinates Tetra Tech activities at NSA Crane	Tetra Tech	Oversees project implementation including scoping, 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. Provides equipment to collect groundwater samples.
Kim Kostzer Curtis Armstrong	Laboratory PM Laboratory PM	Empirical Test America	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: <u>NSA Crane SWMU 28 RCRA Facility Investigation (RFI)</u> Projected Date(s) of Sampling: April 2011 Project Manager: <u>Tony Klimek</u>			Site Name: <u>SWMU 28 – Maintenance Shop Building 1820 Area</u> Site Location: <u>Crane, Indiana</u>		
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@tetrattech.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@tetrattech.com	Management /Oversight
Peggy Churchill	DQO Facilitator	Tetra Tech	321-636-6470	peggy.churchill@tetrattech.com	DQO Facilitator

Comments/Decisions: Discussed SWMU 28 historical use and available data. Discussed the steps for the RFI in accordance with the SAP format.

Consensus Decisions: See meeting minutes in Appendix C.

Tetra Tech presented background information about SWMU 28 and there was a general discussion about the proposed sampling. Four of the consensus decisions are listed below:

1. Sediment samples will be collected on the SE and NW sides of the site and groundwater samples SE of the site to determine if contamination (if any) from the SWMU is leaving the site.
2. Groundwater samples will be collected from temporary wells installed via DPT borings. Five downgradient and one upgradient sample will be collected.
3. Because hydraulic fluids and other oils may contain PCBs, analysis of areas surrounding and downgradient of the USTs and the gravel pads should include PCB analysis.
4. The locations of samples at pipe discharge points may be determined by defining the coordinates of the discharge points on the drawings and then using a global positioning system (GPS) to find those locations in the field.

SAP WORKSHEET NO. 10 -- CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2)

This worksheet presents general background information about SWMU 28 – Maintenance Shop Building 1820 Area 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 28 – Maintenance Shop Building 1820 Area (site) is located in the central area of NSA Crane (see Figure 10-1). SWMU 28 is located south of Highway 99 and west of Highway 78, approximately one half-mile southwest of the intersection between Highway 5 and Highway 45. The site is located in the Garage Area of NSA Crane.

SWMU 28 is an irregular shaped area that is approximately 1,200 feet long in a southwest-northeast direction and 400 feet wide in a southeast-northwest direction. It covers approximately seven acres, as shown on Figure 10-2. The site contains the location of two former maintenance buildings – Building 1820 and Building 1818. Building 1820 was the Heavy Equipment Repair Shop, and Building 1818 was the Automotive Repair Shop. Building 1820 was located in the south side of SWMU 28, and was oriented in the east-west direction. Building 1818 was located in the northeast area of SWMU 28, and had a general north-south orientation. Most of SWMU 28 is currently covered by buildings, asphalt pavement, and gravel.

As shown on Figure 10-3, SWMU 28 is located at the end of a ridge that is oriented in a southwest-northeast direction. Vehicle access to this area is via two roads that extend from the site in a northeast direction. The site slopes topographically from 740 feet above mean sea level (msl) on the northeast to 720 feet above msl on the south. From the edge of the site on the northwest, southwest, and southeast sides, the topography slopes down approximately 50 feet into wooded valleys below the site.

Runoff from SWMU 28 drains from the site into the valleys on three sides and into Boggs Creek approximately 2 miles south-southwest of the site. Boggs Creek flows south and off of the NSA Crane facility approximately 9 miles south of the site.

Physical structures at SWMU 28 (as of 2010) are shown on Figures 10-2 and 10-3. A garage (Building 2713), a pole barn (Building 3387), and a boiler house (Building 1819) are all in active use and are within the SWMU 28 boundary. This investigation addresses historical releases, and not activities that are ongoing at the site; therefore, those areas are not included in the SWMU 28 RFI described in this SAP.

Much of SWMU 28 is maintained as a parking lot/storage area for Garage Area inventory. An outdoor Truck Wash Rack is located in the western area of the site. Gravel stockpiles are located near the

southwest corner on the site. The Building 1818 structure including the floor slab has been removed. The above ground structure for Building 1820 has been removed, but the floor slab and foundation remain in place. Site features associated with the two buildings such as underground storage tanks (USTs), an OWS, an oil drip pan, and solvent tanks were removed along with the former buildings.

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 southwest to the drainage channel toward Boggs Creek. Depth to groundwater at SWMU 28 is unknown, but is estimated to be approximately 20 feet below ground surface (bgs).

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 south, west, and east 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 more than one mile to the west of the site, beyond the western boundary of the facility.

10.2 SMWU 28 HISTORY

The Garage Area is an active maintenance and repair facility for NSA Crane's heavy equipment and automotive fleet. The area has been in operation since the 1940s and is expected to continue similar operations for the foreseeable future. The Garage Area inventory currently includes approximately 250 pieces of construction equipment, including bulldozers, air compressors, and truck-mounted cranes, as well as the activity's automotive equipment.

Former Buildings 1820 and 1818 were constructed during World War II, and were designed to be approximately 270 feet long and 60 feet wide. Originally referred to as Garage 1 (Building 1818) and Garage 2 (Building 1820), the structures were built with 22 bays each. Both were subject to numerous changes and renovations from the late 1940s to the late 1990s, as documented in historical drawings and sketches, which are available for review. Figure 10-4 is an aerial map of the site that shows former Buildings 1818 and 1820 as they existed in 1952. As shown on Figure 10-4, the level area of SWMU 28 was considerably smaller in 1952 than it is currently. Some of the fill used to expand the level area of the site includes former pavement material; therefore, polycyclic aromatic hydrocarbon (PAH) contamination, such as benzo(a)pyrene, may be found downgradient of the site. In 2002, the operations and functions of both buildings were relocated when an addition was constructed on Building 2713, which is in active use and also lies within the boundaries of SWMU 28.

Building 1818 was completely demolished prior to the Building 2713 addition; the former location of Building 1818 now includes part of the Building 2713 addition and a parking lot/storage area. The above ground structure and components of Building 1820 were demolished in 2005, although the building floor slab and foundation remain on-site and are currently used for storage.

10.2.1 Building 1818 History

Building 1818, the Automotive Repair Shop, housed six hydraulic lifts with air trenches and hydraulic lift pits. One lift and air trench was located in the northeastern portion of the building. Four more sets of lifts and air trenches were approximately 24 feet to the south. The final lift and air trench was another 48 feet to the south, also in the eastern portion of the building. The front and back lifts were eight to ten feet below slab. Historical drawings showing the general layout of Building 1818, discharge pipes, and overall site are presented in Appendix F.

A trench, a grease pit, a wheel alignment pit, and a steam pit were all located in the southern half of Building 1818. Building 1818 also contained open-top solvent cleaning tanks and various floor drains, sinks, and pans throughout; drains discharged both to the sewer system and also directly to ditches in the surrounding area. There are three identified historical pipe discharge points, located in the valley east of the former building location, that were associated with Building 1818.

A gravel pad existed adjacent to Building 1818 on the east side of the building, between the location of the former building and the valley below. The gravel pad was identified by current site workers as an area where spills or releases of fuel and waste oil were likely to have occurred. The Building 1818 gravel pad area is now covered with asphalt pavement. A similar gravel pad adjacent to Building 1820 was located on the south side of Building 1820, between the former building location and the valley below. The Building 1820 gravel pad was also identified as an area with high potential for past spills or releases.

A historical report indicates that each year approximately 200 to 300 automotive batteries were disposed behind Building 1818 and Building 1820 (NEESA, 1983). A copy of the report is included in Appendix D. Battery acid was reportedly dumped into the valley behind the buildings. During an August 2010 site visit, interviews with site workers confirmed that automotive batteries were handled and disposed from a battery changing station inside Building 1818. A radiator room was located in the approximate center of the building, on its east side. Based on historical knowledge, it is likely that battery acid was handled in the radiator room, which contained ventilation hoods and open vats. Workers could not confirm reports that battery acid or batteries were disposed in the valley behind the buildings. During a site walk of the area in October 2010, no visible signs of battery dumping or disposal were observed.

Four known former USTs were associated with Building 1818. A 1980 drawing shows two 500-gallon waste oil USTs located approximately 100 feet east-southeast of Building 1818; a 1991 drawing shows plans for two waste oil USTs (one 720-gallon and one 750-gallon) in similar locations to be removed and

backfilled. The 1980 drawing and the 1991 drawing are assumed to represent the same tanks. The 1991 drawing also shows the installation of one new 1,000-gallon double walled waste oil UST on the east side of Building 1818. The 1,000-gallon UST was located closer to Building 1818 than the other USTs located on the east side of the building. Site workers confirmed that the 1,000-gallon UST for waste oil was removed when the building was demolished in 2002. A 1954 drawing shows the installation of a 500-gallon UST, on the west side of the building, used to collect oil from the grease pit inside the building. The removal date of this tank is uncertain. Historical drawings showing the locations of the USTs are presented in Appendix F.

The easternmost 500-gallon UST shown in the 1980 drawing was located on the hillside near Building 1818. Prior to its removal, erosion of the hillside near the unit had exposed part of the tank (A. T. Kearney, 1987). Historical photos showing the vent and fill line, and a release of waste oil, from this UST are included in Appendix F. Other photographs in Appendix F include an oil pan washout/disposal rack near Building 1818. Waste oil from the disposal rack is believed to have drained to a UST.

The 1987 report (A. T. Kearney, 1987) also reported that there were metal roll-off boxes containing scrap cardboard, wood, and general garbage present on the paved areas behind both Building 1818 and Building 1820. These areas also contained steel transfer vaults (hazardous waste transfer containers) that contained up to four drums of hazardous waste.

10.2.2 Building 1820 History

Building 1820, the Heavy Equipment Repair Shop, contained a vehicle wash area with two wash sumps on the west side. Various car wash cleaners and steam cleaners were used in the vehicle wash area. The vehicle wash area drains have historically discharged to the southeast valley. Two service trenches were located near the central-western portion of the building, approximately 24 feet apart. Open-top solvent tanks were located throughout the building, at least two of which contained the solvent agitene (NEESA, 1983). Agitene is a non-chlorinated petroleum-based solvent that contains more than 97% aliphatic petroleum distillates. Aliphatic petroleum compounds may contain benzene and other aromatics. Various other solvents and detergents, including chlorinated solvents, may have been used in Building 1820. An oil pan wash out/disposal rack was located on the south exterior of Building 1820 between the two service trenches. Building 1820 also contained various floor drains, sinks, and pans throughout; drains discharged both to the sewer system and also directly to ditches in the surrounding area. There were multiple identified locations where drains discharged from Building 1820 onto the side of the valley south of the former building location. Historical drawings showing the general layout of Building 1820, discharge pipes, and overall site are presented in Appendix F.

There were two former USTs used for waste oil associated with Building 1820. A historical report also indicates that waste solvent was also disposed in the waste oil tanks (NEESA, 1983). Both a 1945 and a 1980 drawing show a 500-gallon waste oil UST located between the grease pits of Building 1820. A 1991

drawing shows removal of this UST (identified as a 750-gallon tank) and replacement with a new 1,000-gallon double walled UST just south of the same location and outside Building 1820. This new location was in the area of the oil pan wash out area. Historical drawings showing the locations of the USTs are presented in Appendix F. Site workers confirmed that the 1,000-gallon UST was removed when the building was removed.

According to a 1974 design drawing, an OWS was located approximately 30 feet southwest of Building 1820. Prior to 1974, oily wastewater was discharged through floor drains from the building and onto the side of the slope below Building 1820 and eventually into Boggs Creek. After 1974, wastewater from the OWS discharged to the sanitary sewer. Waste oil and wastewater from both Building 1818 and 1820 were reported to drain to the OWS.

There was and currently is an Outside Truck Wash Rack located north of Building 1820 that drains to the northwest valley.

10.3 PREVIOUS ENVIRONMENTAL INVESTIGATIONS AND ACTIONS

Previous environmental inspections and investigations conducted at SWMU 28 include an IAS (NEESA, 1983) and a VSI (A.T. Kearny, 1987). The results of these inspections and investigations are summarized below.

In May 1983, a team from NEESA conducted an IAS as part of the Navy Assessment and Control of Installation Pollutants (NACIP) program. SWMU 28 was not one of the potentially contaminated sites that were identified in the IAS. However, the description of Garage Area operations included the OWS, the truck wash areas, a possible battery disposal area, and the various solvents, cleaners, and waste oil tanks that were in use at Buildings 1818 and 1820.

In March 1987, A.T. Kearny issued a VSI which identified ten "SWMUs" that were associated with activities at Building 1818 and Building 1820 (currently known as SWMU 28) (A.T. Kearny, 1987). The list of "SWMUs" from the VSI does not correspond to the actual list of NSA Crane SWMUs that are managed under RCRA, so units identified in the VSI will be referred to as Areas of Concern (AOCs) in this worksheet to avoid confusion:

"SWMU" in Visual Site Inspection Report	AOC in this SAP	Description
SWMU 29	AOC 29	Auto Maintenance Shop
SWMU 30	AOC 30	Heavy Equipment Maintenance Shop
SWMU 31	AOC 31	Truck Wash Area at Building 1820
SWMU 32	AOC 32	OWS at Building 1820

"SWMU" in Visual Site Inspection Report	AOC in this SAP	Description
SWMU 33	AOC 33	Outside Truck Wash Rack
SWMU 34	AOC 34	Roll-Off Boxes Outside Building 1820
SWMU 35	AOC 35	Yellow painted steel transfer vaults
SWMU 36	AOC 36	Oil Pan Wash Out/Disposal Rack Adjacent to Building 1820
SWMU 37	AOC 37	Underground Waste Oil Storage Tank at Building 1818
SWMU 38	AOC 38	Underground Waste Oil Storage Tank at Building 1820

AOC 29 was identified as the Auto Maintenance Shop and AOC 30 was identified as the Heavy Equipment Maintenance Shop. The report states that these buildings contained noticeable oil spills and solvent contaminated rags, solvent tanks with no secondary containment controls, and that waste oil and wastewater drained into the OWS. The report concluded that the potential for release into soil/groundwater and surface water or generation of subsurface gas was low due to the indoor setting and nature of wastes handled at the buildings. No Further Action (NFA) was suggested for both these units.

AOC 31 was identified as the Truck Wash Area at Building 1820, and was described as two indoor concrete wash racks with sloped concrete floors that drained rinse waters and any rinse oil into two drip tracks which drained to the OWS. The report concluded that the potential for release into soil/groundwater, surface water, and air, or generation of subsurface gas was low due to the unit's design and the dilute nature of wastes handled and suggested NFA for this unit.

AOC 32 was identified as the OWS at Building 1820, and was described as a below-grade sump that received rinse waters containing oil/degreasers from Building 1818 and Building 1820. A thin conveyor belt skimmed oil and deposited it in a separate adjacent concrete sump and the remaining water was discharged to the Sanitary Sewer System. The report concluded that the potential for release into soil/groundwater, surface water, and air, or generation of subsurface gas was low due to the unit's design and the dilute nature of wastes handled and suggested NFA for this unit.

AOC 33 was identified as the Outside Truck Wash Rack, and was described as a truck wash hose and a raised wooden slat platform where washing trucks was performed. The VSI reported that this equipment was adjacent to Building 1818; however, based on the description of the wash rack, it appears to have been located north of the former Building 1820 and west of Building 1818. According to the VSI report, rinse waters drained through the slats and down the hill into an intermittent stream. Although the report indicated that only water was used at the wash rack, no release controls existed for this unit, so continuous release of rinse waters to the ground beneath the unit historically occurred during washing.

The report concluded that the potential for release to soil/groundwater and surface water is high due to the unit's construction. Suggested further action included the containment and treatment of rinse waters generated at the unit, as well as the collection of soil samples from underneath the platform and in the drainage ditch that flows down the hillside. A truck wash rack believed to be AOC 33 is still present on-site and is currently in use, although it now consists of a metal slat platform. Rinse waters are now collected in an overflow/containment pond before discharging into the valley to the west of the wash rack via a drainage pipe. The rinse waters have been monitored by the NSA Crane Environmental Protection Department on a yearly basis since the early 2000s, and the Outside Truck Wash Rack is inspected quarterly. Both historical and current operations of AOC 33 have been reportedly limited to the use of water. As of October 2010, no environmental concern has been associated with the monitored rinse waters, and no National Pollutant Discharge Elimination System (NPDES) permit is required for AOC 33. The ongoing operation is being actively monitored by NSA Crane Environmental Protection Department. There is the potential for contamination from past practices at this facility.

AOC 34 was identified as Roll-Off Boxes Outside Building 1820, and AOC 35 was in the same area and contained hazardous waste transfer containers. AOC 34 was described as typical metal roll-off boxes containing scrap cardboard, wood, and general garbage to be hauled to the sanitary landfill that were located on paved areas behind each building. AOC 35 was described as yellow painted steel transfer vaults in which four drums of waste were placed and transferred to a storage area. The report concluded that the potential for release into soil/groundwater, surface water, and air, or generation of subsurface gas was low due to the containerization and nature of wastes and suggested NFA for these units.

AOC 36 was identified as the Oil Pan Wash Out/Disposal Rack Adjacent to Building 1820, and was described as a metal drip pan erected on wooden posts that gravity fed a pipe which drained into a UST. The ground beneath the drip pan was reportedly covered with oil stains with remnants of oil-encrusted grass present. The report concluded that the potential for release to soil/groundwater was high due to spillage of waste onto the ground and moderate for surface water due to runoff from affected soils. Suggested further action included the removal of obviously contaminated soil, soil sampling to confirm that all impacted soil was removed, and additional steps such as installation of a containment pad to ensure no future oil spills. AOC 36 is no longer present on-site. Site workers confirmed in interviews that the Oil Pan Disposal Rack was removed prior to the demolition of Building 1820. Presently, the area south of former Building 1820 is an asphalt and/or gravel surface. Visible oil stains are no longer present in the area.

AOC 37 was identified as the Underground Waste Oil Storage Tank at Building 1818 and AOC 38 was identified as the Underground Waste Oil Storage Tank at Building 1820. Both units were described as 500-gallon single shell steel storage tanks with no leak detection system. The tanks were used for storage of waste oil prior to transfer. The report concluded that the potential for release to soil/groundwater was high dependent on the age of the tanks and generation of subsurface gas was moderate dependent on the integrity of the tanks. Suggested further action for both AOC 37 and AOC 38

was an inspection to determine the integrity of each unit. Site workers confirmed in interviews that prior to the building demolitions, two USTs were removed from Building 1818, and one UST was removed from Building 1820. However, no documentation regarding their removal has been identified or obtained.

10.4 CONCEPTUAL SITE MODEL

Spills and releases from general site activities at SWMU 28 are the likely sources of potential contamination. The historical information from the VSI and IAS indicate that releases likely occurred directly onto the surface soil and potentially the subsurface soil, as well as discharging onto the sides of the valleys below the site. The historical and current use at SWMU 28 is automotive and heavy equipment repair. Various waste oils, fuels, solvents, detergents, and metals were used and stored on-site. No analytical data currently exists for SWMU 28. Based on site operational data, interviews, and historical information from similar automotive repair shops, volatile organic compounds (VOCs); PAHs; polychlorinated biphenyls (PCBs); total petroleum hydrocarbons (TPH), including Gasoline Range Organics (GRO), Diesel Range Organics (DRO), and Extended Range Organics (ERO); and metals may have been released from processes at the site. The list of target analytes for SWMU 28 is presented in Worksheet No. 15. A CSM schematic is presented on Figure 10-5.

Based on known site history and operations, areas of interest, identified as investigative areas (IA) include the following:

- IA-1 - Building 1818 Gravel Pad
- IA-2 - Building 1820 Gravel Pad
- IA-3 - Building 1818 USTs (AOC 37 from the VSI)
- IA-4 - Building 1820 USTs and Oil Pan Disposal Rack (AOC 38 and AOC 36 from the VSI)
- IA-5 - Pipe Discharge Points
- IA-6 - Battery Disposal Area
- IA-7 - Outside Truck Wash Rack (AOC 33 from the VSI)
- IA-8 - Stream Southeast of Site
- IA-9 - Stream Channel Northwest of Site

10.4.1 Potential Sources and Target Analytes

The target analyte list for this investigation was selected from the list of Priority Pollutant chemicals and is based on the contaminants that were likely or potentially associated with site use. Based on historical information and personnel interviews, spills and releases from general site activities are likely sources of contamination at SWMU 28. The site was primarily used for vehicle maintenance; the USTs stored waste oil and fluids related to those activities. Therefore the contaminants expected at the site are those related to vehicle maintenance. Potential contaminants at SWMU 28 include certain chlorinated and non-chlorinated VOCs; certain SVOCs – limited to PAHs; PCBs; TPH (GRO/DRO/ERO); and certain metals –

limited to cadmium, chromium, copper, lead, and zinc. The potentially impacted environmental media are surface soil, subsurface soil, sediment, surface water, and groundwater.

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 result in a complete exposure pathway. 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 28 include people who currently, or could in the future, interact with contaminated media. Current site users include occupational workers in buildings within the immediate area (Garage Area), maintenance (e.g., utilities) and construction workers, and trespassers. The area is rural, and the nearest residence is located further than one 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 soil, subsurface soil, sediment, surface water, and groundwater. Runoff and drainage from the site discharges into stream channels southeast and northwest of the site. These streams convey the runoff and discharges from SWMU 28 into Boggs Creek approximately two miles from the site. Exposure to surface water and sediment in these stream channels will be evaluated within the risk assessment if surface water and sediment concentrations of target analytes exceed risk based screening criteria.

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 is surface soil, sediment, and surface water. 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 surface water and sediment by direct contact and/or ingestion of sediment and surface water and other food items. Benthic invertebrates or other aquatic organisms 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 28 at concentrations that exceed applicable risk-based human health or ecological screening values. The data from the initial sampling event will be used with supplemental investigation data as deemed necessary by Project Team to locate contaminated areas with elevated contaminants, 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 from exposure to site contaminants, the Project Team will evaluate remedial alternatives in a Corrective Measures Study (CMS).

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, surface water, and groundwater chemical data are needed 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. TPH Fractionation Data. TPH Fractionation data will be obtained on sediment and soil samples when the PSL is exceeded for TPH Ranges DRO/GRO/ERO. TPH Fractionation data will be evaluated for potential risk based on the sum of the aliphatic and/or aromatic TPH ranges and not by the GRO/DRO/ERO fractions, which cannot be used directly to calculate risk. The IDEM Fractionation Tool allows for non-default site-specific TPH closure levels to be determined based upon a TPH Fractionation analysis, which will be necessary if a TPH GRO, DRO, or ERO PSL is exceeded. Laboratory results of the TPH Fractionation analysis will be entered into the IDEM TPH Fractionation Tool along with site-specific information, and the IDEM Fractionation Tool calculates site-specific values.

The IDEM TPH Fractionation Tool is on the IDEM website and can be accessed at: < <http://www.in.gov/idem/4210.htm>>.

3. Subsurface soil field screening data: A photoionization detector (PID) will be used to measure volatile organic vapor levels in subsurface soil samples. Subsurface soil sample depths will be targeted based on the locations of former site features. Analyses will be conducted on the most contaminated subsurface soil interval at each boring location, based on the maximum PID reading and knowledge of historical site features. Visual observations and the presence of odor 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.
4. Physical data: 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.
5. Project Screening Levels: The SWMU 28 RFI requires chemical data that can be compared to current USEPA and IDEM residential surface and subsurface soil, sediment, surface water, and groundwater risk-based screening criteria for an HHRA. The risk and regulatory criteria applicable to the SWMU 28 RFI include the IDEM Risk Integrated System of Closure (RISC) Default Closure Tables, Residential and Industrial Default 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 ESL-SD) (USEPA, 2005) 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 HHRA and ERA 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, surface water, 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 also be presented in the uncertainty section of the risk screening in the RFI Report.

6. 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 Chemical of Potential Concern (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 STUDY AREA BOUNDARIES

The study will be performed in a step-wise manner and will address the entire area of SWMU 28, as shown on Figure 11-1.

Initial Sampling Event

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

1. Horizontal: The horizontal boundary of the study is defined as soil at the outer perimeter of the area, based on information from aerial photographs and previous investigations, as identified on Figure 11-1. The outer limit of SWMU 28 in the initial sampling event will also include the discharge points of drainage pipes from the former buildings and Outside Truck Wash Rack, sediment from the stream channels southeast and northwest of the site, and groundwater downgradient from the former UST locations and Battery Disposal Area. Horizontally, the area within SWMU 28 boundaries will be investigated. Based on field observations and analytical data, step-out samples may be collected during a future field event to better define the limits of contamination. 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 approximately 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. Surface soil data will be used for direct contact soil risk evaluations. Subsurface soil data will be collected at greater than 2 feet bgs at intervals selected based on field screening with a PID and/or visual observations for potential contamination. The interval of interest for subsurface soil is the biased 2-foot interval based on the bottom depths of former site features such as USTs. If no historical knowledge of former site features is available for a subsurface soil location, the interval of interest is the biased 2-foot interval between 2 to 20 feet bgs (or the top of the water table, whichever is shallower) that demonstrates the highest PID reading or is selected by the sampler based on visual observations. If there is no historical knowledge for a location and there are no elevated 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. If subsurface soil contamination is identified through field screening during this sampling event, samples may be collected into the vadose zone just above the water table, estimated to be less than 20 feet bgs. Expansion of the vertical boundary (deeper depths) may be necessary during a future sampling event if target analyte concentrations in subsurface soil samples exceed the PSLs
3. **Sediment:** Sediment throughout the stream bed (as shown on Figure 11-1) is another population of interest; however, if sediment is not widely available, depositional areas will be targeted for sampling. Sediment data will be collected from stream channels southeast and northwest of the site at locations both upstream and downstream of the site. Upstream sediment is of interest to represent background or upgradient conditions and to assist in the delineation of elevated levels of target analytes. If a measured target analyte concentration in any sediment sample on the downstream side or exterior of the sampling pattern exceeds IDEM RISC R-DCLs and site-specific background concentrations (for metals), then additional step-out sediment and co-located surface water sampling in the future will be recommended to the Project Team to delineate the contamination.
4. **Surface Water:** Surface water sample points will be co-located with the furthest downgradient sediment location in each channel of interest (IA-8 and IA-9 as shown on Figure 11-1). If a measured target analyte concentration in any surface water sample exceeds IDEM RISC R-DCLs and site-specific background concentrations (for metals), then additional step-out surface water and co-located sediment sampling will be recommended to the Project Team to delineate the contamination.
5. **Groundwater:** Groundwater generally downgradient of the Building 1818 USTs (IA-3), Building 1820 USTs (IA-4), and Battery Disposal Area (IA-6) may have been impacted by spills or releases. Upgradient groundwater data will also be collected to provide a reference population. Groundwater data will be assessed at the site during the initial sampling round. 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. The groundwater samples will be collected from the surficial, unconfined upper

water bearing zone in unconsolidated material. If target analytes are identified in groundwater in excess of the PSLs, then the need for permanent monitoring wells will be evaluated by the Project Team. The details regarding installation of permanent monitoring wells will be presented in an addendum to this SAP.

6. 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 28 RFI initial sampling event field activities are scheduled for Spring 2011. Subsequent field sampling activities, if deemed necessary based on the initial sampling event results, will be conducted in a timely manner.

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

If any results from samples collected during the initial screening sampling event exceed a PSL (and background concentrations for metals) and the Project Team deems it necessary, one round of step-out sampling will be collected to assist in determining the extent of target analytes that exceed PSLs and to provide adequate data to conduct an HHRA and/or a screening level ERA.

Exposure Unit Boundaries for HHRA and screening level ERA

Risk exposure units (EUs) will be established within areas of SWMU 28 to define where each receptor would be exposed to the various media. Definition of EUs is critical for evaluating exposure to soil by potential human or ecological receptors and will be agreed to by the Project Team based on the initial screening sampling event results and incorporated into the Delineation of PSL Exceedances sampling design and rationale. Known areas of contamination (as determined from the initial screening sampling event), topographic features, and conclusions drawn from historical records will provide the basis for the locations of the EU boundaries.

Specific boundaries for determining exposure point concentrations (EPCs) for soil will be drawn and agreed to by the Project Team to assess potential risk to human health and/or ecological receptors. Known areas of PSL exceedances (as determined from the initial screening sampling event), topographic features, and conclusions drawn from historical records will provide the basis for the locations of the EU boundaries.

11.4 ANALYTIC APPROACH

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

Initial Sampling Event

Based on the results from the initial screening sampling event, determine whether chemical concentrations in site media (surface and subsurface soil, sediment, surface water, and groundwater) exceed the PSLs within and around areas that are the most likely areas to have been impacted by activities at SWMU 28. If chemical concentrations in site media are less than the lowest risk-based screening values, then the Project Team will recommend NFA for the site, and the Navy will submit a project close-out 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, sediment, and groundwater samples in the initial round of sampling are less than PSLs, then recommend NFA.
2. Surface soil, subsurface soil, and sediment samples will be analyzed for TPH (GRO/DRO/ERO). If the concentration of a particular TPH range (GRO, DRO, or ERO) exceeds a PSL for a sample, then the sample will be immediately sent from Empirical to Test America and analyzed for TPH Fractionation within the 14 day holding times to appropriately quantify the specific aliphatic and/or aromatic TPH ranges for use in the risk assessment(s) per the IDEM guidelines regarding TPH.
3. 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 to completely define the contaminated areas to conduct an HHRA and/or an ERA; otherwise, exclude the chemical from further consideration in the risk screening.
4. If COPCs are identified in groundwater as a result of the initial round of sampling, installation of permanent groundwater monitoring wells in a subsequent sampling round will be considered by the

Project Team to assess and monitor groundwater conditions at the site; otherwise, no additional groundwater investigation will be required.

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

If any COPCs are identified in soil, sediment, surface water, or groundwater following the initial sampling event, the Project Team will recommend returning 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 plan for the step-out sampling will be presented in an addendum to this SAP. 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 HHRA and/or ERA, 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 CMS and implementing risk management options such as institutional or engineering controls.

If additional data must be collected based on the decision rules presented above, a risk assessment will be conducted based on all of the applicable data. 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, maintenance, construction worker, and recreational user EUs will be defined as the entire area between the southern stream channel and the northern boundary of SWMU 28. The number of EUs appropriate for risk assessment of soil and sediment at SWMU 28 will be determined once all of the data for SWMU 28 has been collected and evaluated in terms of the spatial distribution of contamination. 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 COPCs, will be used in the

risk assessment(s). Random background samples collected using the Visual Sample Plan (VSP) will be used to determine the correct number of samples and specific sampling locations for random background soil samples that will be needed to support a more complete and accurate risk assessment. VSP is a software tool that supports the development of a defensible sampling plan based on statistical sampling theory and the statistical analysis of sample results to support confident decision making. 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 and surface water, 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 1×10^{-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 are within the acceptable range of 1×10^{-4} and 1×10^{-6} .

For ecological risk screening, the maximum detected concentrations of each COPC in all surface soil, sediment, and surface water samples will be compared to PSLs to determine if an analyte is a COPC. 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 ERA or consider the potential risks with respect to remedial actions.

11.5 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, surface water, 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, 2006a, 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 step-out data collected to conduct risk assessments, as well as 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 a claim of having obtained the project objectives is reasonable. 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 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 $\geq \frac{1}{2}$ 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 $\geq \frac{1}{2}$ 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 $\geq \frac{1}{2}$ LOQ, except common laboratory contaminants, which must be $<$ LOQ.	S & A
Field Duplicate (FD)	All analytical groups	One per 20 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 28.	None
Initial Assessment Study	Initial Assessment Study of Naval Weapons Support Center, Crane, Indiana	NEESA, May 1983	Data was used to identify IAs.	Due to the age of the data, data will not be used in risk calculations. The data would not necessarily represent current site conditions.
Visual Site Inspection	Preliminary Review/Visual Site Inspection	A.T. Kearney, 1987	Data was used to identify IAs.	Due to the age of the data, data will not be used in risk calculations. The data would not necessarily represent current site conditions.
Background Metals Study	Final Base-Wide Background Soil Investigation Report for Naval Surface Warfare Center Crane	Tetra Tech, 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
- Surface Water Sampling
- Groundwater Well Installation and Development
- Groundwater Sampling
- Investigation-Derived Waste (IDW) Management
- 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 two feet of soil (from 0 to 2 feet bgs). Surface soil does not include surface pavement and the ground surface will begin at the bottom of a pavement or gravel layer.

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. Soil samples will be collected from areas as described in Worksheet No. 17.

Subsurface soil samples analyzed for VOCs will be collected using TerraCore samplers and will be field preserved using deionized water (two 40-milliliter [mL] vials) and methanol (one 40-mL vial). Soil samples analyzed for TPH will be sent to Empirical and analyzed on an expedited basis for TPH (GRO/DRO/ERO). Soil samples analyzed for TPH (GRO) will be field preserved using methanol (three 40-ml vials). If TPH (GRO/DRO/ERO) is detected above a PSL in a sample, the sample will be sent to Test America for TPH Fractionation analysis. Test America will conduct TPH Fractionation analysis of the sample within the 14 day holding time.

Sediment Sampling

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

Surface Water Sampling

The surface water sampling procedures discussed in SOP-18 (Surface Water 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). The initial screening groundwater samples will be collected using DPT temporary wells, defined as a 1" PVC screen placed within the DPT.

Groundwater Well Installation and Development

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). If determined to be necessary by the Project Team based on the initial screening sampling event results, additional temporary and/or permanent groundwater wells will be installed in accordance with SOP-12 (Monitoring Well Installation, Appendix A).

Investigation-Derived Waste Management

It is not anticipated that significant volumes of solid or semi-solid investigation derived waste (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 each 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 Test America. 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 numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- 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.
- Description of subcontractor activities.
- Sample pick-up 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.
- Health and safety issues.

- Description of photographs including date, time, photographer, picture number, location, and compass direction of each photograph.

All logbook entries will be written in ink, and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction, and the person making the correction will initial and date the change.

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 the 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, PAHs, PCBs, TPH (GRO/DRO/ERO), Metals, pH, Lead, and TOC will be performed by Empirical, which is a current Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP) accredited laboratory. Chemical analysis for TPH Fractionation (if necessary, based on results of TPH [GRO/DRO/ERO] analyses) will be performed by Test America, which is also a current DoD ELAP accredited laboratory. Copies of the DoD ELAP accreditation for Empirical and Test America are included in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 30. Empirical and Test America 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 Solutions (NIRIS). Percent moisture information will also be captured in the RFI Report.

The analytical data packages provided by Empirical and Test America will be in a Contract Laboratory Program (CLP)-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 Test America 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 Test America. Upon receipt of the data packages from Empirical and Test America, 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 Test America.
- **Data Storage, Archiving, and Retrieval.** The data packages received from Empirical and Test America 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 (CA) 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 UFP-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 be submitted in hardcopy and electronic format to the project stakeholders.

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-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
1,2,4-Trichlorobenzene	120-82-1	0.14	11.1	RBSSL	R5 ESL-S	0.047	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
Benzene	71-43-2	0.0042	0.255	RBSSL	R5 ESL-S	0.0014	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
Ethylbenzene	100-41-4	0.034	5.16	RBSSL	R5 ESL-S	0.011	0.005	0.0025	0.00125
Methyl-tert-butyl ether	1634-04-4	0.056	NC	RBSSL	None	0.019	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
Trichloroethene	79-01-6	0.014	12.4	RBSSL	R5 ESL-S	0.0047	0.005	0.0025	0.00125
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: 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)
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
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.

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: 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, 2005). 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: TPH (GRO/DRO/ERO)

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)
GRO (C5-C12) Gasoline Range	NA	120	NC	R-DCL	None	40	7.50	5.00	2.50
DRO (C8-C28) Diesel Range	NA	230	NC	R-DCL	None	77	6.67	6.67	6.67
ERO (C8-C34) High End Hydrocarbons	NA	230	NC	R-DCL	None	77	6.67	6.67	6.67

1 Surface and subsurface soil screening references: R-DCL – IDEM Residential Default Closure Level (June, 2010). 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: TPH Fractionation (If a TPH GRO, DRO, or ERO PSL is exceeded)

Analyte	CAS Number	PSL (mg/kg)		PSL Reference ¹		PQLG (mg/kg)	Test America		
		HHRA ²	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
EC 5-6 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	2	0.3	0.1
EC > 6-8 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	2	0.3	0.1
EC > 8-10 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5	0.05	0.027
EC > 10-12 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5	0.2	0.095
EC > 12-16 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC > 16-21 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC > 21-34 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC 8-10 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC > 10-12 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5	0.15	0.072
EC > 12-16 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC > 16-21 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1
EC > 21-34 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5	2	1

EC = Equivalent Carbon #

- 1 Surface and subsurface soil screening references: R-DCL – IDEM Residential Default Closure Level (June, 2010). Refer to Appendix E for further explanation and justification of PSLs.
- 2 Value will be determined based on IDEM TPH Fractionation Tool.

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: Metals

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)
Cadmium	7440-43-9	7.0	0.36	R-RSL	Eco-SSL	0.12	0.25	0.10	0.05
Chromium	7440-47-3	0.017	26	RBSSL	Eco-SSL	0.0057	0.25	0.20	0.10
Copper	7440-50-8	310	28	R-RSL	Eco-SSL	9.3	0.5	0.4	0.25
Lead	7439-92-1	81	11	R-DCL	Eco-SSL	3.7	0.15	0.15	0.075
Zinc	7440-66-6	2,300	46	R-RSL	Eco-SSL	15	1.0	0.5	0.25

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, 2005). 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: 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 ESLSD	0.63	0.005	0.0025	0.00125
1,1,2,2-Tetrachloroethane	79-34-5	0.007	0.127	R-DCL	R5 ESLSD	0.0023	0.005	0.0025	0.00125
1,1,2-Trichloroethane	79-00-5	0.03	28.6	R-DCL	R5 ESLSD	0.010	0.005	0.0025	0.00125
1,1-Dichloroethane	75-34-3	3.3	20.1	R-RSL	R5-ESLSD	1.1	0.0005	0.0025	0.00125
1,1-Dichloroethene	75-35-4	0.058	8.28	R-DCL	R5 ESLSD	0.019	0.005	0.0025	0.00125
1,2-Dichloroethane	107-06-2	0.024	21.2	R-DCL	R5 ESLSD	0.0080	0.005	0.0025	0.00125
Benzene	71-43-2	0.034	0.142	R-RSL	R5 ESLSD	0.011	0.005	0.0025	0.00125
Chloroethane	75-00-3	0.65	NC	R-DCL	None	0.22	0.01	0.005	0.0025
Chloromethane	74-87-3	12	NC	R-RSL	R5 ESLSD	4.0	0.01	0.005	0.0025
cis-1,2-Dichloroethene	156-59-2	0.4	0.784	R-DCL	R5 ESLSD	0.13	0.005	0.0025	0.00125
Ethylbenzene	100-41-4	5.4	0.175	R-RSL	R5 ESLSD	0.058	0.005	0.0025	0.00125
MTBE	1634-04-4	0.18	NC	R-DCL	None	0.060	0.005	0.0025	0.00125
Tetrachloroethene	127-18-4	0.058	9.92	R-DCL	R5 ESLSD	0.019	0.005	0.0025	0.00125
Toluene	108-88-3	12	1.22	R-DCL	R5 ESLSD	0.41	0.005	0.0025	0.00125
trans-1,2-Dichloroethene	156-60-5	0.68	0.784	R-DCL	R5 ESLSD	0.23	0.005	0.0025	0.00125
Trichloroethene	79-01-6	0.057	12.4	R-DCL	R5 ESLSD	0.019	0.005	0.0025	0.00125
Vinyl chloride	75-01-4	0.013	0.202	R-DCL	R5 ESLSD	0.0043	0.005	0.0025	0.00125
Xylenes (Total)	1330-20-7	63	0.433	R-RSL	R5 ESLSD	0.14	0.01	0.005	0.0025

Notes:

¹ Sediment screening references: R-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 ESLSD – USEPA Region 5 Ecological Screening Level (August, 2005). 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: 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)
2-Methylnaphthalene	91-57-6	3.1	0.0202	R-DCL	R5 ESLSD	0.0067	0.010	0.005	0.0025
Acenaphthene	83-32-9	130	0.00671	R-DCL	R5 ESLSD	0.0022	0.010	0.005	0.0025
Acenaphthylene	208-96-8	18	0.00587	R-DCL	R5 ESLSD	0.0020	0.010	0.005	0.0025
Anthracene	120-12-7	1,700	0.0572	R-RSL	R5 ESLSD	0.019	0.010	0.005	0.0025
Benzo(a)anthracene	56-55-3	0.15	0.108	R-RSL	R5 ESLSD	0.036	0.010	0.005	0.0025
Benzo(a)pyrene	50-32-8	0.015	0.15	R-RSL	R5 ESLSD	0.0050	0.010	0.005	0.0025
Benzo(b)fluoranthene	205-99-2	0.15	10.4	R-RSL	R5 ESLSD	0.050	0.010	0.005	0.0025
Benzo(g,h,i)perylene	191-24-2	170	0.17	R-RSL	R5 ESLSD	0.057	0.010	0.005	0.0025
Benzo(k)fluoranthene	207-08-9	1.5	0.24	R-RSL	R5 ESLSD	0.080	0.010	0.005	0.0025
Chrysene	218-01-9	15	0.166	R-RSL	R5 ESLSD	0.055	0.010	0.005	0.0025
Dibenzo(a,h)anthracene	53-70-3	0.015	0.033	R-RSL	R5 ESLSD	0.0050	0.010	0.005	0.0025
Fluoranthene	206-44-0	230	0.423	R-RSL	R5 ESLSD	0.14	0.010	0.005	0.0025
Fluorene	86-73-7	170	0.0774	R-DCL	R5 ESLSD	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 ESLSD	0.050	0.010	0.005	0.0025
Naphthalene	91-20-3	0.7	0.176	R-RSL	R5 ESLSD	0.059	0.010	0.005	0.0025
Phenanthrene	85-01-8	13	0.204	R-DCL	R5 ESLSD	0.068	0.010	0.005	0.0025
Pyrene	129-00-0	170	0.195	R-RSL	R5 ESLSD	0.065	0.010	0.005	0.0025

Notes:

- 1 Sediment screening references: R-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Residential Direct Contact, adjusted to 1/10 of value for noncarcinogens (November, 2010); R-DCL – IDEM Residential Default Closure Level (May, 2009); R5 ESLSD – USEPA Region 5 Ecological Screening Level (August, 2005). Refer to Appendix D 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: 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 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1221	11104-28-2	0.14	0.0598	R-RSL	R5 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1232	11141-16-5	0.14	0.0598	R-RSL	R5 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1242	53469-21-9	0.22	0.0598	R-RSL	R5 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1248	12672-29-6	0.22	0.0598	R-RSL	R5 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1254	11097-69-1	0.11	0.0598	R-RSL	R5 ESLSD	0.020	0.017	0.008	0.004
Aroclor-1260	11096-82-5	0.22	0.0598	R-RSL	R5 ESLSD	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); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); R5 ESLSD – USEPA Region 5 Ecological Screening Level, Sediment (August, 2005); R5 ESL-S – USEPA Region 5 Ecological Screening Level, Soil (August, 2005). 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 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: TPH (GRO/DRO/ERO)

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)
GRO (C5-C12) Gasoline Range	NA	120	NC	R-DCL	None	40	7.50	5.00	2.50
DRO (C8-C28) Diesel Range	NA	230	NC	R-DCL	None	77	6.67	6.67	6.67
ERO (C8-C34) High End Hydrocarbons	NA	230	NC	R-DCL	None	77	6.67	6.67	6.67

Notes:

1 Sediment screening references: R-DCL – IDEM Residential Default Closure Level (June, 2010). 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: TPH Fractionation (If a TPH GRO, DRO, or ERO PSL is Exceeded)

Analyte	CAS Number	PSL (mg/kg)		PSL Reference ¹		PQLG (mg/kg)	Test America		
		HHRA ²	ERA	HHRA	ERA		LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
EC 5-6 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	2.0	0.3	0.1
EC > 6-8 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	2.0	0.3	0.1
EC > 8-10 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5.0	0.05	0.027
EC > 10-12 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5.0	0.2	0.095
EC > 12-16 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC > 16-21 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC > 21-34 Aliphatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC 8-10 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC > 10-12 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5.0	0.15	0.072
EC > 12-16 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC > 16-21 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0
EC > 21-34 Aromatics	NA	TBD	NC	R-DCL	None	TBD	5.0	2.0	1.0

Notes:

EC = Equivalent Carbon #

1 Sediment screening references: R-DCL – IDEM Residential Default Closure Level (June, 2010). Refer to Appendix E for further explanation and justification of PSLs.

2 Value will be determined based on IDEM TPH Fractionation Tool.

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: Metals

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)
Cadmium	7440-43-9	7.0	0.99	R-RSL	R-5 ESLSD	0.33	0.25	0.10	0.05
Chromium	7440-47-3	0.29	43.4	R-RSL	R-5 ESLSD	0.097	0.25	0.20	0.10
Copper	7440-50-8	310	31.6	R-RSL	R-5 ESLSD	10	0.5	0.4	0.25
Lead	7439-92-1	81	11	R-DCL	Eco-SSL	3.7	0.15	0.15	0.075
Zinc	7440-66-6	2,300	121	R-RSL	R-5 ESLSD	40	1.0	0.5	0.25

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); R-5 ESLSD – USEPA Region 5 Ecological Screening Level, Sediment (August, 2005); Eco-SSL – USEPA Ecological Soil Screening Levels (2005-2008); Refer to Appendix E for further explanation and justification of PSLs.

Bolded rows indicated 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 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: Surface Water
 Analytical Group: VOCs

Analyte	CAS Number	PSL (µg/kg)		PSL Reference ¹		PQLG (µg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
1,1,1-Trichloroethane	71-55-6	200	76	MCL	R5 ESLW	25	1.0	0.5	0.25
1,1,2,2-Tetrachloroethane	79-34-5	0.067	380	T-RSL	R5 ESLW	0.022	1.0	0.5	0.25
1,1,2-Trichloroethane	79-00-5	0.24	500	T-RSL	R5 ESLW	0.080	1.0	0.5	0.25
1,1-Dichloroethane	75-34-3	2.4	47	T-RSL	R5 ESLW	0.80	1.0	0.5	0.25
1,1-Dichloroethene	75-35-4	7	65	MCL	R5 ESLW	2.3	1.0	0.5	0.25
1,2-Dichloroethane	107-06-2	0.15	910	T-RSL	R5 ESLW	0.050	1.0	0.5	0.25
Benzene	71-43-2	0.41	114	T-RSL	R5 ESLW	0.14	1.0	0.5	0.25
Chloroethane	75-00-3	62	NC	G-DCL	R5 ESLW	21	2.0	1.0	0.50
Chloromethane	74-87-3	19	NC	T-RSL	R5 ESLW	6.3	1.0	0.5	0.25
cis-1,2-Dichloroethene	156-59-2	7.3	970	T-RSL	R5 ESLW	12	1.0	0.5	0.25
Ethylbenzene	100-41-4	1.5	14	T-RSL	R5 ESLW	0.50	1.0	0.5	0.25
MTBE	1634-04-4	12	11,070	T-RSL	R3 BTAG FW SW	4.0	1.0	0.5	0.25
Tetrachloroethene	127-18-4	0.11	45	T-RSL	R5 ESLW	0.037	1.0	0.5	0.25
Toluene	108-88-3	230	253	T-RSL	R5 ESLW	77	1.0	0.5	0.25
trans-1,2-Dichloroethene	156-60-5	11	970	T-RSL	R5 ESLW	3.7	10.0	0.5	0.25
Trichloroethene	79-01-6	2	47	T-RSL	R5 ESLW	0.67	1.0	0.5	0.25
Vinyl chloride	75-01-4	0.016	65.7	T-RSL	R5 ESLW	0.0053	1.0	0.5	0.25
Xylenes (Total)	1330-20-7	20	27	T-RSL	R5 ESLW	6.7	2.0	1.0	0.50

Notes:

µg/L – micrograms per liter

- 1 Surface Water screening references: T-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009); MCL – USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (December, 2009); R5 ESL-W – USEPA Region 5 Ecological Screening Level, Water (August, 2005); R3 BTAG FW SW – USEPA Region 3 Biological Technical Assistance Group Freshwater Surface Water (July, 2006). 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.

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Matrix: Surface Water
 Analytical Group: PAHs

Analyte	CAS Number	PSL (µg/kg)		PSL Reference ¹		PQLG (µg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (µg/kg)	LOD (µg/kg)	DL µ (µg/kg)
2-Methylnaphthalene	91-57-6	15	330	T-RSL	R5 ESLW	5.0	5.0	2.5	1.25
Acenaphthene	83-32-9	220	38	T-RSL	R5 ESLW	13	5.0	2.5	1.25
Acenaphthylene	208-96-8	71	4,840	G-DCL	R5 ESLW	24	5.0	2.5	1.25
Anthracene	120-12-7	1,100	0.035	T-RSL	R5 ESLW	0.012	5.0	2.5	1.25
Benzo(a)anthracene	56-55-3	0.029	0.025	T-RSL	R5 ESLW	0.0083	5.0	2.5	1.25
Benzo(a)pyrene	50-32-8	0.0029	0.014	T-RSL	R5 ESLW	0.00097	5.0	2.5	1.25
Benzo(b)fluoranthene	205-99-2	0.029	9.07	T-RSL	R5 ESLW	0.0097	5.0	2.5	1.25
Benzo(g,h,i)perylene	191-24-2	110	7.64	T-RSL	R5 ESLW	2.5	5.0	2.5	1.25
Benzo(k)fluoranthene	207-08-9	0.29	NC	T-RSL	None	0.097	5.0	2.5	1.25
Chrysene	218-01-9	2.9	NC	T-RSL	None	0.97	5.0	2.5	1.25
Dibenzo(a,h)anthracene	53-70-3	0.0029	NC	T-RSL	None	0.00097	5.0	2.5	1.25
Fluoranthene	206-44-0	150	1.9	T-RSL	R5 ESLW	0.63	5.0	2.5	1.25
Fluorene	86-73-7	150	19	T-RSL	R5 ESLW	6.3	5.0	2.5	1.25
Indeno(1,2,3-cd)pyrene	193-39-5	0.029	4.31	T-RSL	R5 ESLW	0.0097	5.0	2.5	1.25
Naphthalene	91-20-3	0.14	13	T-RSL	R5 ESLW	0.047	5.0	2.5	1.25
Phenanthrene	85-01-8	23	3.6	T-RSL	R5 ESLW	1.2	5.0	2.5	1.25
Pyrene	129-00-0	110	0.3	T-RSL	R5 ESLW	0.10	5.0	2.5	1.25

Notes:

1 Surface Water screening references: T-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); G-DCL – IDEM Groundwater Default Closure Level (May, 2009); MCL – USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (December, 2009); R5 ESL-W – USEPA Region 5 Ecological Screening Level, Water (August, 2005). 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.

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Matrix: Surface Water
 Analytical Group: PCBs

Analyte	CAS Number	PSL (µg/kg)		PSL Reference ¹		PQLG (µg/kg)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (µg/kg)	LOD (µg/kg)	DL (µg/kg)
Aroclor-1016	12674-11-2	0.5	0.00012	MCL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1221	11104-28-2	0.0068	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1232	11141-16-5	0.0068	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1242	53469-21-9	0.034	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1248	12672-29-6	0.034	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1254	11097-69-1	0.034	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Aroclor-1260	11096-82-5	0.034	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125
Total PCBs	1336-36-3	0.17	0.00012	T-RSL	R5 ESLW	0.000040	0.500	0.250	0.125

Notes:

1 Surface Water screening references: T-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (December, 2009); R5 ESL-W – USEPA Region 5 Ecological Screening Level, Water (August, 2005). 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: Surface Water
 Analytical Group: Metals

Analyte	CAS Number	PSL (µg/L)		PSL Reference ¹		PQLG (µg/L)	Empirical		
		HHRA	ERA	HHRA	ERA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Cadmium	7440-43-9	1.8	0.15	T-RSL	R5 ESLW	0.05	5.0	2.0	1.0
Chromium	7440-47-3	0.043	42	T-RSL	R5 ESLW	0.014	10	4.0	2.0
Copper	7440-50-8	15	1.58	T-RSL	R5 ESLW	0.53	10	8.0	4.0
Lead	7439-92-1	15	1.17	MCL	R5 ESLW	0.39	3.0	3.0	1.5
Zinc	7440-66-6	1,100	65.7	T-RSL	R5 ESLW	22	20	10	5.0

Notes:

1 Surface Water screening references: T-RSL – USEPA Regions 3, 6, and 9 Regional Screening Levels, Tapwater, adjusted to 1/10 of value for noncarcinogens (November, 2010); MCL – USEPA Maximum Contaminant Levels, National Primary Drinking Water Regulations (December, 2009); R5 ESL-W – USEPA Region 5 Ecological Screening Level, Water (August, 2005). 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.

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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)
1,1,2,2-Tetrachloroethane	79-34-5	0.067	T-RSL	0.022	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-Dichloroethane	107-06-2	0.15	T-RSL	0.050	1.0	0.5	0.25
Benzene	71-43-2	0.41	T-RSL	0.14	1.0	0.5	0.25
Chloroethane	75-00-3	62	G-DCL	21	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	12	1.0	0.5	0.25
Ethylbenzene	100-41-4	1.5	T-RSL	0.50	1.0	0.5	0.25
Methyl-tert-butyl ether	1634-04-4	12	T-RSL	4.0	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
trans-1,2-Dichloroethene	156-60-5	11	T-RSL	3.7	1.0	0.5	0.25
Trichloroethene	79-01-6	2.0	T-RSL	0.67	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

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; November, 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: PAHs

Analyte	CAS Number	Project Screening Level (ug/L)	Project Screening Level References HHRA	Project Quantitation Limit Goal (ug/L)	Empirical		
		HHRA			LOQ (ug/L)	LOD (ug/L)	DL (ug/L)
2-Methylnaphthalene	91-57-6	15	T-RSL	5.0	5.0	2.50	1.25
Acenaphthene	83-32-9	220	T-RSL	73	5.0	2.50	1.25
Acenaphthylene	208-96-8	220	T-RSL	73	5.0	2.50	1.25
Anthracene	120-12-7	1,100	T-RSL	370	5.0	2.50	1.25
Benzo(a)anthracene	56-55-3	0.029	T-RSL	0.0097	5.0	2.50	1.25
Benzo(a)pyrene	50-32-8	0.0029	T-RSL	0.00097	5.0	2.50	1.25
Benzo(b)fluoranthene	205-99-2	0.029	T-RSL	0.0097	5.0	2.50	1.25
Benzo(g,h,i)perylene	191-24-2	110	T-RSL	37	5.0	2.50	1.25
Benzo(k)fluoranthene	207-08-9	0.29	T-RSL	0.097	5.0	2.50	1.25
Chrysene	218-01-9	2.9	T-RSL	0.97	5.0	2.50	1.25
Dibenzo(a,h)anthracene	53-70-3	0.0029	T-RSL	0.00097	5.0	2.50	1.25
Fluoranthene	206-44-0	150	T-RSL	50	5.0	2.50	1.25
Fluorene	86-73-7	150	T-RSL	50	5.0	2.50	1.25
Indeno(1,2,3-cd)pyrene	193-39-5	0.029	T-RSL	0.0097	5.0	2.50	1.25
Naphthalene	91-20-3	0.14	T-RSL	0.047	5.0	2.50	1.25
Phenanthrene	85-01-8	23	G-DCL	7.7	5.0	2.50	1.25
Pyrene	129-00-0	110	T-RSL	37	5.0	2.50	1.25

Notes:

- 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; November, 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	0.50	MCL	0.17	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; November, 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: Metals

Analyte	CAS Number	PSL (µg/L)	PSL Reference ¹	PQLG (µg/L)	Empirical		
		HHRA	HHRA		LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Cadmium	7440-43-9	1.8	T-RSL	0.60	1.5	0.50	0.25
Chromium	7440-47-3	0.043	T-RSL	0.014	10	4.0	2.0
Copper	7440-50-8	150	T-RSL	50	10	8.0	4.0
Lead	7439-92-1	15	T-RSL	5.0	3.0	3.0	1.5
Zinc	7440-66-6	1,100	T-RSL	370	20	10	5.0

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	05/09/2011	05/27/2011	RFI Report (if no additional sampling)	01/06/2012 (draft) 10/1/2012 (final)
Additional step-out and other sampling, if necessary	Tetra Tech	07/08/2011	07/22/2011	RFI Report (if additional sampling is performed)	04/06/2012 (draft) 01/11/2013 (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 28 and will also include drainage paths to the stream and the streambed. Initial samples will be collected at SWMU 28 to determine if there are target analytes in environmental media as a result of operations that occurred on-site. 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. Additional sampling locations will be presented in an addendum to this SAP.

Chemicals that may have been present at the two vehicle maintenance buildings over its operational history and have the potential to impact environmental media include the following:

- **Degreasing solvents:** Chlorinated solvents that may have been used to clean vehicle parts include tetrachloroethene (PCE), trichloroethene (TCE), 1,1,1-trichloroethane (TCA), and associated daughter products formed in the environment as these compounds degrade, including cis-1,2-dichloroethene (DCE), trans-1,2 DCE, 1,1-DCE, 1,2-dichloroethane (DCA), 1,1-DCA, vinyl chloride, chloroethane, and chloromethane, which may have leaked onto the ground and leached into the subsurface.
- **Fuels, oils, and greases associated with vehicle maintenance and use:** VOCs associated with gasoline, including benzene, toluene, ethylbenzene, and total xylenes (BTEX) and methyl tert-butyl ether (MTBE); and SVOCs associated with waste oil, diesel fuel, motor oils, lubricants, and greases, specifically including PAHs. These compounds can also be quantified by a TPH analysis. The initial TPH analysis will consist of three general ranges roughly based on their boiling points – GRO, DRO, and ERO. If a TPH (GRO/DRO/ERO) concentration in a sample exceeds a PSL, then the sample will also be analyzed for TPH Fractionation. The TPH Fractionation includes seven aliphatic hydrocarbon ranges and five aromatic hydrocarbon ranges that can be used in risk assessments.
- **Hydraulic fluids:** Hydraulic fluids associated with waste oil and vehicle lifts may have contained various PCBs (including the seven TCL Aroclors identified in Worksheet No. 15 and Total PCBs), which may have leaked onto the ground and leached into the subsurface.
- **Metals:** Various metals may have been released onto the ground, which may then have leached into the subsurface. Possible metals contaminants include cadmium, chromium, copper, lead, and zinc from handling motor oils, waste oils, vehicle batteries, and radiators.

Soil Sampling

The initial soil sampling program consists of collecting surface soil and subsurface soil from biased locations across the site. In many instances, surface and subsurface soil samples will be horizontally aligned. Subsurface soil samples will be collected from the depth interval with the greatest potential for contamination based on historical knowledge, site features, and field screening techniques. This approach, coupled with the ability to make field decisions to determine the extent of contamination as described as follows, supports both the delineation of contamination and the risk assessment objectives.

Initial surface and subsurface samples will be collected from locations that have a greater potential for contamination than other areas of the site (based on site history). Biased samples will be collected from these locations that appear most likely to have been impacted by site activities; or in the general vicinity, if no discernable impact is evident. The proposed initial screening soil sampling locations are shown on Figure 17-1, 17-2, and 17-3. If site conditions require a location to be moved (i.e., utilities or parked equipment), the field sampler will move to an alternate location to collect the necessary sample. If this occurs, the field sampler will document the alternate location and the reason in the field logbook. Surface and subsurface soil samples will be analyzed for one or more of the following depending on the CSM and the location of the sample within the site: VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), metals, pH, and lead.

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 – Gravel Areas Adjacent to Building 1818:** The area east of Building 1818 is currently paved, but was historically graveled. Reports indicate that many general releases may have occurred in this area. Soil beneath the gravel area may be contaminated with residual VOCs, PAHs, PCBs, TPH, or metals. The gravel area will be defined as the area immediately adjacent to the eastern face of the building, extending 270 feet in length and 60 feet in width. Five surface soil samples will be located within 30 feet of the eastern wall of the building, and five surface soil samples will be located greater than 30 feet from the eastern wall of the building. Five subsurface soil samples will be vertically aligned with the surface soil samples closest to Building 1818. Surface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Subsurface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, 10 surface soil samples and five subsurface soil samples will be collected from IA-1.
- **IA-2 – Gravel Areas Adjacent to Building 1820:** The area south of Building 1820 is currently paved, but was historically graveled. Reports indicate that many general releases may have occurred in this area. Soil beneath the gravel area may be contaminated with residual VOCs, PAHs, PCBs, TPH, or metals. The gravel area will be defined as the area immediately adjacent to the southern face of the building, extending 270 feet in length and 60 feet in width. Five surface soil samples will be located

within 30 feet of the southern wall of the building, and five surface soil samples will be located greater than 30 feet from the southern wall of the building. Five subsurface soil samples will be vertically aligned with the surface soil samples closest to Building 1818. Surface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Subsurface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, 10 surface soil samples and five subsurface soil samples will be collected from IA-2.

- **IA-3 – Underground Waste Oil Storage Tanks, Building 1818:** There are four known former waste oil UST locations associated with Building 1818. Soil near and beneath the USTs may be contaminated with residual VOCs, PAHs, PCBs, TPH, or metals. Three subsurface soil samples will be collected from three of the former UST locations. An historic photo shows a potential release from the southeast UST in the area; therefore, five subsurface soil samples will be collected from this location. Subsurface sample depths will generally be targeted based on the depths of the USTs. Subsurface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, 14 subsurface soil samples will be collected from IA-3.
- **IA-4 – Underground Waste Oil Storage Tanks, and Oil Pan Disposal Rack, Building 1820:** There are two known former waste oil UST locations associated with Building 1820. One UST location was vertically aligned with the Oil Pan Disposal Rack that formerly existed at Building 1820. Soil near and beneath the USTs and Oil Pan Disposal Rack may be contaminated with residual VOCs, PAHs, PCBs, TPH, or metals. Three subsurface soil samples will be collected from the two former UST locations. Subsurface sample depths will generally be targeted based on the depths of the USTs. Three surface soil samples will be collected from the former location of the Oil Pan Disposal Rack. Surface soil samples will be vertically aligned with the subsurface samples associated with the UST. Surface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Subsurface soil samples will be analyzed for VOCs, PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, three surface soil samples and six subsurface soil samples will be collected from IA-4.
- **IA-5 – Drainage Pipe Discharges:** There are seven identified pipe discharge points located in the valley east of Building 1818 and south of Building 1820 associated with SWMU 28. The discharge points include pipes that drained from floor drains, sumps, and other facilities in and around the buildings. Soil in the drainage paths from drainage outlets in the valley may be contaminated with residual PAHs, PCBs, TPH, or metals. Three of the discharge points are associated with Building 1818, two are associated with Building 3387, and two are associated with Building 1820. One surface soil sample will be collected from directly below the outlet from each discharge point that remains on-site. An additional surface soil sample may be collected from the drainage path associated with each discharge point. Additional surface soil samples will be collected at the discretion of the Tetra Tech FOL, based on field observations. If the drain pipes are determined to be Vitrified Clay Tile (VCT) and PSL exceedances are found in these samples during the initial screening

sampling event, additional sampling may be performed along the length of the pipes, and analysis may warrant the inclusion of VOCs. Six surface soil samples will be collected from the drainage paths associated with the three Building 1818 discharge points, four surface soil samples will be collected from the drainage paths associated with the three Building 1820 discharge points, and four surface soil samples will be collected from the drainage paths associated with the Building 3387 discharge points. Surface soil samples will be analyzed for PAHs, PCBs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, 14 surface soil samples will be collected from IA-5.

- **IA-6 - Battery Disposal Area:** A historical report indicates that for an unknown period of time, 200 to 300 batteries per year were disposed at SWMU 28. Battery acid was reportedly dumped in a valley behind the building, although the activity had stopped prior to the IAS (NEESA, 1983). Based on historical aerial photographs, a valley was centrally located between Building 1818 and 1820. This valley is the current location of Building 3387, and it appears to have been filled after 2003 for the construction of the building. The approximate location of this area is shown on Figure 17-2. This valley may contain the battery disposal area and will be investigated during the initial screening sampling event. Evidence of battery disposal may include, but is not limited to, the presence of automotive batteries or pieces of automotive batteries, stressed or no vegetation, and surface disturbance associated with dumping activities. Multiple DPT soil borings will be placed in the former valley area to locate evidence of the battery disposal. Two surface soil samples will be collected if evidence of battery disposal is observed in the former valley area. If a large area or multiple discrete areas are identified during the visual inspection, additional surface soil samples will be collected as needed based on the field observations. A discrete battery disposal area will be defined as an area that is further than 25 linear feet in all directions from another area identified as showing evidence of battery disposal. Additional samples will be collected at a rate of one sample for every contiguous 500 square foot area containing evidence of historical battery disposal, or two samples from each discrete area identified during the inspection. Additionally, three subsurface soil samples will be collected from the Building 3387 area from depths that are estimated to be the original surface as visually determined by the sampler from the DPT core. Surface and subsurface soil samples will be analyzed for pH and lead. Based on the CSM, four surface soil samples and three subsurface samples will be collected from IA-6.
- **IA-7 – Outside Truck Wash Rack:** Soils near and beneath the Outside Truck Wash Rack may be contaminated with residual PAHs, TPH, or metals. Four surface soil samples will be collected from this area based on the drainage path from the Truck Wash Rack. Surface soil samples will be analyzed for PAHs, TPH (GRO/DRO/ERO), and metals. Based on the CSM, four surface soil samples will be collected from IA-7.

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. The subsurface soil sample will be collected from the specific 2-foot boring that has the greatest potential for

contamination based on historical knowledge and visual and field instrument screening as determined in the field. The subsurface soil samples may be collected to 20 feet bgs or the top of the water table, whichever is shallower. If there are no obvious signs of contamination or no historical knowledge pertaining to the sample location, the sample will be collected from the 2- to 4-foot interval.

Soil Sample Quantities - A minimum of 45 surface soil samples (10 samples from IA-1, 10 samples from IA-2, 3 samples from IA-4, 14 samples from IA-5, 4 samples from IA-6 and 4 samples from IA-7), plus a minimum of 5 field duplicate samples for QC purposes; and 33 subsurface soil samples (5 samples from IA-1, 5 samples from IA-2, 14 samples from IA-3, 6 samples from IA-4, and 3 samples from IA-6), plus a minimum of 4 field duplicate samples for QC purposes, will be collected and analyzed for certain target analytes based on the CSM and as described above. 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. Additional samples may also be collected as described above at IA-5 and IA-6. 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 approximately 25 feet away in each general direction (north, east, south, or west) where PSL exceedances from the initial screening are unbounded. The step-out sampling distances and direction may be adjusted by FOL based on visual observations, and other conditions in the field. 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 will be collected using a DPT or hand augers, if necessary. The locations of these samples will be presented in an addendum to this SAP.

Based on a review of the analytical data and risk screening calculations from the initial screening sampling event, the Project Team will determine if it is necessary to perform an HHRA and/or an ERA in accordance with Sections 11.4 and 11.5.

Sediment Sampling

The initial screening sediment sampling program consists of collecting surface sediment samples from two IAs - the stream southeast of SWMU 28 and the stream channel northwest of SWMU 28. 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 17-3.

IA-8 – Stream Southeast of Site: Sediments in the stream southeast of SWMU 28 may be contaminated with PAHs, PCBs, TPH, or metals. Sediment samples will also be analyzed for TOC to support site-specific risk calculations. Five sediment samples will be collected from this area – one upgradient, two downgradient of Building 1818 and two downgradient of Building 1820. Sediment samples will be analyzed for PAHs, PCBs, TPH (GRO/DRO/ERO), metals, and TOC. Based on the CSM, five sediment samples will be collected from IA-8. The five IA-8 sediment sampling locations are shown on Figure 17-3.

IA-9 – Stream Channel Northwest of Site: Sediments in the stream channel northwest of SWMU 28 may be contaminated with PAHs, PCBs, TPH, or metals. Sediment samples will also be analyzed for TOC to support site-specific risk calculations. Three sediment samples will be collected from this area – one upgradient of SWMU 28 and two downgradient. Sediment samples will be analyzed for PAHs, PCBs, TPH (GRO/DRO/ERO), metals, TOC. The three IA-9 sediment sampling locations are shown on Figure 17-3

Sediment Sample Quantities - A minimum of 8 sediment samples (5 samples from IA-8 and 3 samples from IA-9), plus 1 field duplicate sample for QC purposes, will be collected during the initial screening sampling event and analyzed for potential target analytes based on the CSM and as described above. Additionally, at the discretion of the Tetra Tech FOL, the sampler may collect up to 4 step-out samples when areas of obvious or likely contamination are encountered during the initial sampling event. The flexibility to collect these additional samples extends horizontally. 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. Samples will be collected using a hand trowel.

Based on the initial sample results, the Project Team will determine if one round of step-out sample collection is required. Up to four additional step-out sediment samples will be collected 100 feet further downgradient or upgradient from where the PSL exceedances from the initial sampling event are unbounded. The step-out sampling distances and direction may be adjusted by FOL based on visual

observations, and other conditions in the field. Sections 11.4 and 11.5 will be used to guide the Project Team in this decision as well as sample location maps presented in a SAP Addendum.

Surface Water Sampling

The initial screening surface water sampling program consists of collecting one surface water sample from each of two IAs - the stream southeast of SWMU 28 and the stream channel northwest of SWMU 28. The proposed surface water sample locations were adjusted to follow the drainage path, and further adjustments may be necessary in the field if surface water is not available at the proposed locations. The planned surface water sample locations are presented on Figure 17-3.

- **IA-8 – Stream Southeast of Site:** Surface water in the stream southeast of SWMU 28 may be contaminated with VOCs, PAHs, PCBs, or metals. Water quality parameters will also be recorded at surface water locations. One surface water sample will be co-located with the sediment sample furthest downgradient this stream channel. Surface water samples will be analyzed for VOCs, PAHs, PCBs, and metals. Based on the CSM, one surface water sample will be collected from IA-8.
- **IA-9 – Stream Channel Northwest of Site:** Surface water in the stream channel northwest of SWMU 28 may be contaminated with VOCs, PAHs, PCBs, or metals. Water quality parameters will also be recorded at surface water locations. One surface water sample will be co-located with the sediment sample furthest downgradient this stream channel. Surface water samples will be analyzed for VOCs, PAHs, PCBs, and metals. Based on the CSM, one surface water sample will be collected from IA-9.

Based on the initial screening sample results, the Project Team will determine if one round of step-out sample collection is required. Up to four additional step-out surface water samples will be collected 100 feet further downgradient or upgradient from where the PSL exceedances from the initial screening are unbounded, and step-out samples can be collected. Sections 11.4 and 11.5 will be used to guide the Project Team in this decision as well as sample location maps presented in a SAP addendum.

Groundwater Sampling

Groundwater samples will be collected from six locations during the initial screening sampling event using the DPT rig. Two samples will be collected from locations downgradient of the Building 1818 USTs, two samples will be collected from locations downgradient of the Building 1820 USTs, one sample will be collected downgradient of the Battery Disposal Area, and one sample will be collected from a location upgradient of the site. The planned groundwater sample locations are presented on Figure 17-3. 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.

Six groundwater samples (plus one duplicate sample for QC purposes) will be collected and analyzed for VOCs, PAHs, PCBs, and total (and dissolved, if groundwater is highly turbid) metals. 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 groundwater sample analytical results and other data, the project team may propose additional groundwater investigation activities. These activities may include the installation of permanent groundwater monitoring wells. The details regarding further groundwater investigation activities will be presented in an addendum to this SAP.

Additional Sampling for Delineation of COPCs and Risk Assessments

One additional round of step-out sampling will be performed if initial sampling (as described above) results indicate that target analyte levels exceed PSLs, and COPCs are identified. Additional samples may include surface soil, subsurface soil, sediment, surface water, and/or groundwater. Sample location maps will be presented in an addendum to this SAP.

Field Quality Control Samples

Field QC samples will be collected as part of the investigation, including field duplicates, trip blanks, and equipment rinsate blanks. Worksheet No. 20 presents the field QC sample summary. Also, additional sample volume will be collected as necessary for the laboratory QC of MS/MSD analyses (for VOCs, PAHs, PCBs, and TPH) and MS/laboratory duplicate analyses (for metals).

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
28SB01	28SS01SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB01SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB02	28SS02SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB02SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB03	28SS03SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB03SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				Metals	1	
28SB04	28SS04SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB04SOXXXX	Soil	>2 ³		VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB05	28SS05SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB05SOXXXX	Soil	>2 ³		VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB06	28SS06SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB07	28SS07SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB08	28SS08SO0002	Soil	0 - 2	VOCs	1	SOP-07, SOP-08,

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				PAHs	1	SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB09	28SS09SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB10	28SS10SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB11	28SS11SO0002 and 28SSFDXXXXXX-01 ²	Soil	0 – 2	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				PAHs	1 + 1 FD	
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
	28SB11SOXXXX and 28SBFDXXXXXX-01 ²	Soil	>2 ³	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				PAHs	1 + 1 FD	
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
28SB12	28SS12SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB12SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB13	28SS13SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08,

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				PAHs	1	SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB13SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB14	28SS14SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
	28SB14SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB15	28SS15SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
	28SB15SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB16	28SS16SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB17	28SS17SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08,

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				PAHs	1	SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB18	28SS18SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB19	28SS19SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB20	28SS20SO0002	Soil	0 – 2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB21	28SB21SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB22	28SB22SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB23	28SB23SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB24	28SB24SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB25	28SB25SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB26	28SB26SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB27	28SB27SOXXXX and 28SBFDXXXXXX-02 ²	Soil	>2 ³	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				PAHs	1 + 1 FD	
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
28SB28	28SB28SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				Metals	1	
28SB29	28SB29SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB30	28SB30SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB31	28SB31SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB32	28SB32SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB33	28SB33SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB34	28SB34SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB35	28SS35SO0002	Soil	0-2	VOCs	1 + 1 FD	SOP-07, SOP-08,

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
	and 28SSFXXXXXXXX-02 ²			PAHs	1 + 1 FD	SOP-11
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
	28SB35SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB36	28SS36SO0002	Soil	0-2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
	28SB36SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB37	28SS37SO0002	Soil	0-2	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
	28SB37SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
28SB38	28SB38SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
28SB39	28SB39SOXXXX and 28SBFDXXXXXX-03 ²	Soil	>2 ³	VOCs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				PAHs	1 + 1 FD	
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
28SB40	28SB40SOXXXX	Soil	>2 ³	VOCs	1	SOP-07, SOP-08, SOP-11
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB41	28SS41SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB42	28SS42SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB43	28SS43SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB44	28SS44SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB45	28SS45SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB46	28SS46SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB47	28SS47SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08,

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				PCBs	1	SOP-11
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB48	28SS48SO0002 and 28SSFDXXXXXX-03 ²	Soil	0-2	PAHs	1 + 1 FD	SOP-07, SOP-08, SOP-11
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
28SB49	28SS49SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB50	28SS50SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB51	28SS51SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB52	28SS52SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB53	28SS53SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB54	28SS54SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB55	28SS55SO0002 and 28SSFDXXXXXX-04 ²	Soil	0-2	pH	1 + 1 FD	SOP-07, SOP-08, SOP-11
				Lead	1 + 1 FD	
28SB56	28SS56SO0002	Soil	0-2	pH	1	SOP-07, SOP-08, SOP-11
				Lead	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
28SB57	28SS57SO0002	Soil	0-2	pH	1	SOP-07, SOP-08, SOP-11
				Lead	1	
28SB58	28SS58SO0002	Soil	0-2	pH	1	SOP-07, SOP-08, SOP-11
				Lead	1	
28SB59	28SB59SOXXXX and 28SBFDXXXXXX-04 ²	Soil	>2 ³	pH	1 + 1 FD	SOP-07, SOP-08, SOP-11
				Lead	1 + 1 FD	
28SB60	28SB60SOXXXX	Soil	>2 ³	pH	1	SOP-07, SOP-08, SOP-11
				Lead	1	
28SB61	28SB61SOXXXX	Soil	>2 ³	pH	1	SOP-07, SOP-08, SOP-11
				Lead	1	
28SB62	28SS62SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB63	28SS63SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB64	28SS64SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB65	28SS65SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
28SB66 to 28SBXX ⁴	28SS(66-XX)SO0002	Soil	0-2	PAHs	1	SOP-07, SOP-08, SOP-11
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
	28SB(66-XX)SOXXXX	Soil	>2 ³	VOCs	1	
				PAHs	1	
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
28SD01	28SD010006-01 And 28SDFDXXXXXX-01 ²	Sediment	0-0.5	PAHs	1 + 1 FD	SOP-09
				PCBs	1 + 1 FD	
				TPH (GRO/DRO/ERO)	1 + 1 FD	
				Metals	1 + 1 FD	
				TOC	1 + 1 FD	
28SD02	28SD020006	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD03	28SD030006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD04	28SD040006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD05	28SD050006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD06	28SD060006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD07	28SD070006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
28SD08	28SD080006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				Metals	1	
				TOC	1	
28SD09 to 28SDXX ⁷	28SD(09-XX)0006-01	Sediment	0-0.5	PAHs	1	SOP-09
				PCBs	1	
				TPH (GRO/DRO/ERO)	1	
				Metals	1	
				TOC	1	
				VOCs	1 + 1 FD	
28SW01	28SW01-01 and 28SWFDXXXXXX-01 ²	Surface Water	60% of surface water depth	PAHs	1 + 1 FD	SOP-18
				PCBs	1 + 1 FD	
				Metals	1 + 1 FD	
				VOCs	1	
28SW02	28SW02-01	Surface Water	60% of surface water depth	PAHs	1	SOP-18
				PCBs	1	
				Metals	1	
				VOCs	1	
28SW03 to 28SWXX ⁹	28SW(03-XX)-01	Surface Water	60% of surface water depth	PAHs	1	SOP-18
				PCBs	1	
				Metals	1	
				VOCs	1	
28TW01	28GW01-01 and 28GWFDXXXXXX-01 ²	Groundwater	Shallow (<20)	PAHs	1 + 1 FD	SOP-11
				PCBs	1 + 1 FD	
				Metals	1 + 1 FD	
				VOCs	1	
28TW02	28GW02-01	Groundwater	Shallow (<20)	PAHs	1	SOP-11
				PCBs	1	
				Metals	1	
				VOCs	1	
28TW03	28GW03-01	Groundwater	Shallow (<20)	PAHs	1	SOP-11
				PCBs	1	
				Metals	1	
				VOCs	1	
28TW04	28GW04-01	Groundwater	Shallow (<20)	PAHs	1	SOP-11
				PCBs	1	
				Metals	1	
				VOCs	1	
28TW05	28GW05-01	Groundwater	Shallow (<20)	PAHs	1	SOP-11

Sampling Location	ID Number	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹
				PCBs	1	
				Metals	1	
28TW06	28GW06-01	Groundwater	Shallow (<20)	VOCs	1	SOP-11
				PAHs	1	
				PCBs	1	
				Metals	1	

- 1 SOP or worksheet that describes the sample collection procedures (Worksheet No. 21).
- 2 Field duplicate locations may change in the field based on visual and olfactory observations and PID readings, and "XXXXXX" represents date collected.
- 3 Depth of the samples will be determined in the field.
- 4 Up to 12 additional step-out soil samples may be collected at the discretion of the Tetra Tech FOL during the initial sampling event.
- 5 Additional samples may be collected to delineate COPCs and will be presented in an addendum to this SAP.
- 6 Additional random background soil samples may be collected to support risk assessments. The sample quantities and sample locations will be determined using VSP and will be presented in an addendum to this SAP.
- 7 Up to four additional step-out sediment samples may be collected at the discretion of the Tetra Tech FOL during the initial screening sampling event.
- 8 Up to four additional step-out sediment samples may be collected to delineate COPCs.
- 9 The number of step-out surface water samples and their locations will be determined by the Project Team based on the initial screening sample event data.
- 10 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, surface water 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 grams (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, surface water and aqueous QC samples	PAHs	SW-846 3510C/3520/8270C-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	PAHs	SW-846 3546/8270C-Low, Empirical SOP-201/343	One 4-ounce (oz) glass jar with a Teflon-lined lid	30 g	Cool to ≤6 °C	14 days until extraction, 40 days to analysis
Groundwater, surface water and aqueous QC samples	PCBs	SW-846 3510C 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	PCBs	SW-846 3546/3550/8082A, Empirical SOP-211/343	One 4-oz glass jar with a Teflon-lined lid	30 g	Cool to ≤6 °C	14 days until extraction, 40 days to analysis
Groundwater, surface water and aqueous QC samples	Metals (and Dissolved Metals)	SW-846 3010A/6010C, Empirical SOP-100/105	One 500-mL plastic bottle	50 mL	Nitric acid (HNO ₃) to pH <2; Cool to ≤6 °C	180 days to analysis
Soil and sediment	Metals	SW-846 3050B/6010C, Empirical SOP-100/105	One 4-oz glass jar with a Teflon-lined lid	1 to 2 grams	Cool to ≤6 °C	180 days to analysis
Sediment	TOC	Lloyd Kahn, Empirical SOP-221	One 4-oz glass jar with a Teflon-lined lid	30 g	Cool to ≤6 °C	14 days to analysis

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)
Soil and Sediment	TPH (GRO/DRO/ERO)	SW-846 3550B/8015B, Empirical SOP-219/320	One 5-g Encore sampler or terracore (GRO) One 4-oz glass jar with a Teflon-lined lid (DRO/ERO)	5 g 25 g	Preserve in methanol in the field, freeze to < -10°C in the lab Cool to ≤6 °C	14 days until extraction, 28 days to analysis
Aqueous QC samples	TPH (GRO/DRO/ERO)	SW-846 3510C/8015B, Empirical SOP-219/322	One 1-L glass amber bottle	1,000 mL	HCl to pH<2; cool to ≤6 °C	7 days until extraction, 28 days to analysis
Soil and Sediment	TPH Fractionation	SW-846 3550B /8015B, NWTPH-HCID Mod, Test America TA-GS-0326	One 4-oz glass jar with a Teflon-lined lid	30 g	Cool to ≤6 °C	14 days until extraction, 28 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 VOA Trip Blanks	Total No. of Samples to Lab
Soil	VOCs	53	3	3/3	3	5	64
	PAHs	71	4	4/4	4	NA	79
	PCBs	67	4	4/4	4	NA	75
	TPH	71	4	4/4	4	NA	79
	Metals	71	4	4/4	4	NA	79
	pH	7	1	1/1	0	NA	8
	Lead	7	1	1/1	1	NA	9
Sediment	PAHs	8	1	1/1	0	NA	9
	PCBs	8	1	1/1	0	NA	9
	TPH	8	1	1/1	0	NA	9
	Metals	8	1	1/1	0	NA	9
	TOC	8	1	1/1	0	NA	9
Surface Water	VOCs	2	0	0/0	0	1	3
	PAHs	2	0	0/0	0	0	2
	PCBs	2	0	0/0	0	0	2
	Total Metals	2	0	0/0	0	0	2
	Dissolved Metals	0 minimum, 2 maximum	0	0/0	1 ²	0	3
Groundwater	VOCs	6	1	1/1	1	1	9
	PAHs	6	1	1/1	1	NA	8
	PCBs	6	1	1/1	1	NA	8
	Total Metals	6	1	1/1	0	NA	7
	Dissolved Metals	0 minimum, 6 maximum	1	1/1	1 ²	NA	8

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/MSDs 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, 01/11, Rev. 0	Tetra Tech	GPS unit	Y (project-specific)	Contained in Appendix A
SOP-02	Sample Labeling, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-03	Sample Identification Nomenclature, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-04	Sample Custody and Documentation of Field Activities, 01/11, Rev. 0	Tetra Tech	Field logbook, sample log sheets, boring logs	Y (project-specific)	Contained in Appendix A
SOP-05	Sample Preservation, Packaging, and Shipping, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-06	Decontamination of Field Sampling Equipment, 01/11, 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, 01/11, Rev. 0	Tetra Tech	Stainless steel auger bucket, extension rods, and T-handle	Y (project-specific)	Contained in Appendix A
SOP-08	Soil Sample Logging, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-09	Sediment Sampling, 01/11, Rev. 0	Tetra Tech	Stainless steel or disposable trowels	Y (project-specific)	Contained in Appendix A
SOP-10	Management of Investigation-Derived Waste, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-11	Subsurface Soil and Groundwater Sampling Using Direct-Push Technology, 01/11, Rev. 0	Tetra Tech	DPT Rig	Y (project-specific)	Contained in Appendix A
SOP-13	Monitoring Well Development, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-14	Measurement of Water Levels, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-15	Low-Flow Well Purging and Stabilization, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-16	Monitoring Well Sampling, 01/11, Rev. 0	Tetra Tech	NA	Y (project-specific)	Contained in Appendix A
SOP-17	Calibration and Care of Water Quality Meters, 01/11, Rev. 0	Tetra Tech	Multi-parameter water quality meter,	Y (project-specific)	Contained in Appendix A
SOP-18	Surface Water Sampling, 01/11, Rev. 0	Tetra Tech	Multi-parameter water quality meter,	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	SOP-17, 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	SOP-17, Manufacturer's Guidance Manual	To be used to determine the need to collect Dissolved Metals samples (if >5 NTUs).
Electric Water Level Indicator	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or replacement	Tetra Tech FOL or designee	SOP-14, Manufacturer's Guidance Manual	None
Photo Ionization Detector	Visual Inspection Calibration/Verification	Daily Beginning and end of day	Manufacturer's Guidance	Operator correction or replacement	Tetra Tech FOL or designee	SOP-07, SOP-09, SOP-11, SOP-18, 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, 09/01/10)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/ Metals Digestion	NA/Preparation	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 19 th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil, sediment, groundwater, surface water, and aqueous QC samples/ Metals	ICP-AES	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, surface water, and aqueous QC samples/ Low Level PAHs	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, surface water, 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, surface water, and aqueous QC samples/ PCBs	GC/ECD	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-219	Gas Chromatography/ Flame Ionization Detector (GC/FID) Nonhalogenated Volatile Organics and Total Petroleum Hydrocarbons (TPH) by Method 8015B/8015C/TN EPH/GRO (Revision 14, 12/01/10)	Definitive	Soil and sediment/ TPH GRO/DRO/ERO	GC/FID	Empirical	Y (IDEM-Specific Ranges)
Empirical SOP-221	TOC SM5310C, SW846 Method 9060/9060A and Lloyd Kahn Method (Revision 9, 07/12/10)	Definitive	Soil, sediment, groundwater/ 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, 09/07/10)	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, surface water, and aqueous QC samples/ PAHs Extraction	NA/ Extraction	Empirical	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, surface water, and aqueous QC samples/ PCBs Extraction	NA/ Extraction	Empirical	N
Empirical SOP-320	Total Petroleum Hydrocarbons (TPH) Non-Aqueous Matrix (Low Level) by USEPA SW846 Method 8015B, Large Sonication Horn (Revision 10, 09/09/10)	Definitive	Soil and sediment/ TPH DRO/ERO Extraction	NA/ Extraction	Empirical	N
Empirical SOP-322	Total Petroleum Hydrocarbons (TPH) Aqueous Matrix by USEPA SW846 Method 8015B (Revision 10, 09/09/10)	Definitive	Aqueous QC samples/ TPH DRO/ERO 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/ PAHs 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)	N/A	Log-in	NA/ Log-in	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-QS-14	Analytical Laboratory Waste Disposal (Revision 6, 08/31/10)	N/A	Disposal	NA/ Disposal	Empirical	N
Test America TA-GS-0326	Hydrocarbon Identification Method [Method NWTPH-HCID Mod] SOP for TPH Fractionation Sample Processing (Revision 8, March 2010)	Definitive	Soil and Sediment/ TPH Fractionation	GC/FID	Test America	N

Copies of laboratory SOPs listed are included in Appendix B.

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	Bromofluorobenzene (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 (SW-846 8260B; 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 $\leq 30\%$; and %RSD for each target analyte must be $\leq 15\%$, 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 ± 0.06 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 $\leq 20\%$.	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 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 (SW-846 8270C Low Level Full Scan; Section 7.3.1; Table 3).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst/ Supervisor	Empirical SOP-201	
	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 $\leq 30\%$; and %RSD for each target analyte must be $\leq 15\%$, 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 80-120% of the true value. SPCC RFs must be ≥ 0.050 ; CCCs must be $\leq 20\%D$.	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 ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor		
	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 $\leq 20\%D$.	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		Empirical SOP-201

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 $\leq 20\%$ 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.	Evaluate, repeat, if still failing, recalibrate.	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 $\leq 20\%$.	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-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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
ICP-AES Metals (continued)	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	Empirical SOP-105
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	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 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.	ICS A recoveries must be within the absolute value of the LOD; and ICS B recoveries must be within 80-120 %R of true value.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	
GC/FID TPH - GRO	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, major instrument change, when CCV does not meet criteria.	The %RSD for each target analyte must be $\leq 20\%$, 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	Empirical SOP-219
	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.	Evaluate, repeat, if still failing, recalibrate.	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 $\leq 20\%$.	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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/FID TPH – DRO/ERO	ICAL – A minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, major instrument change, when CCV does not meet criteria.	The %RSD for each target analyte must be $\leq 20\%$, 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	Empirical SOP-219
	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.	Evaluate, repeat, if still failing, recalibrate.	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 $\leq 20\%$.	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	
GC/FID/PID and GC/FID TPH Fractionation	ICAL – a minimum of a 5-point calibration is prepared for all target analytes and hydrocarbon ranges	Upon instrument receipt, major instrument change, when CCV does not meet criteria.	The %RSD for each target analyte must be $\leq 20\%$, or r must be ≥ 0.995 ; or r^2 must be ≥ 0.995 (minimum of 6 points required for second order).	One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision.	Analyst/ Supervisor	Test America TA-GS-0326
	ICV	Once after each ICAL prior to sample analysis.	The %R of all target analytes must be within 80-120% of true value. The %R of all hydrocarbon ranges must be within 80-120% of true value for DRO/ERO/EPH and within 70-130% of true value for GRO/PH.	Evaluate, repeat, if still failing, recalibrate.	Analyst/ Supervisor	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %D of all target analytes and hydrocarbon ranges must be $\leq 20\%$.	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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA	SOP Reference
GC/FID/PID and GC/FID TPH Fractionation(continued)	CCB	After the initial CCV, after every 10 samples following the CCV, and at the end of the sequence following the CCV.	No target analytes detected > ½ LOQ.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	Test America TA-GS-0326
	RT Reference Standard	RT window width is set at ± 3 standard deviations from the mean RT, or a minimum of 0.03 minutes.	Toluene must be resolved from the solvent peak.	If the acceptance criteria are not met, check instrument conditions and calibration materials, correct as necessary and repeat analysis of the reference standard before proceeding with the analysis of samples.	Analyst/ Supervisor	
TOC Analyzer	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/ Supervisor	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/ Supervisor	
	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/ Supervisor	

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 ⁽¹⁾	SOP Reference ⁽²⁾
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	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-202
GC/MS	Check pressure and gas supply daily. Change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in Equipment Maintenance SOP.	Low Level PAHs	Ion source, injector liner, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-201
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.	PCBs	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-211
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	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-105
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TPH/GRO	Injector liner, septa, column, column flow.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-219

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person ⁽¹⁾	SOP Reference ⁽²⁾
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	TPH/DRO/ ERO	Injector liner, septa, column, column flow.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-219
GC/FID/PID and GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed.	TPH Fractionation	Injector liner, septa, column, column flow.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Test America TA-GS-0326
TOC Analyzer	Replace sample tubing, clean sample boat, replace syringe.	TOC	Tubing, sample boat, syringe	As needed.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-221

Notes:

- 1 Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #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 Test America
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Empirical and Test America
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory, TPH Fractionation Preparation Laboratory / Empirical and Test America
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory, TPH Fractionation Laboratory/ Empirical and Test America
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 Test America

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. The need for TPH Fractionation analysis by Test America will be based on PSL exceedences of TPH GRO/DRO/ERO Ranges as determined by Empirical; a note will be included on Chain-of-custody for expedited analysis of TPH and immediate forwarding of samples that exceed a TPH Range PSL to Test America. 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, which are provided in Appendix B.

Laboratory Sample Custody Procedures

Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (Empirical SOP 404 [Sample Receiving and Sample Storage] SOP), which is 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 type (sample medium and/or QC sample designation), sample location, and sample depth. SOP-03 addresses sample identification nomenclature (Appendix A).

Site Identifier:

28 = SWMU 28

Sample Medium:

SS = Surface Soil

SB = Subsurface Soil

SD = Sediment

SW = Surface Water

GW = Groundwater

QA/QC Sample Designation:

FD = Field Duplicate

TB = Trip Blank

RB = Equipment Rinsate Blank

Sample Location:

Each sampling and soil boring location will be assigned a specific two digit number, as identified in Worksheet No. 18. Sample locations within a given boring will be assigned a two digit consecutive number in the order of collection.

For soil samples, the soil sample depth will be indicated by a four digit number. The first two digits will represent the upper limit of the sample depth interval (rounded to the nearest foot) and the bottom two digits will represent the lower limit of the depth interval.

QC Sample Number:

All QC samples will be assigned a sequential sample number per day. For example, the first trip blank shipped each day will be assigned the tracking number 11TBMMDDYY-01.

The FD will be given the same type of sample designation as the samples so that it will be “blind” to the laboratory. The sampling time recorded on the chain-of-custody form, labels and tags for the duplicate samples will be 0000. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

All pertinent information regarding sample identification will be recorded in the field logbooks and on sample log sheets where appropriate.

SAP WORKSHEET NO. 28 -- LABORATORY QC SAMPLES TABLE

(UFP-QAPP Manual Section 3.4)

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 $\leq \frac{1}{2}$ 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 Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM. RPD must be $\leq 30\%$ (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, Supervisor, 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 QSM Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be $\leq 30\%$.	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 Standard (IS)	Every field sample, standard, and QC sample - three per sample- Fluorobenzene Chlorobenzene-d ₅ 1,4-dichlorobezene-d ₄	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-d ₄ Toluene-d ₈ 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

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 $\leq \frac{1}{2}$ 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 Low Level PAHs criteria 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 except Low Level PAHs criteria that are provided in Appendix B. The RPD between MS and MSD should be $\leq 30\%$.	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	Low Level PAHs					
Analytical Method / SOP Reference	SW-846 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-d ₄ Naphthalene-d ₈ Acenaphthene-d ₁₀ Phenanthrene-d ₁₀ Chrysene-d ₁₂ Perylene-d ₁₂	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-d ₆ Nitrobenzene-d ₅ 2-Fluorobiphenyl 2,4,6-Tribromophenol Terphenyl-d ₁₄	%Rs must meet the Low Level PAHs criteria 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	PCBs					
Analytical Method / SOP Reference	SW-846 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 $\leq \frac{1}{2}$ 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 $< \text{LOQ}$ or $> 10\text{X}$ the blank concentration. Otherwise, re-prepare a blank and samples $> \text{LOQ}$ and $< 10\text{X}$ 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. 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 $< \text{LOQ}$, 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 $\leq 30\%$.	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	PCBs					
Analytical Method / SOP Reference	SW-846 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					
Analytical Method / SOP Reference	SW-846 6010C Empirical SOP-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 $\leq \frac{1}{2}$ 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 $\leq 20\%$ 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	One per preparatory batch with sample concentration(s) >50x LOD.	The 5-fold dilution result must agree within $\pm 10\%D$ 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					
Analytical Method / SOP Reference	SW-846 6010C Empirical SOP-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	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, and Aqueous QC Samples					
Analytical Group	TPH GRO					
Analytical Method / SOP Reference	SW-846 8015B Empirical SOP-219					
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 $\leq \frac{1}{2}$ LOQ.	If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.	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 50-150% of true value.	If LCS acceptance limits are not met, the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the associated sample must be re-extracted and reanalyzed.	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 should be within 50-150% of true value (if sample is < 4x spike added). The RPD between MS and MSD should be $\leq 30\%$.	CA will not be taken for samples when %Rs 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
Surrogate	All field and QC samples - one per sample BFB.	The %R of the surrogate must fall within 50-150% as established by WA DOE.	If surrogate %Rs are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. If the surrogate %R is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative.	Analyst, Supervisor, Data Validator	Accuracy	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, and Aqueous QC Samples					
Analytical Group	TPH DRO/ERO					
Analytical Method / SOP Reference	SW-846 Method 8015B Empirical SOP-219					
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 $\leq \frac{1}{2}$ LOQ.	If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.	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 50-150% of true value.	If LCS acceptance limits are not met, the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the associated sample must be re-extracted and reanalyzed.	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 should be within 50-15% of true value (if sample is < 4x spike added). The RPD between MS and MSD should be $\leq 30\%$.	CA will not be taken for samples when %Rs 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
Surrogate	All field and QC samples - one per sample o-Terphenyl.	The %R of the surrogate must fall within 50-150% as established by WA DOE.	If surrogate %R is outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. If the surrogate %R is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative.	Analyst, Supervisor, Data Validator	Accuracy	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 and Sediment Samples					
Analytical Group	TPH Fractionation					
Analytical Method / SOP Reference	NWTPH-HCID Mod Test America TA-GS-0326, TA-GS-0356, TA-GV-0390					
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 $\leq \frac{1}{2}$ LOQ.	If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.	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 70-130% of true value for all ranges, except the C ₈ -C ₁₀ aliphatic and C ₈ -C ₁₀ aromatics, which should be within an advisory limit of 50-150%.	If LCS acceptance limits are not met, the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the associated sample must be re-extracted and reanalyzed.	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 should be within 70-130% of true value (if sample is < 4x spike added) for all ranges, except the C ₈ -C ₁₀ aliphatic and C ₈ -C ₁₀ aromatics, which should be within an advisory limit of 50-150%. The RPD between MS and MSD should be $\leq 25\%$.	CA will not be taken for samples when %Rs 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
IS	Every field sample, standard, and QC sample - one per sample 1,2,3-Trifluorobenzene.	RT 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 – one per sample (GRO/VPH) BFB three per sample (DRO/ERO/EPH) o-Terphenyl p-Terphenyl-d14 1-Chlorooctadecane.	The %R of the surrogates must fall within 60-140%.	If surrogate %Rs are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. If the surrogate %R is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Fractionation Surrogate Check Standard	All field and QC samples – one per sample (DRO/ERO/EPH Only)	%R should be <10% for both the aliphatic fraction and the aromatic fraction.	None. This is used to monitor the fractionation process of the samples and batch QC.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

Matrix	Soil and Sediment Samples					
Analytical Group	TPH Fractionation					
Analytical Method / SOP Reference	NWTPH-HCID Mod Test America TA-GS-0326, TA-GS-0356, TA-GV-0390					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
(DRO/ERO/EPH Only)	5,6,7,8-Tetrahydrol-1-naphthol					
Fractionation Check Standard (DRO/ERO/EPH Only)	One per preparatory batch of 20 or fewer samples of similar matrix.	%R must be within 70-130% of true value for all hydrocarbon ranges.	Failure to meet this criterion indicates the need for review or re-development of the fractionation procedure. The aliphatic/aromatic fractionation is a critical component of this analytical method. Do not report analytical data for samples associated with a failed fractionation check.	Analyst, Supervisor, Data Validator	Accuracy	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 $\leq \frac{1}{2}$ LOQ.	Correct problem, re-prepare and reanalyze along with associated 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 $\leq 20\%$.	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
<p>Field Documents Field Logbook (and sampling notes) Field Sample 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</p>	<p>Field documents will be maintained in the project file located in the Tetra Tech Cincinnati, Ohio office.</p>
<p>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</p>	<p>Laboratory documents will be included in the hardcopy and 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.</p> <p>Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.</p>
<p>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)</p>	<p>All assessment documents will be maintained in the Tetra Tech Cincinnati, Ohio project file.</p>
<p>Reports SWMU 28 RFI Report.</p>	<p>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.</p>

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 #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
	PAHs		SW-846 8270C-Low			
	PCBs		SW-846 8082A			
	Metals		SW-846 6010C			
	TPH (GRO/DRO/ERO)		SW-846 Method 8015B			
Soil and sediment	TPH Fractionation	See Worksheet #18	NWTPH-HCID Mod WADOE Method	21 calendar days	Curtis Armstrong curtis.armstrong@testamericainc.com Test America Inc. 5755 8 th Street East Tacoma, WA 98424 (253) 922-2310	NA
Sediment	TOC	See Worksheet #18	Lloyd Kahn	21 calendar days	Kim Kostzer kkostzer@empirlabs.com Empirical 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	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 two years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical and Test America	Laboratory QAM or Laboratory Manager, Empirical and Test America	Laboratory QAM or Laboratory Manager, Empirical and Test America

1 Empirical and Test America are DoD ELAP accredited by a recognized Accrediting Body. The DoD ELAP accreditation letters are 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	Marcia McGinnity, Laboratory QAM, Empirical Dave Wunderlich, Laboratory QAM, Test America	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 Test America	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 Test America 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 Test America 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 Test America
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 Test America
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 stored 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	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available.	
		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.	
		Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36.	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Ensure that the LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		Discuss the impact of 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.	
		Summarize deviations from methods, procedures, or contracts in the Data Validation Report. If possible 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.	
		Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	

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, Low Level PAHs, and PCBs	Data validation will be performed using criteria for SW-846 Methods 8260B, 8270C-Low, 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	Data validation will be performed using criteria for SW-846 Method 6010C 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	Sediment	TOC	Limited data validation* will be performed using criteria for the Lloyd Kahn Method listed in Worksheets Nos.12, 15, 24, and 28 and the current DoD QSM.	Data Validation Specialist, Tetra Tech
Iia and Iib	Soil and Sediment	TPH (GRO/DRO/ERO) and TPH Fractionation	Data validation will be performed using criteria for SW-846 Method 8015B listed in Worksheets Nos. 12, 15, 24, and 28 and the current DoD QSM 4.1. If not included in the aforementioned, then the logic outlined in the "USEPA 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

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 that 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.

REFERENCES

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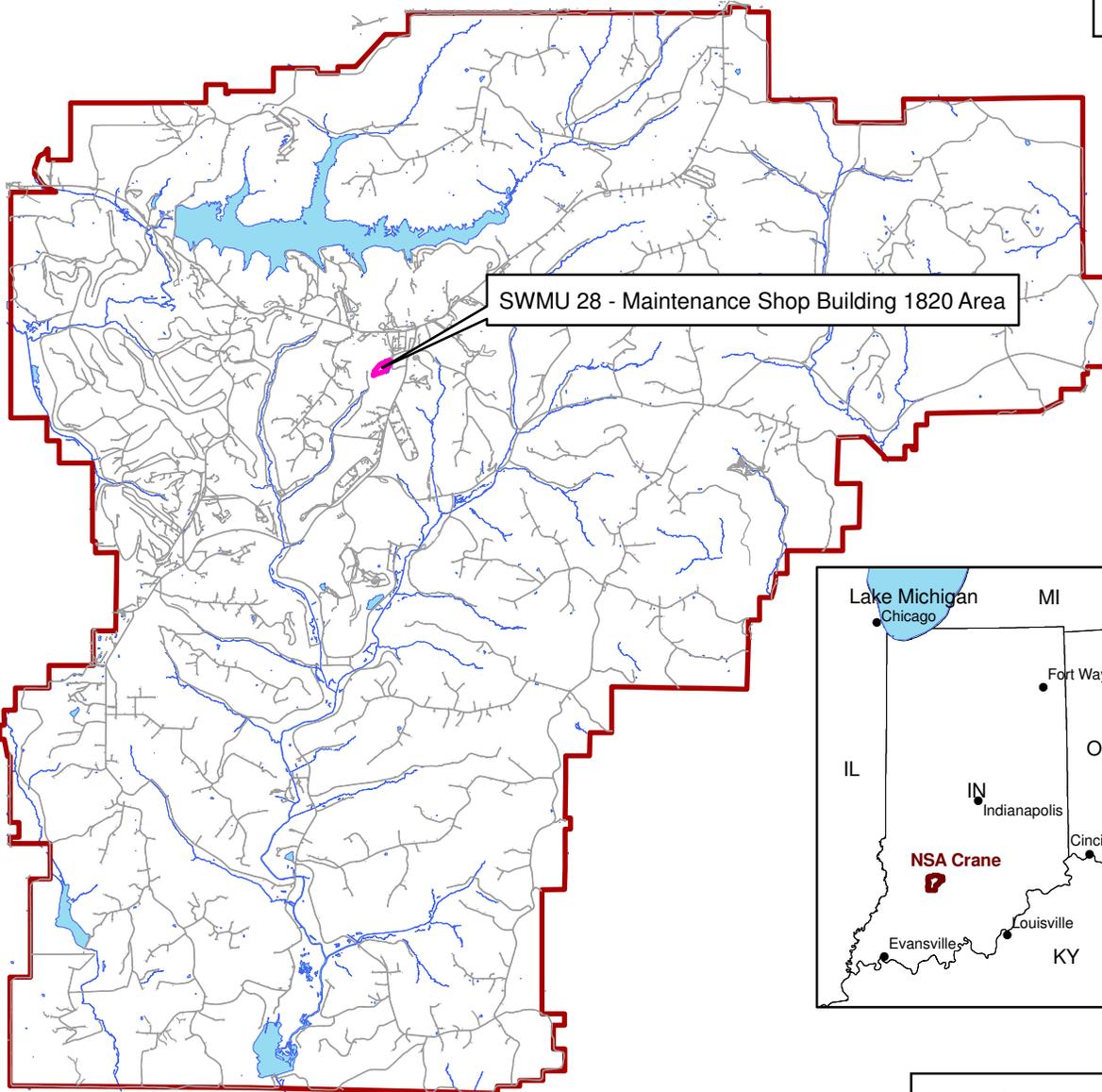
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FIGURES

- 10-1 Base and Site Location Map
- 10-2 Site Layout – 2010 Aerial
- 10-3 Site Plan
- 10-4 Historical Site Layout – 1952 Aerial Photo
- 10-5 Conceptual Site Model
- 10-6 Human Conceptual Exposure Model Diagram
- 10-7 Ecological Conceptual Exposure Model Diagram
- 11-1 Sampling Investigative Areas
- 17-1 Sampling Locations - Former Building 1818 Area
- 17-2 Sampling Locations - Former Building 1820 Area
- 17-3 Sampling Locations – Groundwater, Adjacent Streams and Outside Truck Wash Rack



SWMU 28 - Maintenance Shop Building 1820 Area



Legend

- Road
- ◻ SWMU 28
- ▭ Base Boundary
- Water



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



BASE AND SITE LOCATION MAP
 SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
 NSA CRANE
 CRANE, INDIANA

CONTRACT NUMBER CTO F273	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 10-1	REV 0



Legend

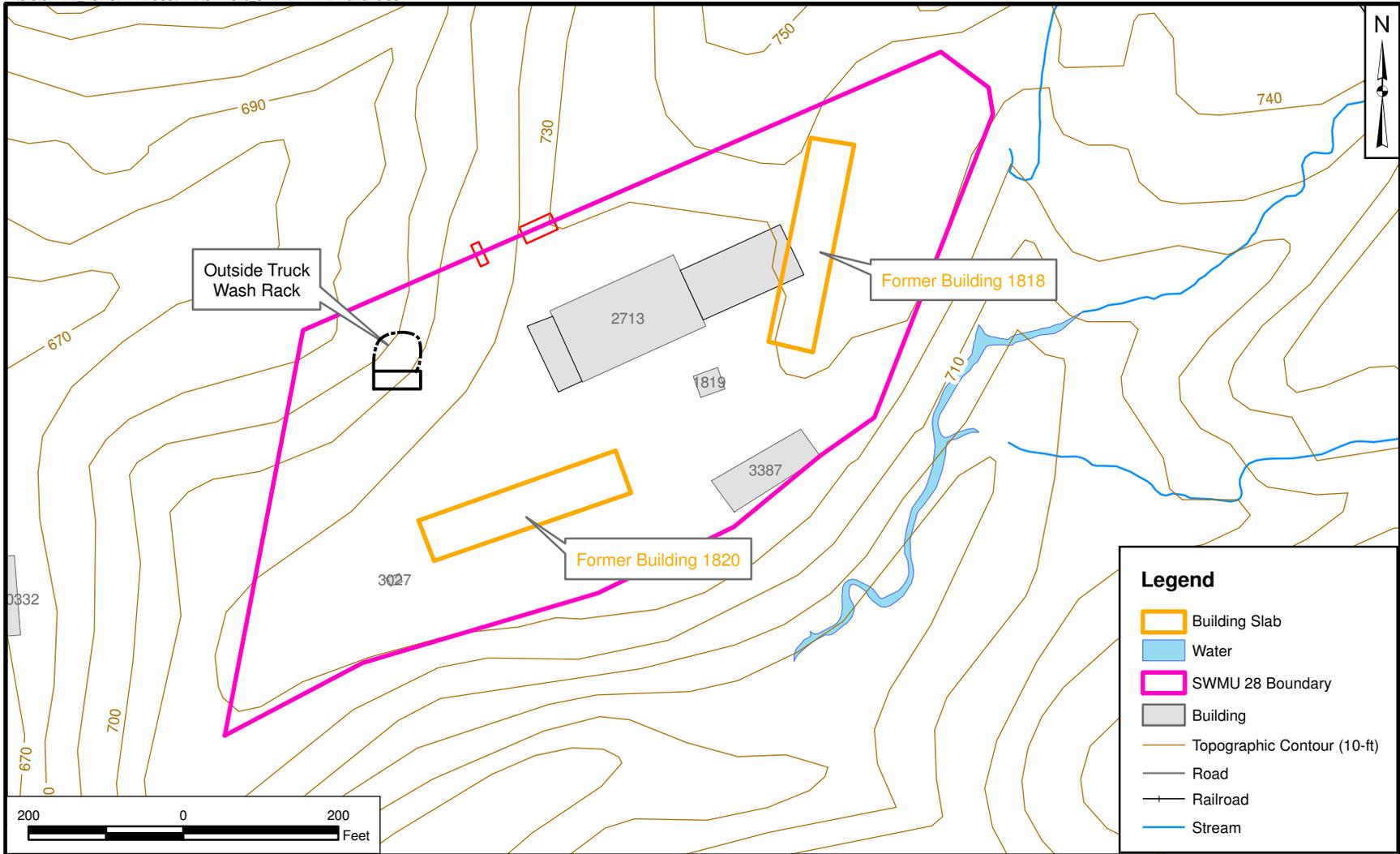
- SWMU 28 Boundary
- Building Slabs
- Railroad

DRAWN BY	DATE
S. STROZ	08/16/10
CHECKED BY	DATE
T. KLIMEK	10/28/10
REVISED BY	DATE
SCALE AS NOTED	



SITE LAYOUT - 2010 AERIAL
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER F273	
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APPROVED BY	DATE
FIGURE NO.	REV
10-2	0



Legend

- Building Slab
- Water
- SWMU 28 Boundary
- Building
- Topographic Contour (10-ft)
- Road
- Railroad
- Stream



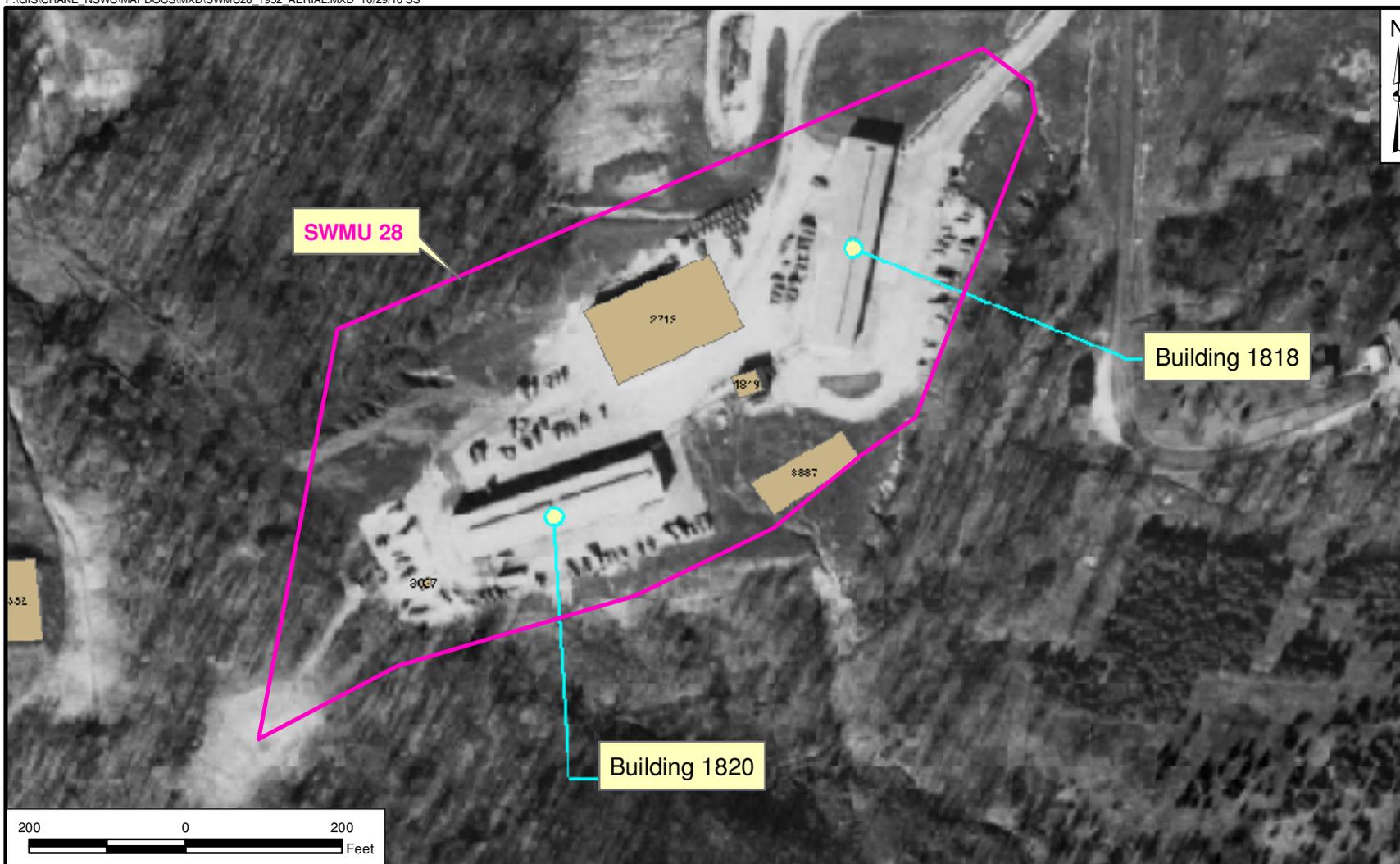
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CHECKED BY	DATE
T. KLIMEK	10/28/10
REVISED BY	DATE



SCALE
AS NOTED

SITE PLAN
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA

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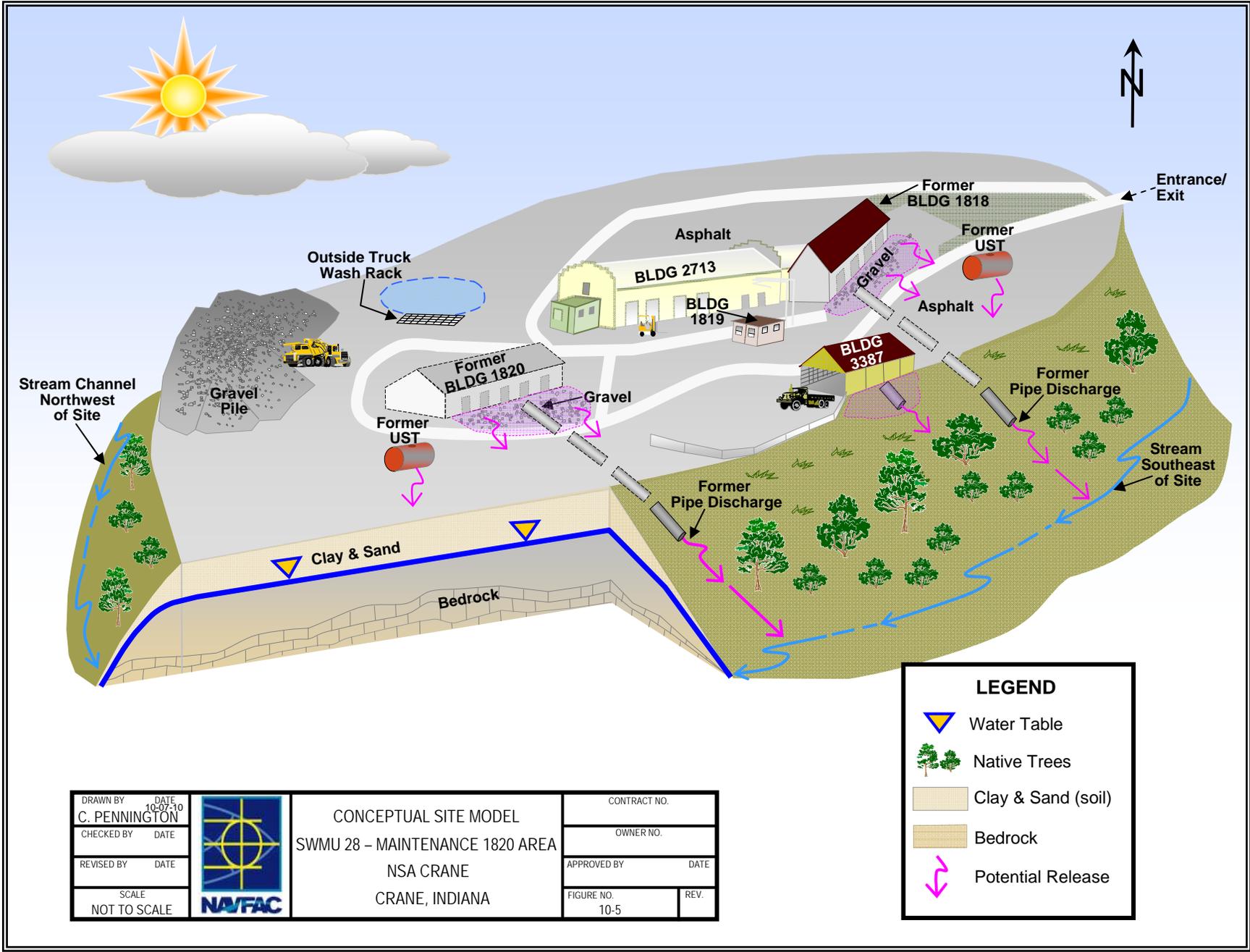
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S. STROZ	09/03/10
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T. KLIMEK	10/29/10
REVISED BY	DATE



SCALE
AS NOTED

HISTORICAL SITE LAYOUT - 1952 AERIAL PHOTO
 SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
 NSA CRANE
 CRANE, INDIANA

CONTRACT NUMBER F273	
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C. PENNINGTON	10-07-10
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NOT TO SCALE	

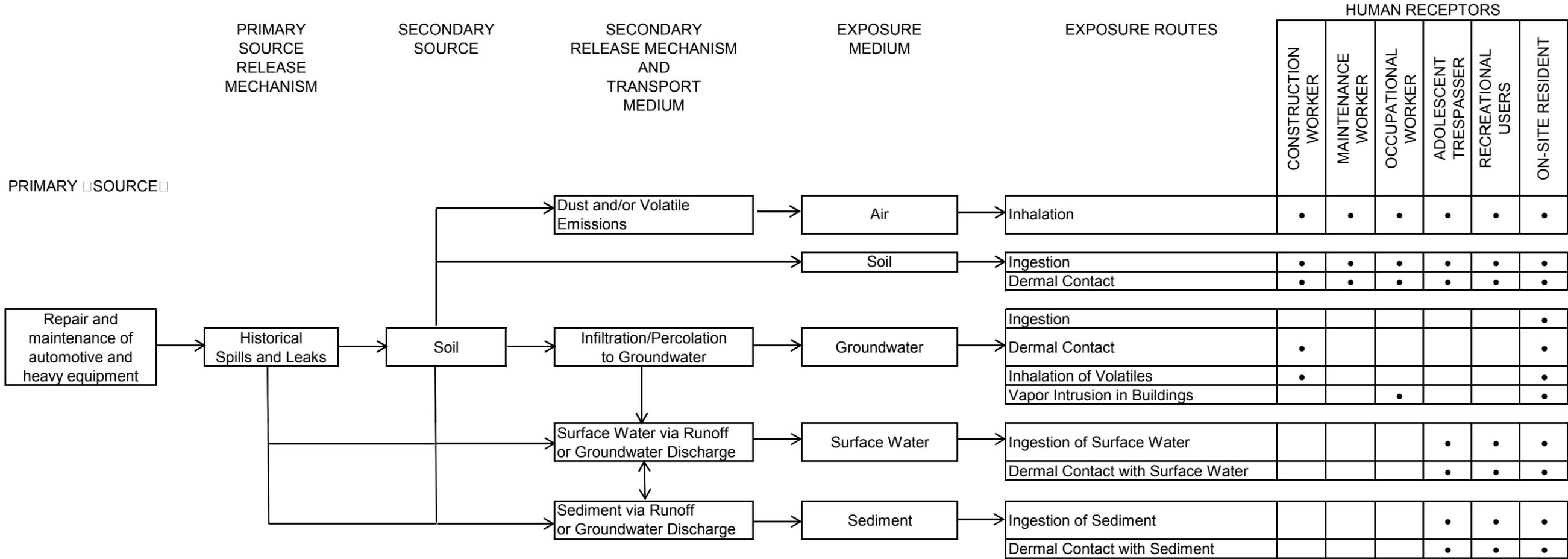


CONCEPTUAL SITE MODEL
 SWMU 28 - MAINTENANCE 1820 AREA
 NSA CRANE
 CRANE, INDIANA

CONTRACT NO.	
OWNER NO.	
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FIGURE NO.	REV.
10-5	

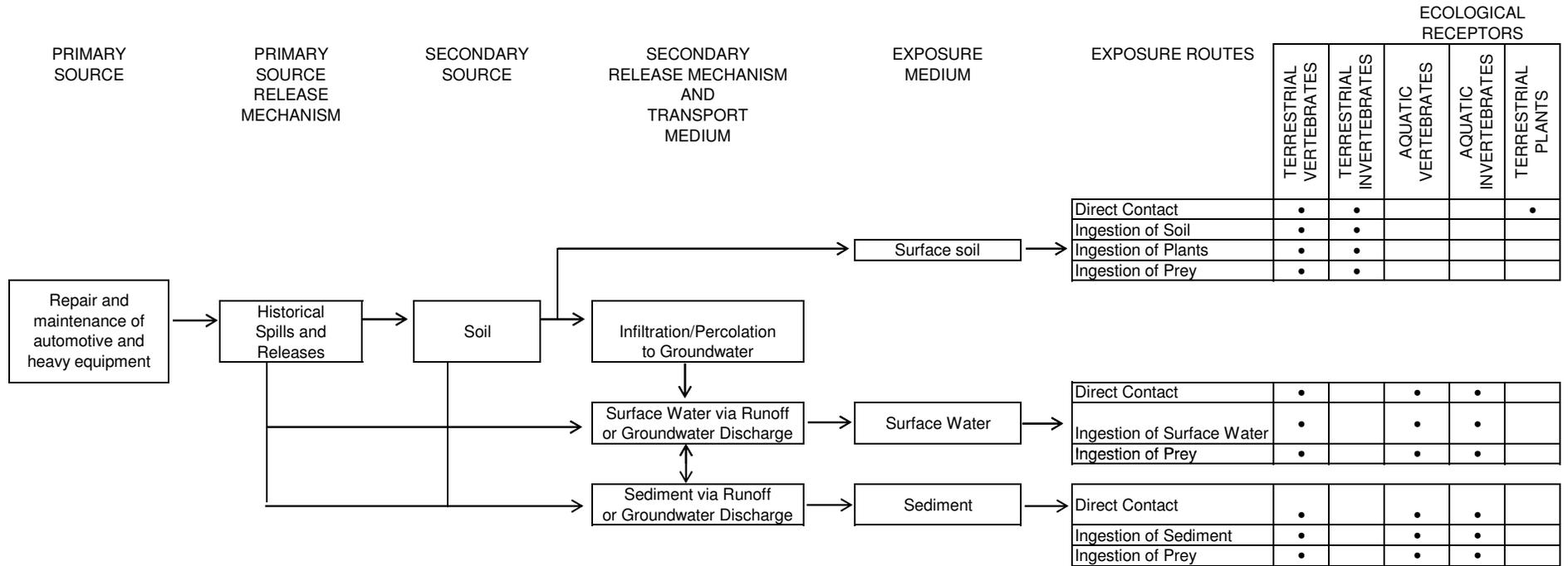
LEGEND	
	Water Table
	Native Trees
	Clay & Sand (soil)
	Bedrock
	Potential Release

**FIGURE 10-6
HUMAN CONCEPTUAL EXPOSURE MODEL DIAGRAM
SWMU 28
NSA CRANE, INDIANA**

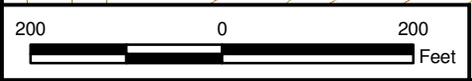
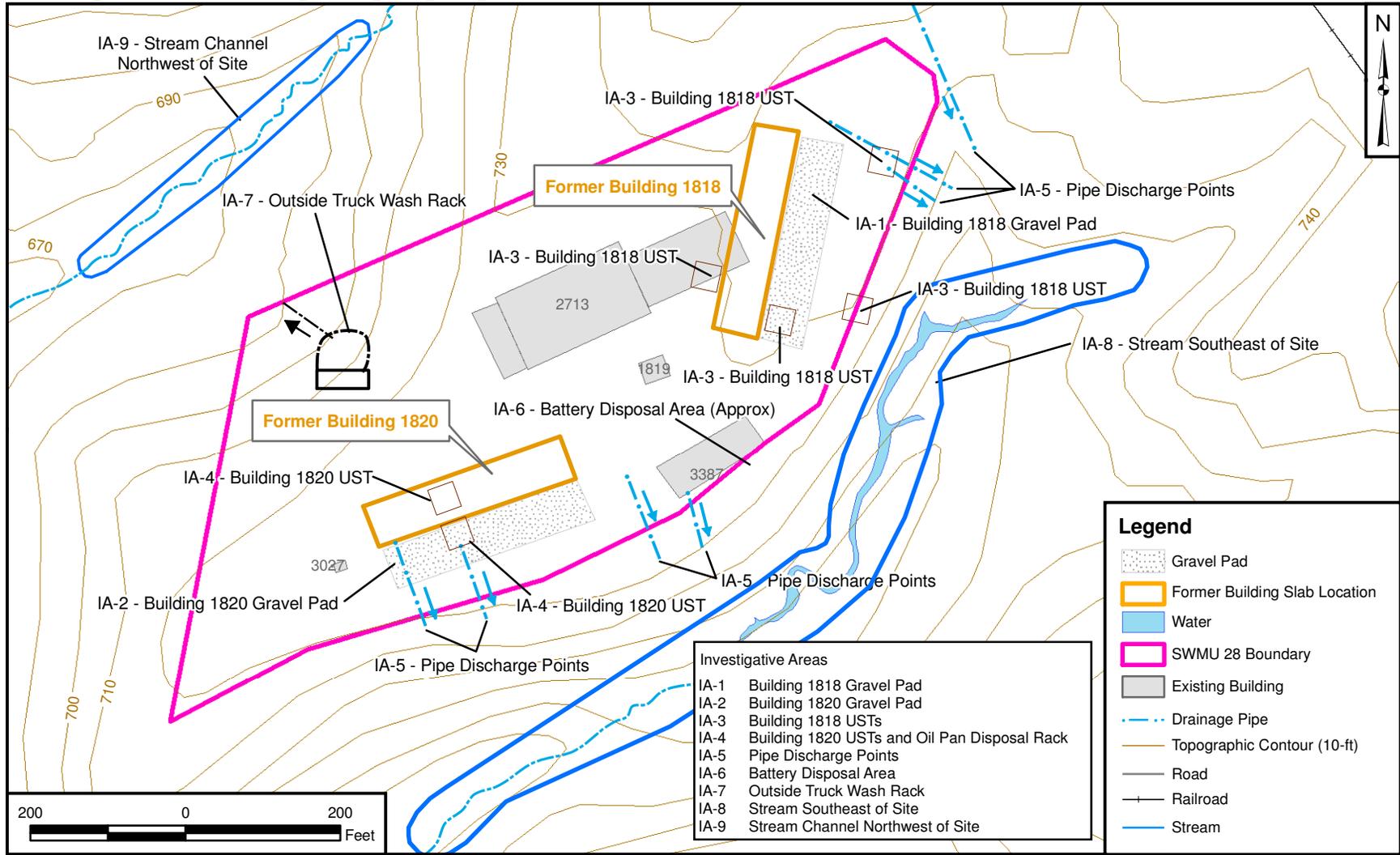


• Indicates receptor for evaluation

**FIGURE 10-7
 ECOLOGICAL CONCEPTUAL EXPOSURE MODEL DIAGRAM
 SWMU 28
 NSA CRANE, INDIANA**



• Indicates receptor for evaluation

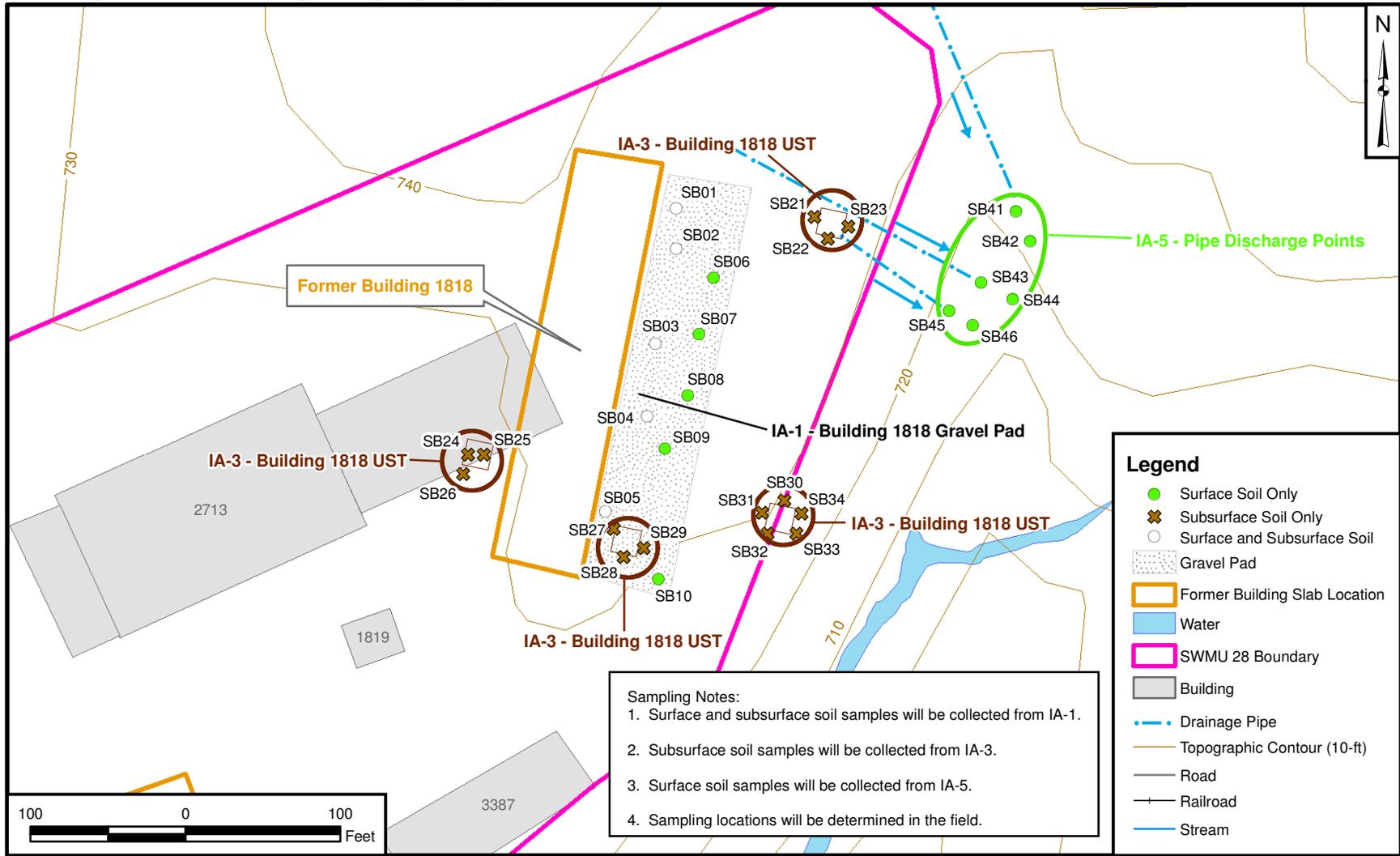


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CHECKED BY T. KLIMEK	DATE 4/7/11
REVISED BY K. MOORE	DATE 4/7/11
SCALE AS NOTED	



SAMPLING INVESTIGATIVE AREAS
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER F273	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 11-1	REV 0



Sampling Notes:
 1. Surface and subsurface soil samples will be collected from IA-1.
 2. Subsurface soil samples will be collected from IA-3.
 3. Surface soil samples will be collected from IA-5.
 4. Sampling locations will be determined in the field.

Legend

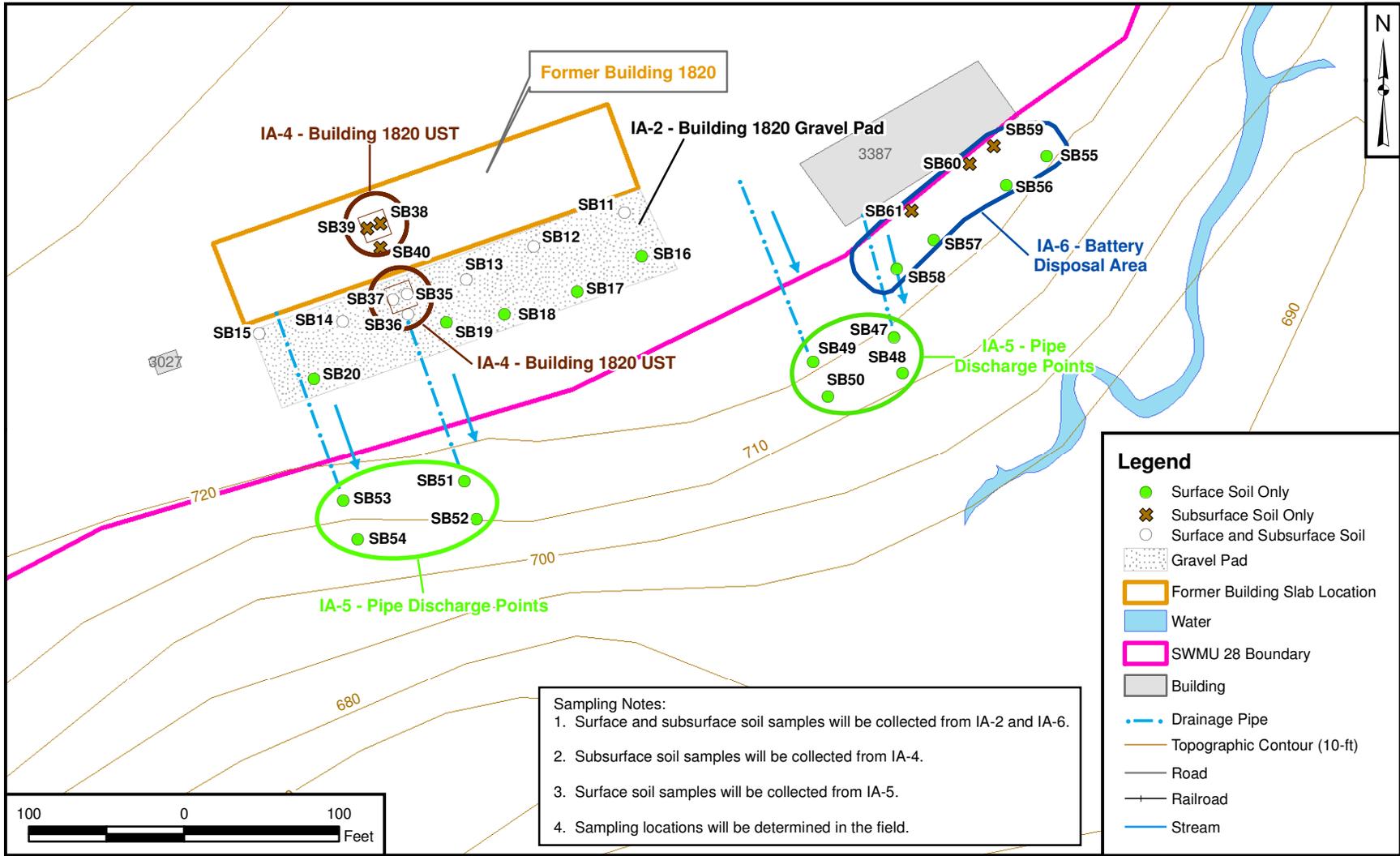
- Surface Soil Only
- ✕ Subsurface Soil Only
- Surface and Subsurface Soil
- ▨ Gravel Pad
- ▭ Former Building Slab Location
- Water
- ▭ SWMU 28 Boundary
- ▭ Building
- Drainage Pipe
- Topographic Contour (10-ft)
- Road
- Railroad
- Stream

DRAWN BY S. STROZ	DATE 10/06/10
CHECKED BY T. KLIMEK	DATE 4/7/11
REVISED BY K. MOORE	DATE 4/7/11
SCALE AS NOTED	



SAMPLING LOCATIONS - FORMER BUILDING 1818 AREA
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER F273	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 17-1	REV 0



Sampling Notes:
 1. Surface and subsurface soil samples will be collected from IA-2 and IA-6.
 2. Subsurface soil samples will be collected from IA-4.
 3. Surface soil samples will be collected from IA-5.
 4. Sampling locations will be determined in the field.

Legend

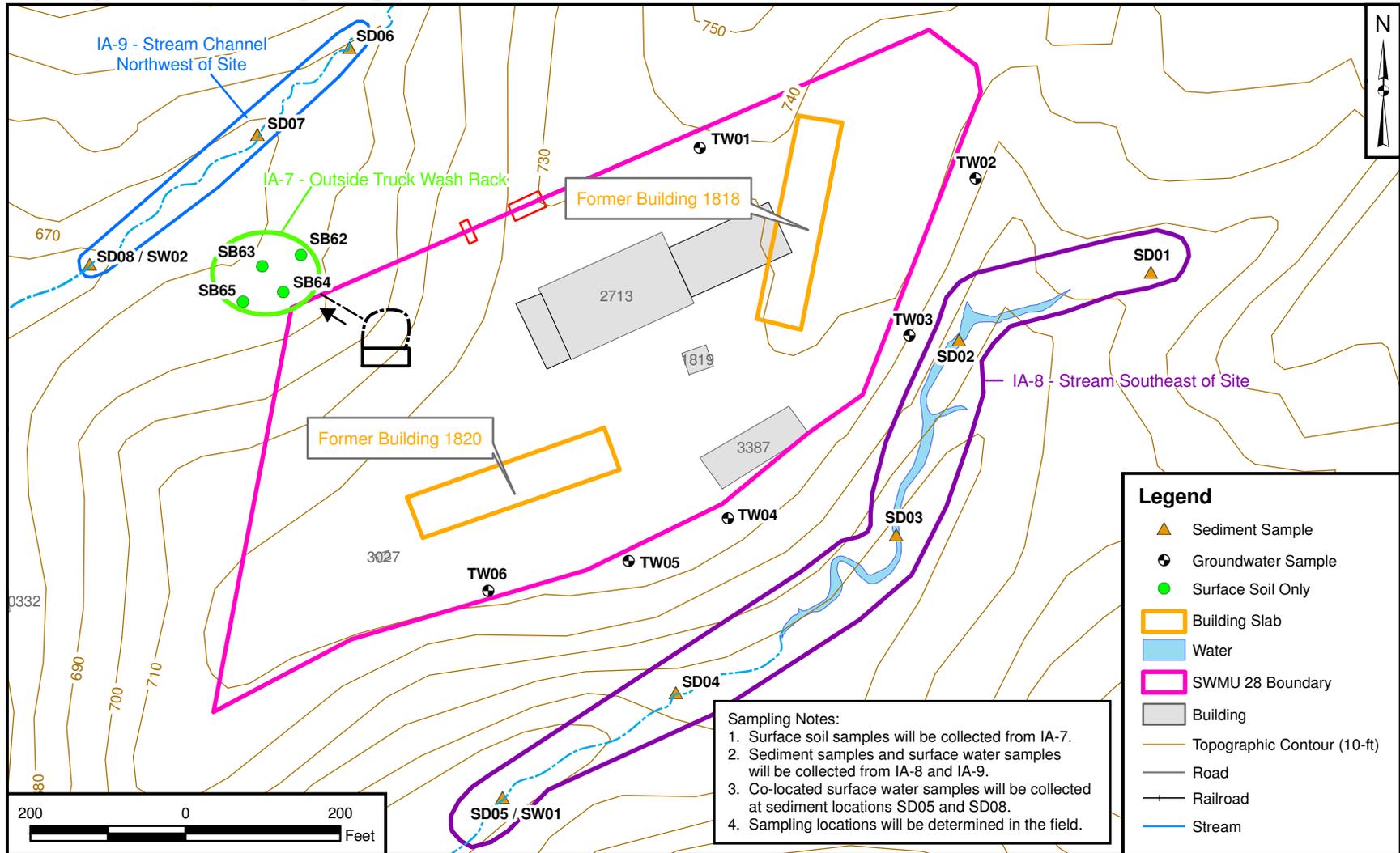
- Surface Soil Only
- ✕ Subsurface Soil Only
- Surface and Subsurface Soil
- ▨ Gravel Pad
- ▭ Former Building Slab Location
- Water
- ▭ SWMU 28 Boundary
- ▭ Building
- Drainage Pipe
- Topographic Contour (10-ft)
- Road
- Railroad
- Stream

DRAWN BY S. STROZ	DATE 10/06/10
CHECKED BY T. KLIMEK	DATE 4/11/11
REVISED BY K. MOORE	DATE 4/7/11
SCALE AS NOTED	



SAMPLING LOCATIONS - FORMER BUILDING 1820 AREA
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER F273	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 17-2	REV 0



DRAWN BY S. STROZ	DATE 10/29/10
CHECKED BY T. KLIMEK	DATE 4/11/11
REVISED BY K. MOORE	DATE 4/7/11

SCALE
AS NOTED



**SAMPLING LOCATIONS - GROUNDWATER, ADJACENT
STREAMS AND OUTSIDE TRUCK WASH RACK
SWMU 28 - MAINTENANCE SHOP BUILDING 1820 AREA
NSA CRANE
CRANE, INDIANA**

CONTRACT NUMBER F273	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 17-3	REV 0

APPENDIX A
FIELD STANDARD OPERATING PROCEDURES

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.msp?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@tetrattech.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@tetrattech.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 F273)
- 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 Confirmation Samples

All confirmation 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 Confirmation 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.

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

NN	AA	AANNNA	NNNN (Soils and Sediment only)
SWMU Number	Matrix	Sample Location Number	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha
 N = Numeric

SWMU Number (NN):

28 = SWMU 28

Matrix Code (AA):

SS = Surface Soil Sample
 SB = Subsurface Soil Sample
 SD = Sediment Sample
 GW = Groundwater Sample
 SW = Surface Water 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. (If composite samples are collected: "location" refers to a particular sampling grid represented by a composite sample).

3.1.2 Examples of Sample Nomenclature

The first surface soil sample collected from SWMU 28, at a depth of 0- to 2-feet bgs would be labeled as "28SS01SO0002".

The first sediment sample collected from sampling location 01 at SWMU 28 would be labeled as 28SD010006-01

3.3 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.

3.3.1 QC Sample Numbering

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

NN	AA	NNNNNN	NN
SWMU Number	QC Type	Date	Sequence Number (per day)

The QC types are identified as:

TB = Trip Blank

RB = Rinsate Blank

FD = Field Duplicate

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).

3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at SWMU 28 for a surface soil sample collected on collection date (mmddyear) would be designated as 28SSFDFXXXXXX-01.

The second duplicate of the day taken at SWMU 28 of a subsurface soil sample collected on collection date (mmddyear) would be designated as 28SBFDFXXXXXX-02.

The first rinsate blank associated with surface soil samples collected on collection date (mmddyear) would be designated as 28SSRBXXXXXX-01.

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:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- 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

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 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 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 TtNUS 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 3
 SOIL AND SEDIMENT SAMPLE LOG SHEET**

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____		Sample ID No.: _____		
Project No.: _____		Sample Location: _____		
<input type="checkbox"/> Surface Soil <input type="checkbox"/> Subsurface Soil <input type="checkbox"/> Sediment <input type="checkbox"/> Other: _____ <input type="checkbox"/> QA Sample Type: _____		Sampled By: _____ C.O.C. No.: _____ Type of Sample: <input type="checkbox"/> Low Concentration <input type="checkbox"/> 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):				
OBSERVATIONS / NOTES:			MAP:	
Circle if Applicable:			Signature(s):	
<input type="checkbox"/> MS/MSD	<input type="checkbox"/> Duplicate ID No.:			

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

Isopropanol (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 of 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, litter, etc.)
- 3.3 Turn the hand auger sampler into the ground to a depth of 6-inches. The 0- to 6-inch 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.
- 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: _____		Sample ID No.: _____		
Project No.: _____		Sample Location: _____		
<input type="checkbox"/> Surface Soil <input type="checkbox"/> Subsurface Soil <input type="checkbox"/> Sediment <input type="checkbox"/> Other: _____ <input type="checkbox"/> QA Sample Type: _____		Sampled By: _____ C.O.C. No.: _____ Type of Sample: <input type="checkbox"/> Low Concentration <input type="checkbox"/> 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):				
OBSERVATIONS / NOTES:			MAP:	
Circle if Applicable:			Signature(s):	
<input type="checkbox"/> MS/MSD	<input type="checkbox"/> Duplicate ID No.:			

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 inch Φ -1/2 inch Φ)" 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
Sand	fine 3/4" to No. 4	19.1 to 4.76
	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
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 22

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

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 fine-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. 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

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.

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- or 5-foot x 2-inch diameter open barrel or closed barrel sampler
- Probe sampling adapters
- Disposable acetate liners for soil barrel sampler
- Cast aluminum or steel expendable drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- 1-inch PVC well screen point

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 and/or inertial lift pump and/or bladder pump, silicon tubing, polyethylene (PE) tubing, water quality meter with a flow through cell (YSI 600 series or equivalent), 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.

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.

Open or closed barrel 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- or 5-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- or 5-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 in accordance with the decontamination SOP (SOP-06).

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:

- 4- or 5-foot x 2-inch diameter open barrel or closed barrel drive rods
- Probe sampling adapters
- Cast aluminum or steel expendable drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- 1 -inch diameter slotted PVC screens with slot widths of 0.01 to 0.02- inch
- Schedule 40 or schedule 80 PVC threaded or flush-jointed casings
- 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 and/or inertial pump and/or bladder pump
- Standard decontamination equipment and solutions

6.2 DPT Sampling Methodologies

Once the outer drive rod equipped with an expendable drive point or tip has been advanced by DPT to the target depth and/or the water table has been encountered, the 1-inch diameter PVC casing and screen will be lowered into the drive casing. Then, the screen will be attached to the drive point and the drive casing will be retracted, leaving a temporary well to collect a groundwater sample at the water table. If required, an annular seal and filter pack can also be incorporated into well design by utilizing appropriate grout machines and pre-pack filters.

- 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 and/or inertial 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 5 NTU
 - Specific conductance +/- 3%
 - Temperature +/- 3%
 - eH +/- 10 millivolts
 - Dissolved oxygen +/- 10%
- Samples will be collected using the peristaltic pump or a bladder pump (if inertial pump is used for development) set at 0.2 liters per minute or lower, depending on the yield of the formation.
- If using a bladder pump, samples will be collected directly from the pump discharge, and purging will begin immediately after development.
- If using a peristaltic pump, samples (with the exception of samples to be analyzed for VOCs) will be collected directly from the pump discharge. The peristaltic 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/bubble wrap 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
 - PAHs
 - PCBs
 - Total metals
 - Dissolved metals (if required)
- 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 when using a

peristaltic pump. 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.
- Peristaltic pump: 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 using the "straw method." The straw method involves 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

BEDROCK MONITORING WELL SHEET		WELL No.: _____
		PERMIT No: _____
PROJECT: _____	DRILLING Co.: _____	BORING No.: _____
PROJECT No.: _____	DRILLER: _____	DATE COMPLETED: _____
SITE: _____	DRILLING METHOD: _____	NORTHING: _____
GEOLOGIST: _____	DEV. METHOD: _____	EASTING: _____

	<p>Elevation of Top of Casing: _____</p> <p>Stick Up of Casing Above Ground Surface: _____</p> <p>Elevation of Top of Riser: _____</p> <p>I.D. of Surface Casing: _____</p> <p>Type of Surface Casing: _____</p> <p>Type of Surface Seal: _____</p> <p>I.D. of Permanent Casing: _____</p> <p>I.D. of Riser: _____</p> <p>Type of Riser: _____</p> <p>Borehole Diameter: _____</p> <p>Type of Backfill: _____</p> <p>Elevation / Depth Top of Seal: _____ / _____</p> <p>Elevation / Depth Top of Bedrock: _____ / _____</p> <p>Type of Seal: _____</p> <p>Elevation / Depth of Top of Fine Sand: _____ / _____</p> <p>Elevation / Depth of Top of Filter Pack: _____ / _____</p> <p>Elevation / Depth of Top of Screen: _____ / _____</p> <p>Type of Screen: _____</p> <p>Slot Size x Length: _____</p> <p>I.D. of Screen: _____</p> <p>Type of Filter Pack: _____</p> <p>Diameter of Hole in Bedrock: Core / Ream: _____</p> <p>Elevation / Depth of Bottom of Screen: _____ / _____</p> <p>Elevation / Total Depth of Borehole: _____ / _____</p>
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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

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

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 °C

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 Form 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

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)
- 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

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

STANDARD OPERATING PROCEDURE

SOP-18

SURFACE WATER SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface water samples at the Naval Support Activity (NSA) Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Surface Water Sample Log Sheet: A copy of this form is attached at the end of this SOP.

Field logbook

Writing utensil

Multi-parameter water-quality meter: The water-quality meter is used for the measurement of dissolved oxygen, pH, specific conductance, temperature, and oxidation-reduction potential (see SOP-17).

LaMotte Turbidity Meter: Used to measure turbidity in the field (see SOP-17).

Disposable sample containers: Disposable sample containers are used to fill sample containers and transport sample(s) to a pump for filtering.

Labeled sample containers: Prelabeled, certified-clean sample containers will be provided by the laboratory that performs the analyses.

0.45-micron filter assembly: These are single-use filter cartridges used to filter samples scheduled for dissolved metals analyses. The filters become investigation-derived waste (IDW) after one use.

Peristaltic pump

Silicon tubing

Ziploc-type plastic storage bags

Shipping containers (coolers)

Trip blank sample (if VOC samples are being collected)

Temperature blank

3.0 SURFACE WATER SAMPLING PROCEDURES

3.1 The same methods will be used to collect surface water and seep samples. Sampling will start at the downstream end of a stream and proceed to the farthest upstream location.

- 3.2 While standing downstream or from the bank, gently remove any floating leaves or twigs that may be present in a sample pool area in a manner that will not disturb the bottom sediment.
- 3.3 While standing downstream or from the bank, place the sample container in the water at the sampling location at a 45-degree angle and lower it to approximately half the sample pool depth. With the mouth of the container facing upstream, fill the container with water, being careful not to disturb the sediment.
- 3.4 All samples will be collected into certified-clean, pre-preserved bottles (if preservation is required for the analysis to be performed) supplied by the laboratory performing the analyses. Sample containers for volatile constituents (VOCs) must be completely filled so no headspace exists in the container. Other sample containers should not be filled completely; a small amount of air should be left at the top. Sample containers will be collected in the following sequence:

Volatile organic compounds (VOCs)

Other Organics

Total metals

Nitrate

Nitrite

Total suspended solids (TSS)

Dissolved metals

- 3.5 Record the date and time that the sample containers are filled on the Surface Water Sample Log Sheet, the sample labels, and the Chain-of-Custody Form.
- 3.6 After the sample label is completed and checked, place the sample container into a ziploc-type plastic storage bag and place the plastic storage bag holding the sample container into a cooler containing ice.
- 3.7 Repeat steps 3.3 through 3.6 until all the sample bottles containing unfiltered samples have been filled.
- 3.8 Fill two 1-liter unpreserved polyethylene bottles. Use these bottles to transfer the sample for field filtering. Set up a peristaltic pump for filtering of the dissolved metals samples. Using new, clean, disposable silicone tubing and a 0.45-micron filter, place the intake tubing from the pump into the

transfer bottle with the filter attached to the discharge end and start the pump. Pre-rinse the filter with approximately 50 milliliters of sample water prior to filling the sample containers.

- 3.9 Using the discharge from the filter cartridge, fill one 1-liter polyethylene sample bottle for dissolved metals. Repeat steps 3.8 and 3.9 for these sample containers.
- 3.10 Obtain measurements of dissolved oxygen, pH, specific conductance, temperature, turbidity, and oxidation-reduction potential using the multi-parameter water-quality meter and LaMotte Turbidity Meter (see SOP-17). Record the readings in the appropriate fields on the Surface Water Sample Log Sheet.
- 3.11 Estimate the flow rate of the stream or spring. This is an estimate only. Round the flow rate to the nearest 5 gallons and record this number on the Surface Water Sample Log Sheet.
- 3.12 Decontaminate all equipment and load the equipment and the sample cooler in the sample vehicle for transport.

4.0 ATTACHMENTS

1. Surface Water Sample Log Sheet

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX B
LAB STANDARD OPERATING PROCEDURES
TABLE OF CONTENTS

Empirical Laboratories

Empirical Laboratories DoD Certificate of Accreditation
SOP-100 Metals Digestion/Preparation
SOP-105 Metals
SOP-175 Cyanide
SOP-201 SVOCs and Low-Concentration PAHs
SOP-202 VOCs Aqueous
SOP-208 Herbicides
SOP-211 Pesticides/PCBs
SOP-219 TPH
SOP-221 Total Organic Carbon
SOP-225 VOCs Non-Aqueous
SOP-300 SVOCs Aqueous
SOP-302 Pesticides/PCBs Aqueous
SOP-320 TPH Non Aqueous
SOP-322 TPH Aqueous
SOP-343 BNA and Pesticide/ PCB
QS10-R14 Sample Receiving, Storage, and Login
QS14-R06 Waste Disposal

Test America Laboratories

Test America DoD Certificate of Accreditation
Test America DoE Certificate of Accreditation
TA-GS-0326 Hydrocarbon Identification
TA-GS-0356 EPH
TA-GS-0309 VPH



**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

A handwritten signature in black ink, appearing to read 'R.D.L.', positioned above a horizontal line.

**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

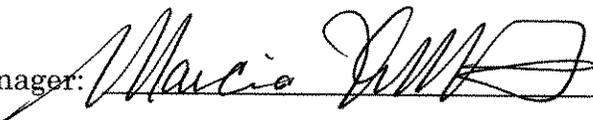
METALS DIGESTION/PREPARATION

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 liter bottles. Samples should be preserved with concentrated HNO₃ to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO₃ 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 (HNO₃). 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 (H₂SO₄). 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 HNO₃, 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 CLPIIM0 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 HNO₃, 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% HNO₃ and 5% HCl. Use 0.125 mLs HNO₃ 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 transferred 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 than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 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% HNO₃ 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 HNO₃ 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 HNO₃ 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 HNO₃ 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 HNO₃. **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 HNO₃ 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 HNO₃ 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 HNO₃ 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 HNO₃ 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 HNO₃ 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 homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
- b. Add 10 mL of 1:1 nitric acid (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 equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated 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 HNO₃, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO₃, repeat this step (addition of 5 mL of concentrated HNO₃) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO₃. 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 H₂O₂ 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 H₂O₂ to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H₂O₂.)
- 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 should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

XII. CORRECTIVE ACTIONS

- A. Sample boils during digestion.
 1. Redigest another sample aliquot.
- B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.
 1. Redigest another sample aliquot.
 2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

- A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.
- B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.
- C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.
- D. There are several precautions that must be taken to minimize the possibility of contamination.
 1. All metals glassware must be kept separate from all other laboratory glassware.
 2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.
- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
- F. Please consult Waste Disposal SOP-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 105 REVISION #: 16 EFFECTIVE DATE: 041110

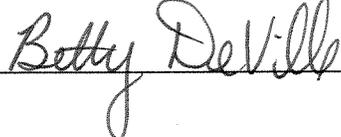
**METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE**

**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:  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 \pm 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 (0.001 gm).
- 9.11. Variable Eppendorf Pipettes 1000 μ L; 5000 μ L
- 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), HNO₃. 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% HNO₃ 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 CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier 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 1 gram 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 HNO₃ 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 $\pm 5\%$ for 200.7 and within $\pm 10\%$ for all other methods.

12.1.4. ICB/CCB less than two times \pm MDL or less than \pm LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within $\pm 20\%$ initially and client specific requirement of $\pm 30\%$ at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times \pm MDL or less than \pm LOD unless verified standard contamination for DOD. The IFB must recover within \pm 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 \pm 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 \pm MDL or $<\pm$ 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 \pm 10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 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 \pm 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 $\pm \frac{1}{2}$ RL for DOD or \pm RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either 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 \pm 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 $\pm 20\%$ 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% HNO₃ / 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 ($\pm 10\%$) for 200.7 ($\pm 5\%$) and ICB ($< \pm 2 \times \text{MDL}$, $< \pm \text{LOD-DOD}$ or $\pm \text{RL/CRDL}$ 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 $\pm 20\%$ for DOD projects and $\pm 30\%$ for samples analyzed for 6010C. Then reanalyze the

highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. 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: $< \pm\text{MDL}$ or $\pm\text{RL}/\text{CRDL}$ for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within $\pm 20\%$. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

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 $< \pm 2 \times \text{MDL}$ or $< \pm \text{LOD}$ 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 $< \text{LOD}$ (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within $\pm 20\%$ 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% HNO₃ / 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 $\pm 10\%$ 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 $< 2x \pm MDL$, for CLP $< RL$, for **DOD no analytes detected $> \pm LOD$** . 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: $Ca \text{ mg/L} \times 2.5 + Mg \text{ mg/L} \times 4.1 = \text{total Hardness in mg/L as } CaCO_3$.

15.2. The instrument will generate data results in mg/L or $\mu\text{g/L}$ (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.

$$SR \text{ (ug/g or mg/kg)} = IR * DF * FED / SM$$

SR	=	Sample result
IR	=	Instrument result ($\mu\text{g/L}$)
DF	=	Dilution factor (post digestion)
FED	=	Final volume of digestate (L)
SM	=	Sample mass digested (g)

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 Manual 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 through 10.7.3 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 $\pm 50\%$.

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 $\pm 10\%$ or $\pm 5\%$ 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 \pm MDL or within \pm 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 \pm 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 \pm 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 \leq 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 \pm 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 \pm 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 $\pm 2 \times \text{MDL}$ or CRDL for CLP, DOD no analytes detected $> \pm \text{LOD}$.
 - 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 $\pm \frac{1}{2} \text{RL}$ and $\pm \text{RL}$ 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 $\pm 20\%$ (if both are $> 5 \times \text{CRDL}$) or \pm the CRDL (if either are $< 5 \times \text{CRDL}$).
 - a. The associated sample data must be qualified on the final report.
- 19.3.2. Spike analysis recovery not within $\pm 20\%$.
 - 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 $\pm 25\%$ for SW6010B, DOD or $\pm 20\%$ 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 $\pm 10\%$
 - a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?

- i. If no, the serial dilution data can not be evaluated.
- iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

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 Regulations, 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	<ul style="list-style-type: none"> once per calibration 	<ul style="list-style-type: none"> IFA less than LOD if not verified contamination of standard. IFB must be within $\pm 20\%$. 	<ul style="list-style-type: none"> Check IEC corrections for metals in the IFA.
Calibration Curve	<ul style="list-style-type: none"> 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). 	<ul style="list-style-type: none"> Linear calibration Corr. of 0.998 Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICB	At the beginning of every sequence	Must meet the $< \pm \text{LOD}$ for DOD or $< 2 \times \text{MDL}$	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-client samples 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than $\frac{1}{2} \pm \text{RL}$. 	<ul style="list-style-type: none"> 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-prepare 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.	<ul style="list-style-type: none"> • 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	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Must meet the criteria of the BS for average accuracy 	<ul style="list-style-type: none"> • 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**

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.

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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

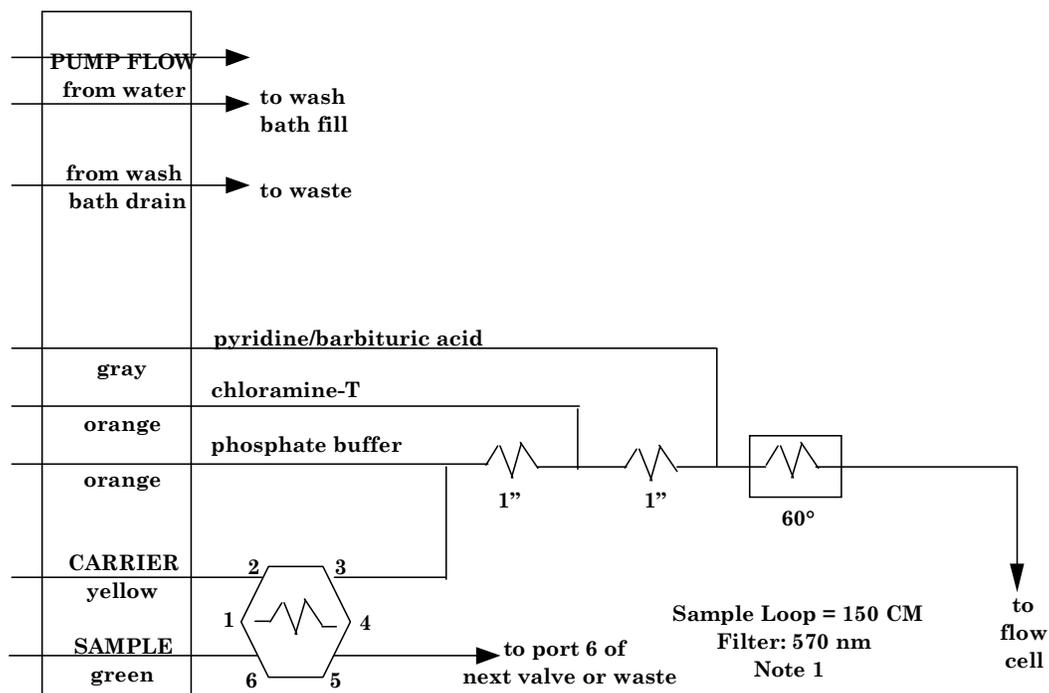
(Peak should be centered in chord 3)

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

Note1: A 3.0 m x 0.022" id back pressure loop is placed at the exit of the flow cell.

1" is 70.0 cm of tubing on a 1 in coil support

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 μ L/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 $< \pm 2x$ MDL or $< +$ CRDL or $\frac{1}{2}$ 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 $\pm 15\%$.
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 $\pm 30\%$.

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 $\pm 10\%$
 - 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 $\pm 10\%$
 - 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 \pm RL or CRDL or $\frac{1}{2}$ 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 \pm RL or CRDL or $\frac{1}{2}$ 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 ($\pm 20\%$).
 - 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 $\pm 15\%$.
 - 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 $\pm 20\%$
 - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within $\pm 20\%$
 - 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	<ul style="list-style-type: none"> Prior to analyzing any samples A minimum of 5-points for linear fits Low standard at the RL/LOD level 	<ul style="list-style-type: none"> For Linear fits a RF of 0.995 Average RSD $\leq 20\%$ Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICV	Alternate source standard to be analyzed after every calibration curve	Must meet 85 to 115%	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-client samples 	<ul style="list-style-type: none"> $\pm 10\%$ difference 	<ul style="list-style-type: none"> Follow guidelines for SOP QS05
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> $\pm 10\%$ difference 	<ul style="list-style-type: none"> Follow guidelines for SOP QS05
BLK/CCB	One per prep batch/ after every CCV	<ul style="list-style-type: none"> Must be less than $\frac{1}{2}$ RL/Must be less than the LOD 	<ul style="list-style-type: none"> 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-prepare 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	<ul style="list-style-type: none"> $\pm 20\%$ difference 	<ul style="list-style-type: none"> Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	<ul style="list-style-type: none"> $\pm 20\%$ difference 	<ul style="list-style-type: none"> 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.	<ul style="list-style-type: none"> • $\pm 20\%$ difference 	<ul style="list-style-type: none"> • Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	<ul style="list-style-type: none"> • $\pm 20\%$ difference 	<ul style="list-style-type: none"> • Follow guidelines from SOP QS05
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Average percent recovery should be between 80-120%, with a 20% standard deviation. 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis
MDL Study	Once per year for specific state requirements	<ul style="list-style-type: none"> • Calculated value must be greater than 10% of the Spike Level • Calculated value must be less than the Spike level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	<ul style="list-style-type: none"> • Parameter must be detected • 2nd column / detector confirmation is required • the response must be 3-times the noise level 	<ul style="list-style-type: none"> • Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	<ul style="list-style-type: none"> • Bias Requirement: 50-150% • The LOQ value must be greater than the LOD value 	<ul style="list-style-type: none"> • 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.

Table of Contents

1. Identification of the Test Method
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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 Quantitation).

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 5µL 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 $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ 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:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

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 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 $\mu\text{g/L}$.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

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 $\mu\text{g/L}$.

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 $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

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 $\leq 20\%$ 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 $\leq 20\%$. 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.	<p>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</p> <p>2. RSD for RFs for CCCs: SVOCs $\leq 30\%$ and one option below: Option 1: RSD for each analyte $\leq 15\%$; [$\leq 20\%$ for non-DoD 8270D; or, $\leq 35\%$ for non-DoD 625] Option 2: linear least squares regression $r \geq 0.995$ or $r^2 \geq 0.990$; [$r \geq 0.990$ 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).</p>	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 $\pm 20\%$ of true value [$\pm 25\%$ for non-DoD 8270C; or, $\pm 30\%$ 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 $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ 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: $RPD \leq 30\%$ 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)

QC Check	Minimum Frequency	Acceptance Criteria			Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	Surrogate	Water	Solid	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. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.	
		Nitrobenzene-d5	40-110	35-100			
		2-Fluorobiphenyl	50-110	45-105			
		Terphenyl-d14	50-135	30-125			
		Phenol-d6	10-115	40-100			
		2-Fluorophenol	20-110	35-105			
		2,4,6-Tribromophenol	40-125	35-125			
		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.					
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 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

<u>base/neutral mix (2000ppm)</u>	<u>acids mix (2000ppm)</u>
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	<u>semivoa misc. mix(2000ppm)</u>
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	<u>Benzidine mix (2000ppm)</u>
4-Chlorophenyl-phenylether	Benzidine
Diethylphthalate	3,3'-Dichlorobenzidine
Benzo(a)anthracene	
Bis(2-ethylhexyl)phthalate	
Butylbenzylphthalate	
Chrysene	<u>Individual or misc. mixes (2000/5000/20,000ppm)</u>
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-methylnaphthalene
Dibenz(a,h)anthracene	2,6-dichlorophenol
Di-n-octylphthalate	2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene	

<u>BNA internals (2000ppm)</u>	<u>Acid surrogate (7500ppm)</u>
1,4-Dichlorobenzene-d4 (L.S)(1)	2-Fluorophenol (S)
Naphthalene-d8 (L.S)(35)	Phenol-d6 (S)
Acenaphthene-d10 (L.S) (59)	2,4,6-Tribromophenol (S)
Phenanthrene-d10 (L.S) (79)	2,-Chlorophenol-d4 (S)
Chrysene-d12 (L.S) (92))	<u>BN surrogate (5000ppm)</u>
Perylene-d12 (L.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-Nitrosopyrrolidine	1	100		59	
N-Nitrosomorpholine	1	56		59	
O-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)

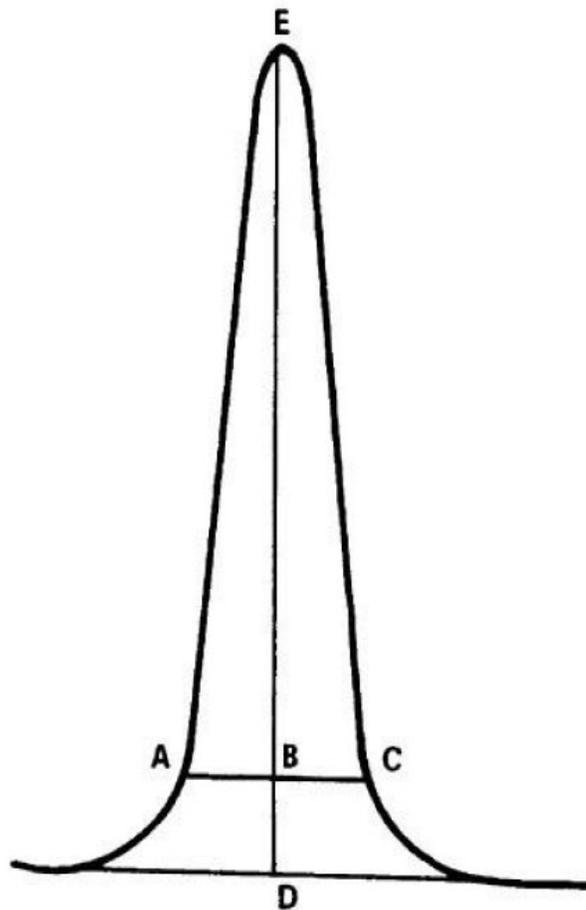
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene

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.

Table of Contents

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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, NaHSO₄ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- 10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ or less from the date they are received/prepared. The freezer temperature is monitored daily with 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 50 μL syringe, 40 μL 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 $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ or less for up to 6 months (**1 week for South Carolina samples**). A direct injection of 1 μL (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 1 μL 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 $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 1 week. A 100μg/L (5mL purge) standard is made using 50μL of this standard to 50mL of 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 $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ 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 1.0 μ L 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 background 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 $\pm 20\%$ for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

- 15.2.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, $D = 1$. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{\text{RF}}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

- 15.2.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(W_s)(1000)}$$

where: A_s , A_{is} , C_{is} , D , and \overline{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).
- 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, BFB Tuning Criteria
- 22.6 Table 6, Analyst Checklist
- 22.7 Table 7, INTERNAL STANDARD ASSOCIATION

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.	<p>1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>2. RSD for RFs for CCCs: VOCs $\leq 30\%$ and one option below: Option 1: RSD for each analyte $\leq 15\%$; Option 2: linear least squares regression $r \geq 0.995$; 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).</p>	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 $\pm 20\%$ of true value. [$\pm 25\%$ 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.	<u>1. Average RF for SPCCs:</u> VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. <u>2. %Difference/Drift for all target compounds and surrogates:</u> VOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ 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 \pm 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.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ 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: $RPD \leq 30\%$ 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.	Surrogate	WATER	SOIL	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.
		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.					
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)

	Yes	No	NA	Second Review	Level
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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 7, Internal Standard Association

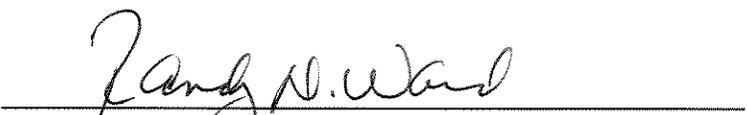
Analyte	Internal Standard	Analyte	Internal Standard
1,1,1-Trichloroethane	Fluorobenzene	1,1,1,2-Tetrachloroethane	d5-Chlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Fluorobenzene	1,1,2-Trichloroethane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2,3-Trichloropropane	d5-Chlorobenzene
1,1-Dichloropropene	Fluorobenzene	1,2-Dibromoethane (EDB)	d5-Chlorobenzene
1,2-Dichloroethane	Fluorobenzene	1,3-Dichloropropane	d5-Chlorobenzene
1,2-Dichloroethane-d4	Fluorobenzene	1-Chlorohexane	d5-Chlorobenzene
1,2-Dichloroethane (total)	Fluorobenzene	2-Hexanone	d5-Chlorobenzene
1,2-Dichloropropane	Fluorobenzene	Bromofluorobenzene	d5-Chlorobenzene
1,4-Dioxane	Fluorobenzene	Bromoform	d5-Chlorobenzene
2,2-Dichloropropane	Fluorobenzene	Chlorobenzene	d5-Chlorobenzene
2-Butanone	Fluorobenzene	Chlorobenzene-d5	d5-Chlorobenzene
2-Chloroethyl vinyl ether	Fluorobenzene	Dibromochloromethane	d5-Chlorobenzene
4-Methyl-2-pentanone	Fluorobenzene	Ethyl Methacrylate	d5-Chlorobenzene
Acetaldehyde	Fluorobenzene	Ethylbenzene	d5-Chlorobenzene
Acetone	Fluorobenzene	m,p-Xylene	d5-Chlorobenzene
Acetonitrile	Fluorobenzene	Methacrylonitrile	d5-Chlorobenzene
Acrolein	Fluorobenzene	o-Xylene	d5-Chlorobenzene
Acrylonitrile	Fluorobenzene	Styrene	d5-Chlorobenzene
Allyl chloride	Fluorobenzene	Tetrachloroethane	d5-Chlorobenzene
Benzene	Fluorobenzene	Toluene	d5-Chlorobenzene
Bromochloromethane	Fluorobenzene	Toluene-d8	d5-Chlorobenzene
Bromodichloromethane	Fluorobenzene	trans-1,3-Dichloropropene	d5-Chlorobenzene
Bromomethane	Fluorobenzene	Xylenes (total)	d5-Chlorobenzene
Carbon disulfide	Fluorobenzene	1,1,2,2-Tetrachloroethane	1,4-dichlorobenzene-d4
Carbon tetrachloride	Fluorobenzene	1,2,3-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroethane	Fluorobenzene	1,2,4-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroform	Fluorobenzene	1,2,4-Trimethylbenzene	1,4-dichlorobenzene-d4
Chloromethane	Fluorobenzene	1,2-Dibromo-3-chloropropane	1,4-dichlorobenzene-d4
Chloroprene	Fluorobenzene	1,2-Dichlorobenzene	1,4-dichlorobenzene-d4
cis-1,2-Dichloroethane	Fluorobenzene	1,3,5-Trimethylbenzene	1,4-dichlorobenzene-d4
cis-1,3-Dichloropropene	Fluorobenzene	1,3-Dichlorobenzene	1,4-dichlorobenzene-d4
Cyclohexane	Fluorobenzene	1,4-Dichlorobenzene	1,4-dichlorobenzene-d4
Dibromofluoromethane	Fluorobenzene	1,4-Dichlorobenzene-d4	1,4-dichlorobenzene-d4
Dibromomethane	Fluorobenzene	2-Chlorotoluene	1,4-dichlorobenzene-d4
Dichlorodifluoromethane	Fluorobenzene	4-Chlorotoluene	1,4-dichlorobenzene-d4
Diisopropyl Ether	Fluorobenzene	Bromobenzene	1,4-dichlorobenzene-d4
Ethyl tert-Butyl Ether	Fluorobenzene	cis-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Fluorobenzene	Fluorobenzene	Hexachlorobutadiene	1,4-dichlorobenzene-d4
Hexane	Fluorobenzene	Naphthalene	1,4-dichlorobenzene-d4
Iodomethane	Fluorobenzene	n-Butylbenzene	1,4-dichlorobenzene-d4
Isobutyl alcohol	Fluorobenzene	n-Propylbenzene	1,4-dichlorobenzene-d4
Isopropylbenzene	Fluorobenzene	p-Isopropyltoluene	1,4-dichlorobenzene-d4
Methyl Acetate	Fluorobenzene	sec-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Methacrylate	Fluorobenzene	tert-Butylbenzene	1,4-dichlorobenzene-d4
Methyl t-Butyl Ether	Fluorobenzene	trans-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Methylcyclohexane	Fluorobenzene		
Methylene chloride	Fluorobenzene		
Propionitrile	Fluorobenzene		
t-Butyl alcohol	Fluorobenzene		
Tert-Amyl Methyl Ether	Fluorobenzene		
Tetrahydrofuran	Fluorobenzene		
trans-1,2-Dichloroethane	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 0.5 μ m film thickness fused silica column.

5.2.3 RTX CLP II(or equivalent): 30 m x 0.32 mm ID 0.25 μ m 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.

<u>Analyte</u> <u>(Methyl Ester)</u>	<u>Conversion</u> <u>Factor</u>
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 ≥ 0.995 . Quadratic can be used if correlation coefficient is ≥ 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 $\pm 20\%$ 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:

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

8.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

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 $\pm 15\%$ 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 $\mu\text{g/L}$.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

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 $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

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 \pm 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 contaminants; < 1/2 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	SAMPLE	LCS	LCS	QC.	%	LIMITS
COMPOUND	ADDED (µg/L)	CONCENTRATION (µg/L)	CONCENTRATION	CONCENTRATION (µg/L)	REC #	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	2 nd 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	2 nd 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.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
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15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
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22. Tables, Diagrams, Flow charts and Validation Data

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:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

- 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.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

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 $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

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 ± 0.030 min.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation $\leq 15\%$ for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation $\leq 15\%$ 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 $\leq 20\%$; Option 2: linear least squares regression: $r \geq 0.995$; 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 $\pm 20\%$ 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 $\pm 20\%$ 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 $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem, then see SOP QS05. If required, prep 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 prep 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 \leq 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 \leq 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

	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: SOP 219

REVISION #: 14

EFFECTIVE DATE: 20101201

**GAS CHROMATOGRAPHY/FLAME IONIZATION DETECTOR
(GC/FID) NONHALOGENATED VOLATILE ORGANICS
AND TOTAL PETROLEUM HYDROCARBONS (TPH)
BY METHOD 8015B/8015C/TN EPH/GRO**

APPROVALS:

Lab Director:



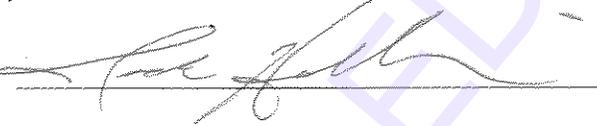
Date: 12/1/10

Data Quality Manager:



Date: 12/1/10

Section Supervisor:



Date: 12/1/10

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Changes Summary

Revision 14, 20101201

- The SOP is an update from Revision 13 dated 09/20/10.
- Updated current temperature programs used.
- Added surrogates and gasoline (instead of individual components) to calibration requirement.
- Added a note clarifying how to prep GRO water samples.

Revision 13, 20100920

- The SOP is an update from Revision 12 dated 09/09/10.
- All temperature references of 1°C-4.4°C were revised to reflect 0°C-6°C.

Revision 12, 09/09/10

- The SOP is an update from Revision 11 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.
- References to alcohol or glycol analysis have been removed.
- SOP references have been updated.
- Tables 1 and 2 have been added.

Table of Contents

1. Identification of the Test Method
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**GAS CHROMATOGRAPHY/FLAME IONIZATION DETECTOR
(GC/FID) NONHALOGENATED VOLATILE ORGANICS
AND TOTAL PETROLEUM HYDROCARBONS (TPH)
BY METHOD 8015B/8015C/TN EPH/GRO**

1.0 Identification of the Test Method

The GC/FID system is used to analyze nonhalogenated Volatile Organics (VO), TPH and gasoline range organics/diesel range organics (GRO/DRO) compounds.

2.0 Applicable Matrix or Matrices

This Standard Operating Procedure, SOP, is used for the analysis of Gasoline range organic and Diesel range organic compounds in a variety of matrices including Solids: Soil, Sediments, etc. and Waters.

3.0 Detection Limit

See **Table 1** of this SOP.

4.0 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. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table. We presently use this method to analyze ethylene glycol, methanol, GRO/DRO and TPH. This SOP will describe the different analyses performed using a temperature programmable gas chromatograph configured with a flame ionization detector (FID).

5.0 Summary of the Test Methods

- 5.1 GRO: Are Purged using a purge and trap method. Samples are then injected onto a GC. The analytes are run in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the FID.
- 5.2 DRO: After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are run in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the FID.
- 5.3 DROs/GROs are multi-component ranges. They are identified based on Retention time windows set by the first CCV. Ranges 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.

Acronyms

°C	-	degrees centigrade
CF	-	calibration factor
CRDL	-	contract required detection limit
%D	-	percent difference
DOD	-	Department of Defense
DRO	-	diesel range organics
EPH	-	extractable petroleum hydrocarbons
FID	-	flame ionization detector
GC	-	gas chromatograph
GRO	-	gasoline range organics
LCS	-	laboratory control sample
LCSD	-	laboratory control sample duplicate
MDL	-	method detection limit
mg/KG	-	milligrams per kilogram
mg/L	-	milligrams per liter
µL	-	microliter
µm	-	micrometer
ml	-	milliliter
mm	-	millimeter
MS	-	matrix spike
MSD	-	matrix spike duplicate
%RSD	-	percent relative standard deviation
RT	-	retention time
SOP	-	standard operating procedure
TPH	-	total petroleum hydrocarbons
VPH	-	volatile petroleum hydrocarbons

7.0 Interferences

Section 3.0 of SW-846 Method 8015B and Section 4.0 of Method 8015C details interferences and potential problems which may be encountered when dealing with non-halogenated organic analyses by this method.

8.0 Safety

Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab wide. 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.

- 8.1 Care should be used in handling all samples.
- 8.2 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 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 in the Data Quality Managers office.

9.0 Equipment & Supplies

- 9.1 Gas Chromatograph
 - 9.1.1 HP 5890 Series II (temperature programmable). – DRO/GRO
- 9.2 Autosampler and Concentrator
 - 9.2.1 HP-7673 injector - DRO
 - 9.2.2 OI 4560 Concentrator - GRO
 - 9.3.3 Tekmar 3000 Concentrator - GRO
 - 9.3.4 ARCHON 5100 Autosampler - GRO
- 9.3 Columns-Capillary columns.
 - 9.3.1 RTX-5 - 30 meter x 0.32 mm ID 0.25 μ m film thickness fused silica - used for DRO analyses.
 - 9.3.2 RTX-502.2 - 105 meter x 0.53 mm ID, 3.0 μ m film thickness fused silica - used for TN GRO analyses used for other volatile analyses.
- 9.4 Data Acquisition and Processing Software
 - 9.4.1 HP Chemstation system is interfaced to the HP-GC for data acquisition and storage.
 - 9.4.2 TARGET data system is interfaced to the acquisition systems. 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 specific reagents and standards used for the performance of the method:
 - 10.1.1 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.
 - 10.1.2 Stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. GRO standard mixtures contain analytes from C6-C10, and are purchased from the vendors mentioned above. DRO standard mixtures contain analytes from C10-C28 and are also purchased from the vendors mentioned above. The source is dependent on method and client requirements. Make certain to verify the state for which the samples are to be analyzed so the appropriate calibration standards are used. 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 Intermediate standards are prepared from the stock standards. Intermediate and working standards are made up using the appropriate solvent or laboratory reagent blank water and noted using the LIMS system, detailing how they were made, solvent used (reagent water, methylene chloride, methanol), date made, expiration date (six months or sooner from date of preparation) and given a sequential number.

10.3 TPH-GRO/DRO

10.3.1 GRO

A ten component standard, (C6-C10), from Restek (Wisconsin PVOC mix or equivalent) is used for setting the retention time range. 4-bromofluorobenzene from Restek or equivalent is used as the surrogate and should be calibrated in the same manner as target analytes. Calibration standards must be prepared at a minimum of five concentration levels for each parameter of interest through dilution of the intermediate stock standard in appropriate solvent or laboratory reagent blank water. Use a gasoline standard for Method 8015 for calibration and quantitation. One of the concentration levels should be near but above the method detection limit (MDL). The remaining concentration levels should correspond to the expected range of concentrations found in the real samples or defines the working range of the GC-FID system.

10.3.2 DRO

A solution containing even numbered alkanes from C8 to C40 is run to determine the retention times for the appropriate analyses. Ortho-Terphenyl from Restek or equivalent is used as the surrogate and should be calibrated in the same manner as target analytes. Calibration standards must be prepared at a minimum of five concentration levels for each parameter of interest through dilution of the intermediate stock standard in the appropriate solvent or laboratory reagent blank water. Use a diesel standard for Method 8015 for calibration and quantitation. One of the concentration levels should be near but above the method detection limit (MDL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or defines the working range of the GC-FID system.

10.3.3 Surrogate Standards

For GRO (BFB) and DRO (OTP) analysis are used to monitor the performance of the analytical system, and the effectiveness of the method in dealing with each sample matrix. Corrective action is taken when surrogates are out of recovery limits. An NCR form is filled out within 24 hours and the supervisor is notified immediately. The supervisor will then make suggestions as to what action needs to be taken such as the sample may require re-extraction, re analysis, or the report flagged for a QC problem.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. All water volatile samples are stored in the water walk-in cooler and soils in the soil walk-in cooler at a temperature of 0°C – 6°C. Water and soil volatile samples have holding times of 14 days from date of sampling if preserved (unless otherwise specified for the project). All

organic extractable water and soil samples are stored in their respective walk in coolers at a temperature of 0°C – 6°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 0°C – 6°C. Soil samples have a holding time of 14 days, waters 7 days from date of sampling for extraction (unless otherwise specified for the project). Extracts have a holding time of 40 days for analysis.

12.0 Quality Control

Quality control for this method can be referenced in SW-846 Method 8000B Section 8.0.

- 12.1 A method blank is required every 20 samples or at the frequency required by the client or regulatory agency (1/matrix/batch/20 which ever is at the determined frequency). **See table 2** for acceptance criteria and corrective action.
- 12.2 An MS/MSD pair are required every 20 samples per matrix. **See table 2** for acceptance criteria and corrective action.
- 12.3 A Laboratory Control Sample (LCS) is required every 20 samples or at the frequency required by the client or regulatory agency. **See table 2** for acceptance criteria and corrective action.
 - 12.3.1 TPH TN Method criteria require LCS/LCSD for GRO and DRO. The recommended control spike for GRO is API PS-6 or other gasoline of similar composition and the DRO control standard spike is a commercial diesel #2. In both cases (GRO and DRO) the required recovery for the LCS is 50-150%. GRO or DRO sample analyses can not proceed until this criteria is met.
 - 12.3.2 The other analytes measured by this method do not specify an exact recovery range, but these ranges are developed in-house by charting LCS recoveries. Default limits of 50%-150% are used until in-house limits are generated.
 - 12.3.3 Calculate surrogate recovery on all samples, blanks, and spikes. The surrogate recovery is checked to see if it is within the recovery limits. **See table 2** for acceptance criteria and corrective action.
- 12.4 Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with these methods. See SOP QS08 for guidance.

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.

Refer to SW-846 Method 8000B/8000C for proper calibration techniques.

 - 13.1.1 Five point calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters.

13.1.2 The area for GRO and DRO in each calibration point is determined by using a baseline to baseline integration over the appropriate retention time range. Refer to SOP QS07 for guidance on manual integration. The calibration factor (CF) for each point is determined by dividing the total area by the concentration of the solution. The percent relative standard deviation of the calibration factor (CF) should be less than 20 percent (25 percent for TN-TPH) over the working range for each analyte of interest. If the percent relative standard deviation (% RSD) of the calibration factor is less than 20 percent over the working range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve (linear curve corr. >0.995, quadratic >0.99 with six points). The curve is then verified using a second source standard (80-120% criteria).

13.1.3 The calibration curve must be verified every day through the analysis of a mid-level standard at the beginning and end of the sequence and after every 10 field samples. The percent difference back to the curve must not exceed 20 percent (25 percent for TN-TPH) for any analyte of interest. If this criteria is not met corrective action must be taken before sample analyses continues. This might include maintenance of the analytical system (see 14.1.3) and/or recalibration of each analyte that exceeded this criteria. Mid-level checks must be analyzed every 10 samples for TN-TPH and 10 field samples for DOD.

14.0 Procedure

The following information describes the instrument and QC requirements to analyze the compounds that we do by this method.

14.1 The Volatile Petroleum compounds VPH/GRO, are introduced into the temperature programmable gas chromatograph by transfer from a purge-and-trap concentrator (Method 5030B), boiling point ranges from 60°C-170°C.

Note: When preparing waters for GRO analysis, measure a 5mL aliquot of the sample by removing the plunger of a 5.0mL syringe and **pouring** the sample into the barrel of the syringe. Replace the plunger, invert the syringe, force out any air bubble, and measure 5ml (discarding any excess). Place in a VOA vial and cap tightly.

14.2 TPH extractable petroleum hydrocarbons EPH/DRO, boiling point range >170°C, require extraction (see SOP-320, and 322) into methylene chloride followed by direct injection.

14.3 Instrumentation

14.3.1 Purge and trap conditions - GRO

- a. Purge: 11 minutes at 40° C.
- b. Desorb: 2.0 minutes at 240° C.
- c. Bake: 10 minutes at 260° C.

14.3.2 GC and GC oven conditions (all temperature programs can be adjusted to better fit the range of analytes requested).

14.3.2.1 GRO (5ml purge and trap)

- a. Initial Temperature: 35° C hold for 5.0 minutes.
- b. Ramp1: 10° C/min to 200° C

- c. Ramp2: 5° C/min to 220° C.
- d. Final Temperature: 220° C hold for 1.5 minutes.

- 14.3.2.2 DRO (1.0 µL direct injection of extract)
- a. Initial Temperature: 45° C hold for 2.0 minutes.
 - b. Ramp1: 40° C/minute to 340° C
 - c. Final Temperature 340° C hold for 16.0 minutes

- 14.3.2.3 Alcohol (Direct aqueous injection)
- a. Flow: Between 8.0 - 10.0 mL / minute.
 - b. Initial Temperature: 40° C hold for 8.0 minutes.
 - c. Ramp: 10°C / minute to 200° C.
 - d. Final Temperature 200° C hold for 1.0 minute.

- 14.3.2.4 Ethylene Glycol (Direct aqueous injection)
- a. Flow: 8.0 - 10.0 mL / minute.
 - b. Initial Temperature: 110° C hold for 1.0 minute
 - c. Ramp: 8.0° C / minute to 220° C.
 - d. Final Temperature: 220° C hold for 1.0 minute.

14.4.3 Maintenance

14.4.3.1 Purge and Trap.

- a. Bake out the transfer line at 125° C and bake out the trap.
- b. Flush out the sample and/or transfer lines with methanol.
- c. Change the trap.

14.4.3.2 Gas Chromatograph.

- a. Clean or deactivate glass injection port insert or replace with a cleaned and deactivated insert.
- b. Trim the first few inches of the injection port side of the column.
- c. Remove the column and back-flush according to the manufacture instructions.
- d. If all else fails to correct the problem, the metal injector body may need to be deactivated or the column replaced.

14.5 Sample analysis will begin after the system performs the various checks outlined in Section 12.

14.5.1. A mid-level standard must be run at the beginning and end of the sequence and after every 10 field samples (every 10 samples for TPH) and cannot exceed 20 percent (25 percent for TN-TPH) difference from the initial calibration. A mid-level standard must also be analyzed at the end of the analysis sequence. If the mid-level check at the end fails, it is an indicator that GC maintenance is required (see 14.1.3).

14.5.2 The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples. Retention times for the range GRO C6-C10 is set by subtracting 0.03min. from the RT of 2-Methyl Pentane and adding 0.13min. to the RT of 1,2,4-Trimethylbenzene.

Retention times for the range DRO C10-C28 is set by subtracting 0.05min. from the RT of C10 and adding 0.05min. to the RT of C28.

- 14.6 Following sample analysis, the data is reduced using the instrument data system. The following must be checked to see if the samples will require re-analyses or dilution.
- 14.6.1 The analyte concentration 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 top half of the curve.
- 14.6.2 Surrogate standard recovery must be checked to determine if it is within control limits (see 12.3.3 sec. A).
- 14.7 Any questions left by this SOP should be answered by reading the referenced methods, paying close attention to SW-846 Method 8015B/8015C or the State specific TPH Method. If questions still remain unanswered, check with the Organic Lab Manager, Technical Director or Data Quality Manager.

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. Calculations:

Calibration Factor (CF) = $\frac{\text{Total Area within Retention Time Range}}{\text{Mass Injected (in ng)}}$

15.2.1 Aqueous Sample:

$$\text{Concentration} = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(V_s)}$$

where:

A_x = Area of the appropriate carbon range of analyte in the sample.

V_t = Total volume of the concentrated extract (µL).

D = Dilution Factor if the sample were diluted prior to analysis. If no dilution was made, D = 1.

CF = Mean calibration factor from the initial calibration (area/ng).

V_i = Volume of extract injected (µL). The injection volume for samples and calibration standards must be the same. For purge and trap analyses V_i, is not applicable and therefore set to 1.

V_s = Volume of the aqueous sample extracted in mL.

Using the units specified here for these terms will result in a concentration in units of ng/mL which is equivalent to µg/L.

15.2.2 Non-aqueous sample

$$\text{Concentration} = \frac{(A_x)(V_t)(D)}{(CF)(V_i)(W_s)}$$

where:

A_x , V_t , D , CF , and V_i are the same as for aqueous samples and W_s = Weight of the sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data. *Using the units specified here for these terms will result in a concentration in units of ng/g which is equivalent to ug/kg.*

16.0 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria.

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. Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

21.0 References

- 21.1 *Test Methods for Evaluating Solid Waste, SW-846.*
- 21.2 *Tennessee Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID.*

21.3 *Tennessee Method for Determination of Gasoline Range Organics.*

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters with the applicable MDL/DL, LOD, LOQ/RL and Units.
- 22.2 Table 2, for all technical methods, should always be the QA/QC summary table.
- 22.3 Table 4, Data Reviewers Checklist

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TABLE 1

Analyte	MDL/DL	LOD	MRL/LOQ	Units
TPH DRO	6.67	6.67	6.67	mg/Kg
TPH GRO	2.50	5.00	7.50	mg/Kg
TPH DRO	0.100	0.100	0.100	mg/L
TPH GRO	0.0500	0.100	0.150	mg/L

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Table 2. Organic Analysis by Gas Chromatography (Method 8015)

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 or 0.03min., whichever is greater.	NA.	NA.	
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 $\leq 20\%$ Option 2: linear least squares regression: $r \geq 0.995$ 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 projects.
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.	

Table 2. Organic Analysis by Gas Chromatography (Method 8015)

Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects) , and at the end of the analysis sequence.	All project analytes within established retention time windows. <u>GC methods:</u> All project analytes within $\pm 20\%$ of expected value from the ICAL;	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 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 should 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 $> \frac{1}{2}$ RL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	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.
Laboratory control sample (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.

Table 2. Organic Analysis by Gas Chromatography (Method 8015)

Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	For matrix evaluation, use LCS acceptance 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% (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.	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.	

ANALYST DATA REVIEW CHECKLIST

Instrument: _____

Run Date: _____

Sample Number(s):
Batch Number(s):
Method: 8015DRO/TNEPH/FLPRO

	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of five Calibration Standards?	_____	_____	_____	_____
2. Is the low standard at or below the LOQ/RL?	_____	_____	_____	_____
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 20 samples (10 for DoD) and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
3. Are Surrogate recoveries within QC limits?	_____	_____	_____	_____
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?	_____	_____	_____	_____
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than 1/2 the LOQ/RL?	_____	_____	_____	_____
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 with an example calculation included each batch?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Do all manual integrations have before/after intialed/dated/coded and 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: 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: D. Reed Date: 7/14/10

Data Quality Manager: Marcia Williams Date: 7/12/10

Section Supervisor: Betty DeVilb 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 Lloyd 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 CO₂ 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 \leq 2) with HCl or H₂SO₄. Both water and soil samples are stored at 4°C.

IV. INTERFERENCES

4.1 WATER METHOD

- 4.1.1 Removal of 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 CO₂ 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 ($\pm 2^\circ\text{C}$).

- 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 Instrument 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 μ L 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.

μ L 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 100 μ L of the 30% Sucrose standard with 500 μ L 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 (\pm 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 $\pm 20\%$ (Soil) or $\pm 10\%$ (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 $\pm 30\%$ (Soil) or $\pm 15\%$ (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 $\pm 20\%$ aqueous or $\pm 50\%$ soil/sediment
 - i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within $\pm 25\%$ aqueous or $\pm 50\%$ 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 eighth 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 are 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 lightning 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 correct 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 lightning bolt symbol then select "use settings on PC". Wait for initializing. When screen goes away 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, CH₃OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, NaHSO₄ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of NaHSO₄ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the NaHSO₄ 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 ± 0.01 g 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 MANAGEMENT AND 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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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-methylnaphthalene #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

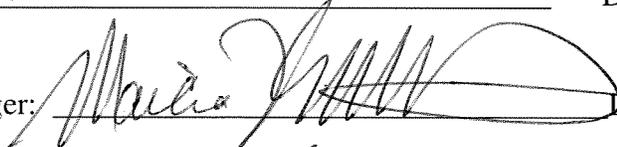
REVISION #: 17

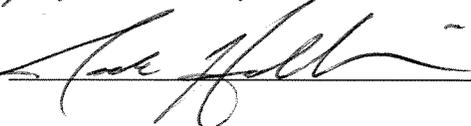
EFFECTIVE DATE: 042610

**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.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
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9. Equipment & Supplies
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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 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 ($\pm 5^{\circ}\text{C}$); 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 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 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 320

REVISION #: 10

EFFECTIVE DATE: 20100909

**TOTAL PETROLEUM HYDROCARBON (TPH) NON-AQUEOUS
MATRIX (LOW LEVEL) by USEPA SW-846 METHOD 8015B
LARGE SONICATION HORN**

APPROVALS:

Lab Director: _____  Date: 09/09/10

Data Quality Manager: _____  Date: 09/09/10

Section Supervisor: _____  Date: 09/09/10

Changes Summary

Revision 10, 09/09/10

- The SOP was updated to reflect references to LIMS, current SOPs, and reagents.

Revision 09, 08/29/10

- The SOP is an update from Revision 08 dated 09/24/08

**TOTAL PETROLEUM HYDROCARBONS (TPH)
NON-AQUEOUS MATRIX (LOW LEVEL) by
SW-846 Method 8015B and
Method from the Tennessee Division of Underground Storage Tanks,
Effective April 1, 1992**

I. SCOPE AND APPLICATION

1. This SOP describes the extraction of total petroleum hydrocarbons from soil by sonication extraction using SW846 Method 3550B.

II. SUMMARY

1. The samples are extracted with methylene chloride, dried with sodium sulfate, and concentrated.

III. INTERFERENCES

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.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes. Glassware should be solvent rinsed to avoid this problem.

IV. APPARATUS AND MATERIALS

- Beaker - 400 mL
- Fleaker Beaker - 1200 mL
- Funnel - glass
- Drying Column (Chromatographic column) - 20 mm I.D. x 300 mm
- Vial - 2 mL amber with Teflon-lined screw cap
- Syringe - 1 mL, 500 μ L
- Graduated cylinder - Glass, Class A, 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Ring stand - 3 prong
- Burette clamp - double
- Rings - large enough to hold KD setup
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator
- TurboVap Concentrator and concentrator tubes
- Balance - capable of weighing to 0.1 gram
- Sonicator - horn-type sonicator equipped with a 1/2 inch titanium tip
- Sonabox - sound proof box that will hold sonicator
- Filter paper - 15 inch Whatman No. 41 or equivalent

V. REAGENTS

- Sodium Sulfate (Na_2SO_4) - Granular, anhydrous, trace pure 10-60 mesh (purchased in a 2.5 kg glass amber jug from VWR - Baker #EM-SX0760E-3 or equivalent) placed in a Pyrex pan and heated at 400°C minimum of 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.

NOTE: We deviate from SW-846 protocol as follows: We do not cool our sodium sulfate in a desiccator. This deviation has not presented a problem.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 in a white plastic tub or equivalent).
- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.**)(Dichloromethane - Omnisolv - suitable for spectrophotometer and gas chromatography # DX0831-1 or equivalent)
- Acetone - suitable for spectrophotometer and gas chromatography (Omnisolv AX0116-1 or equivalent)
- TPH Surrogate - Surrogate (OTP)solution is prepared in acetone @ a concentration of $20\ \mu\text{g}/\text{mL}$. Use 1 mL per 25 grams of sample.
- TPH Spike - A spiking solution is prepared at a concentration of $1000\ \mu\text{g}/\text{mL}$ in acetone.

VI. PROCEDURE

1. Get samples from cooler. Inspect as to whether they are in glass jar and have a Teflon lid. Find out if any special dilutions need to be made for this client, or if the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a low level extraction is truly necessary.
2. Get out enough 400 mL beakers to extract the number of samples you have plus any additional spikes, laboratory control samples, and a method blank. A method blank must be processed with each set of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). Duplicate laboratory control samples should be processed for each batch up to a maximum of 20 samples which consist of weighing 25 grams of sodium sulfate and adding 1.0 mL of the TPH Spike to each. Rinse with methylene chloride. Make labels as follows on your Avery labels: Lab #.
3. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trash can.
4. 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 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

5. Place a 400 mL beaker on the balance and zero it. Using a spatula, transfer approximately 25 grams of sample to the nearest 0.1 gram. Record this amount on bench sheet in LIMS. Put your label on the side of the 400 mL beaker. Weigh up 3 beakers of the same sample for the spike and its duplicate.
6. Add approximately 50 grams of sodium sulfate to the 400 mL beaker. Cover the beaker with foil and continue to weigh up the remaining samples. For the method blank, simply weigh up 25 grams of sodium sulfate.
7. Using a spatula and/or a glass rod, mix each 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.
8. Using the 1.0 mL glass syringe marked TPH surrogate, add 1.0 mL of TPH surrogate to each sample, blank, laboratory control sample, and spike.

NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

9. For the TPH sample in each analytical batch selected for spiking, use a community 1 mL syringe rinsed well with methanol, acetone, and methylene chloride, and finally rinsed with acetone, to add 1.0 mL of the TPH Spike.

10. Add 50 mL of Methylene Chloride to each sample, method blank, laboratory control sample, and spike; or enough to cover the sediment with about ½” of solvent on top.
11. In the bench sheet in LIMS, enter the Client name, the Lab #, the date extracted, the 400 mL beaker #, the initial weight and the final volume and anything unusual that may have occurred with this sample.
13. Get a ring stand and attach two rings. Place a funnel in the top ring. Fold a piece of 15 inch filter paper in half and in half again. Open it up so that it makes a bowl and place into the funnel. Put a small amount of sodium sulfate into the bottom of the filter to help seat it in the funnel. Rinse the filter and sodium sulfate with methylene chloride. Seat funnel on top of Turbovap tube that has been rinsed with methylene chloride.
14. Using a paper towel dowsed in methylene chloride, clean the ultrasonic horn. Rinse the horn twice with methylene chloride into a waste beaker.

NOTE: The cleaning of the horn is probably one of most important steps in this procedure to prevent cross - contamination between the samples.
15. Place the sample to be extracted in the sonabox so that the bottom surface of the 3/4 inch sonicator is about 1/2 inch below the surface of the solvent, but above the sediment layer. Sonicate for 3 minutes with output control knob set at 10, turn mode switch on, set pulse and percent-duty cycle at 50%. Remove the sample from the box and decant the solvent onto the surface of the sodium sulfate in the funnel.
16. Repeat STEPS 14 and 15 two more times with two additional 50-60 mL portions of methylene chloride.
17. While waiting for the funnel to quit dripping, record the numbers of the concentrator tube on the bench sheet.
18. After the volume in the funnel has receded so that no obvious solvent is on top, depending on how quickly, the extract has drained through, you may wish to make up a new filter in the same manner as explained in STEP 12. Rinse the sample with 10 to 20 mL of methylene chloride and allow to drain. Discard the used filter in the trash and replace with the new one. Rinse the 400 mL sample beaker and its contents twice with 10 to 20 mL of methylene chloride, transferring this rinse to the funnel. When this has completely drained into the TV tube, rinse the funnel for the final time and allow it to drain. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.

TURBO-VAP CONCENTRATION

- Remove the tube to a metal holder. To help prevent cross contamination, place a piece of aluminum foil over the Turbo-Vap tube and punch a small hole in the top so that the nitrogen can be accessed.

- **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.
- 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).
- When the beep sounds indicating the end of concentration, the extract will be at approximately one mL (half way up tip of tube). Remove the tube from the bath. Hold the tube and the sample vial in one hand at about a 45° angle. 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.
- Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2-mL vial. Add methylene chloride 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. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

26. For sample analyses reference SOP-219.

VII. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-QS08 for guidance.

VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-QS14 for the 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.

IX. METHOD PERFORMANCE

Refer to SOP-219 for method performance.

X. HEALTH AND SAFETY

Refer to the MSDS sheets for the chemicals used for health and safety information.
Also see SOP-QS13 for proper use of methylene chloride.

XI. REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. TN Division of Underground Storage Tanks.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 322

REVISION #: 10

EFFECTIVE DATE: 20100909

**TOTAL PETROLEUM HYDROCARBONS (TPH) AQUEOUS
MATRIX by USEPA SW846 METHOD 8015B**

APPROVALS:

Lab Director:  Date: 09/09/10

Data Quality Manager:  Date: 09/09/10

Section Supervisor:  Date: 09/09/10

Changes Summary

Revision 10, 09/09/10

- The SOP was updated to reflect references to LIMS, current SOPs, and reagents.

Revision 09, 08/29/10

- The SOP is an update from Revision 08 dated 09/24/09.

**Total Petroleum Hydrocarbons (TPH) AQUEOUS MATRIX by
USEPA SW-846 Method 8015B
Method from the Tennessee Division of Underground Storage Tanks,
Effective April 1, 1992**

I. SCOPE AND APPLICATION

1. This SOP describes the extraction of total petroleum hydrocarbons from water by separatory funnel extraction using SW846 Method 3510C.

II. SUMMARY

1. The samples are extracted with methylene chloride, dried with sodium sulfate, and concentrated.

III. INTERFERENCES

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.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes. Glassware should be solvent rinsed to avoid this problem.

IV. APPARATUS AND MATERIALS

- Separatory Funnel - 2 Liter with Teflon stopcock
- Beaker - 400 mL
- Drying Column (Chromatographic column) - 20 mm I.D. x 300 mm
- Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator.
- Metal rack - capable of holding six glass evaporation tubes.
- Turbo-Vap Evaporator - heated and capable of temperature control (+5°C); the bath should be vented into a hood.
- Vials 10 mL glass, with Teflon-lined screw cap
- pH indicator paper - wide range (1.0 and 12.0)
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL and 100 mL
- Pasteur pipette - length 9"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Volumetrics - Class A, glass, 1000 mL and 500 mL
- Ring stand - 3 prong
- Burette clamp - double
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator

V. REAGENTS

- Reagent water - Reagent water is Modulab water gathered in a carboy from source in the wet chemistry lab.
- Sodium Sulfate (Na_2SO_4) - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in a 2.5 kg glass amber jug from VWR - Baker #EM-SX0760E-3 or equivalent) placed in a Pyrex pan and heated at 400°C minimum 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.

NOTE: We deviate from SW-846 protocol as follows: We do not cool our sodium sulfate in a desiccator. This deviation has not presented a problem.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 in a white plastic tub or equivalent).
- Extraction Solvent - Methylene Chloride (**Please see SOP-336 before handling this solvent in our laboratory.**) (Dichloromethane - Omnisolv - suitable for spectrophotometer and gas chromatography # DX0831-1 or equivalent)
- Methanol - suitable for use in gas chromatography (Omnisolv MX0484-1 or equivalent)
- Acetone - suitable for spectrophotometer and gas chromatography (Omnisolv AX0116-1 or equivalent)
- TPH Surrogate - Surrogate (OTP) solution is prepared in acetone at a concentration of $20\ \mu\text{g/mL}$. Use 1 mL per 1000 mL of sample.
- TPH Spike - A spiking solution is prepared at a concentration of $1000\ \mu\text{g/mL}$ in acetone. Use 1 mL per 1000 mL of sample.

VI. PROCEDURE

1. Get samples from cooler. 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, if the sample is a SLUDGE (use only 100 mL and dilute to 1000 mL with reagent water), or if the sample has a particularly bad matrix, see your supervisor to find out what dilution, if any, should be made.
2. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume.
3. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes, laboratory control samples, and a method blank. A method blank must be processed with each set of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In addition, duplicate laboratory control samples which contain 1 mL of the TPH Spike each in a blank matrix are to be extracted for each batch up to 20 samples.

Rinse with methylene chloride. Label the separatory funnels as follows on your Avery labels: Lab #.

4. Using the 1000 mL glass graduated cylinder, measure 1 liter of reagent water from the carboy and transfer it to a separatory funnel for the Method Blank. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Add 60 mL of Methylene Chloride to the empty sample bottles.
5. Using the 1.0 mL glass syringe marked TPH surrogate, add 1.0 mL of TPH surrogate to each sample, blank, lab control sample, and spike.

NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

6. For the TPH sample in each analytical batch selected for spiking, use a community 1 mL syringe rinsed well with methanol, acetone, and methylene chloride, and finally rinsed with acetone to add 1.0 mL of the TPH spike.

NOTE: Due to limited volume received, usually it is necessary to use half a liter to do a spike so that a spike duplicate can be extracted also. If only one liter is provided for spiking purposes, use a 500 mL glass cylinder to measure out half the sample. Transfer to a separatory funnel labeled for the Spike. Measure the remaining sample and transfer to a separatory funnel labeled Spike Duplicate. Add 1/2 the normal amount of spiking solution and 1/2 the normal amount of surrogate.

7. Stopper funnel, swirl, and invert so the glass stopper gets wet. Using wide-range pH paper, check pH of sample by touching pH - sensitive paper to the drop of liquid hanging from the glass stopper. Record this initial pH on the bench sheet in LIMS if it is anything other than 2 or less. Adjust the pH to less than 2 and notify your supervisor.
8. Swirl the 60 mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel. Using a 100 mL glass cylinder, add 60 mL of methylene chloride to the Method Blank. If using a liter, add 60 mL to the Spikes.
9. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

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.

10. Allow the sample to set for 10 minutes 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 50 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400 mL glass beaker.

11. Repeat STEPS 8 through 10 one more time, only this time you may take the methylene chloride bottle directly to the separatory funnel and pump it twice. This should be 60 mL as it is set on 30 mL. Combine the two solvent extracts into the same 400 mL beaker.
12. On the bench sheet for the batch, enter anything unusual that may have occurred with this sample.
13. 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 orange holders are available for the 250 mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle with similar weight using reagent water. Set the rpm at 2500 and the temperature at 25°C. Close the lid and be sure to press it down until you hear it lock. 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. Remove any water layer from the extract in the beaker or centrifuge bottle, with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer. Use the smallest amount possible of Na₂SO₄ by sprinkling the top layer with Na₂SO₄ until it hardens, separates, and drops to the bottom.

TURBO-VAP CONCENTRATION

15. Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed funnel. Pour the extract through the funnel. It will collect in the tube. Rinse the 400 mL beaker twice with 10 mL of methylene chloride. The Turbo-Vap tubes only hold 200 mL. Rinse the funnel with 10 - 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 180 mL to avoid splattering on the lid of the Turbo-Vap. While waiting for the funnel to quit dripping, record the numbers of the Turbo-Vap tube on the batch bench sheet and remove the tube to a metal holder.
16. Turbo-Vap Operation: Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. Adjust the settings of the Turbo-Vap so that the solvent exchange is at "0" and dryness is at "NO". Push the reset button with the lid up to make the indicator light go to green at each individual station. Temperature of the bath should be at 45°C ± 1.0°C. **Higher temperature will result in lost of low boiling point analytes.**
17. 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).

18. When the beep sounds indicating the end of concentration, the extract will be at approximately one mL. Remove the tube from the bath. Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" Pasteur pipette to draw up the 1.0 mL 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.
19. Add ≈0.25 mL of Meclz to the tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 2 mL vial. **Using a 2.0 mL vial filled to exactly 1.0 mL w/Meclz measure the actual volume of the sample. Adjust the volume to exactly 1.0 mL with methylene chloride or if further concentration is required use nitrogen blow down. Repeat the measurement with the 1.0 mL vial until an exact 1.0 mL sample volume is obtained.** Cover the extract with a Teflon-sealed screw cap. The extract obtained above may now be analyzed. Refrigerate at 4°C.
20. 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 bench sheet to the nearest 10 mL. Record this information on the bench sheet in LIMS.
21. For sample analyses reference SOP-219.

VII. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-QS08 for guidance.

VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-QS14 for the 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.

IX. METHOD PERFORMANCE

Refer to SOP-219 for method performance.

X. HEALTH AND SAFETY

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-QS13 for proper use of methylene chloride.

XI. REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. TN Division of Underground Storage Tanks.

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 (Na_2SO_4) – 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 spectrophotometry 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-metaxylene/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.

- 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.
- Quantitatively transfer samples to microwave tubes. Make sure samples are loaded in the rack in the order of the bench sheet.
- 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.
- 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.
- 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 P4 filter 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.

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

QUALITY SYSTEMS: QS10

REVISION #: 14 EFFECTIVE DATE: 20100907

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

APPROVALS:

Lab Director: RED Date: 9/8/10

Data Quality Manager: Marcia MA Date: 9/8/10

Section Supervisor: Whiffel 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 HNO₃ 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 HNO₃ 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 H₂SO₄ (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 Na₂S₂O₃ (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 or 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 (0811248-06).
 - 5.1.22.2 A 2-digit numeral of the month. Example (0811248-06).
 - 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 (0811248-06).
 - 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 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)

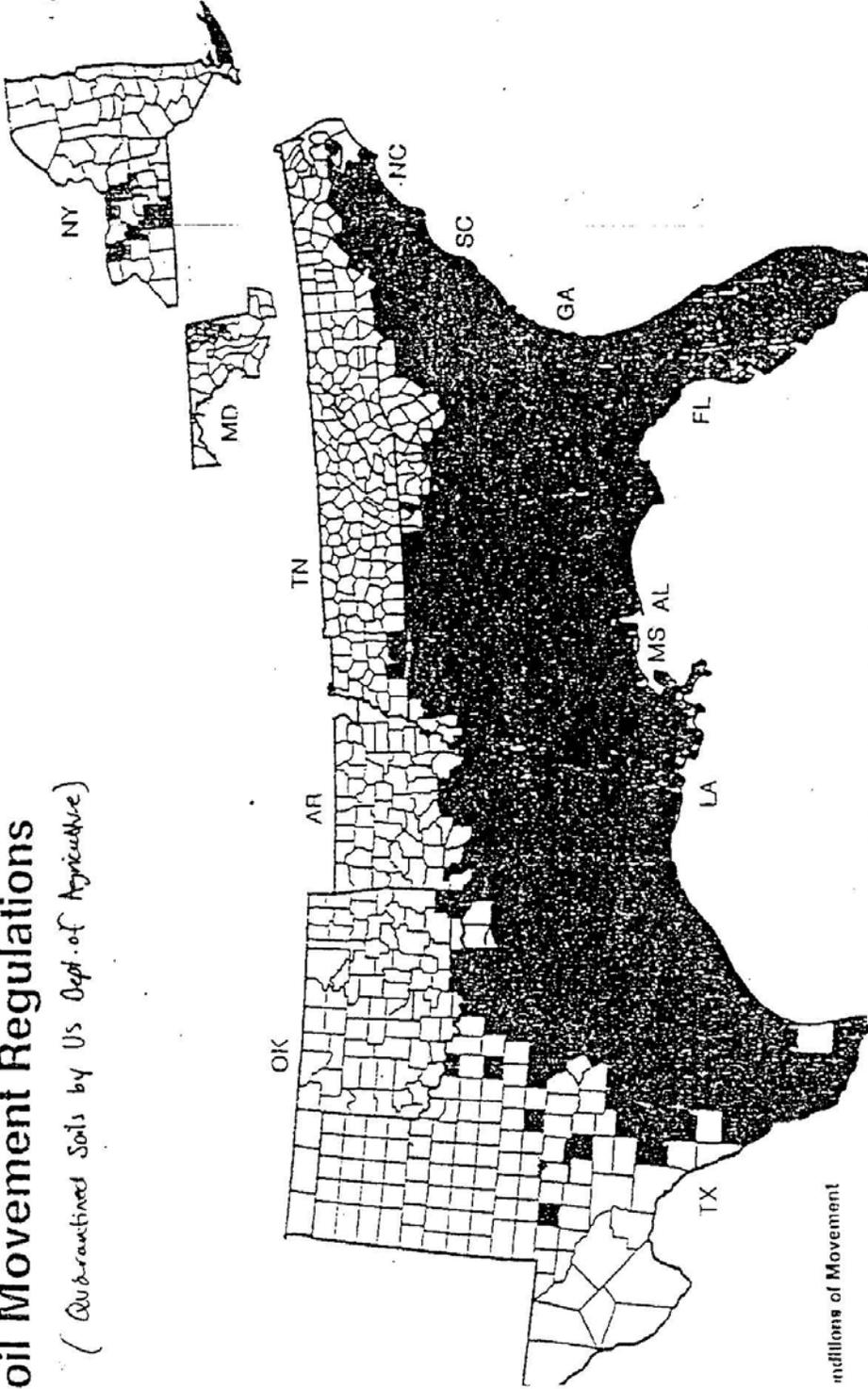
Parameter	Holding Time
pH	Immediate ^a
Sulfite	Immediate ^a
Temperature	Immediate ^a
Residual Chlorine	Immediate ^a
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) <i>Drinking Water only</i>	30 hours
BOD	48 hours
Color	48 hours
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.

oil Movement Regulations

(Constrained Soils by US Dept. of Agriculture)



millions of Movement

Restrictions are imposed on the movement of regulated articles from a regulated area into or through within regulated areas within red areas may be updated.

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 X

Preservatives		Types of Container	
NI	<i>HNO₃</i>	A	<i>1 LITER - PLASTIC</i>
NF	<i>HNO₃ (Filtered)</i>	B	<i>500 mL - PLASTIC</i>
SU	<i>H₂SO₄</i>	C	<i>250 mL - PLASTIC</i>
SH	<i>NaOH</i>	D	<i>120 mL - PLASTIC</i>
ZN	<i>ZnAC / NaOH</i>	EN	<i>ENCORE PAK</i>
HY	<i>HCl</i>	F	<i>1 LITER - GLASS CLEAR WIDE MOUTH</i>
		G	<i>1 LITER - GLASS CLEAR BOSTON ROUND</i>
		H	<i>1 LITER - GLASS AMBER</i>
		I	<i>250 ml. - AMBER</i>
		J	<i>VOA VIALS - (40 ml.)</i>
		K	<i>500 ml. - (16 oz)</i>
		L	<i>250 ml. - (8 oz)</i>
		M	<i>125 ml. - (4 oz)</i>
		N	<i>60 ml. - (2 oz)</i>
		O	<i>OTHER</i>
		P	<i>PLASTIC BAG -1 Gallon</i>

Attachment XIII

SAMPLE LOG CHANGE FORM

DATE:

Workorder:

CLIENT:

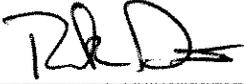
PARAMETERS:

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

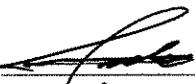
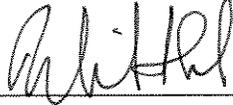
QUALITY SYSTEMS: QS14 REVISION #: 06 EFFECTIVE DATE: 20100831

ANALYTICAL LABORATORY WASTE DISPOSAL

APPROVALS:

Lab Director:  Date: 9/8/10

Data Quality Manager:  Date: 9/8/10

Section Supervisor:   Date: 9/8/10
mm
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.



**LABORATORY
ACCREDITATION
BUREAU**

Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2236

TestAmerica Laboratories, Inc

5755 8th Street East
Tacoma, WA 98424

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: January 19, 2013

**R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 19th of January 2010**

*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).

Scope of Accreditation For TestAmerica Laboratories, Inc.

5755 8th Street East
Tacoma, WA 98424
Dave Wunderlich
1-253-922-2310

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to TestAmerica Laboratories, Inc. to perform the following tests:

Accreditation granted through: January 19, 2013

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010B/200.7	Silver
ICP-AES	EPA 6010B/200.7	Aluminum
ICP-AES	EPA 6010B/200.7	Arsenic
ICP-AES	EPA 6010B/200.7	Boron
ICP-AES	EPA 6010B/200.7	Barium
ICP-AES	EPA 6010B/200.7	Beryllium
ICP-AES	EPA 6010B/200.7	Calcium
ICP-AES	EPA 6010B/200.7	Cadmium
ICP-AES	EPA 6010B/200.7	Cobalt
ICP-AES	EPA 6010B/200.7	Chromium
ICP-AES	EPA 6010B/200.7	Copper
ICP-AES	EPA 6010B/200.7	Iron
ICP-AES	EPA 6010B/200.7	Potassium
ICP-AES	EPA 6010B/200.7	Magnesium
ICP-AES	EPA 6010B/200.7	Manganese
ICP-AES	EPA 6010B/200.7	Molybdenum
ICP-AES	EPA 6010B/200.7	Sodium
ICP-AES	EPA 6010B/200.7	Nickel
ICP-AES	EPA 6010B/200.7	Lead
ICP-AES	EPA 6010B/200.7	Antimony
ICP-AES	EPA 6010B/200.7	Selenium

Non-Potable Water		
Technology	Method	Analyte
ICP-AES	EPA 6010B/200.7	Silicon
ICP-AES	EPA 6010B/200.7	Tin
ICP-AES	EPA 6010B/200.7	Titanium
ICP-AES	EPA 6010B/200.7	Strontium
ICP-AES	EPA 6010B/200.7	Thallium
ICP-AES	EPA 6010B/200.7	Vanadium
ICP-AES	EPA 6010B/200.7	Zinc
ICP-MS	EPA 6020/200.8	Silver
ICP-MS	EPA 6020/200.8	Arsenic
ICP-MS	EPA 6020/200.8	Barium
ICP-MS	EPA 6020/200.8	Beryllium
ICP-MS	EPA 6020/200.8	Cadmium
ICP-MS	EPA 6020/200.8	Cobalt
ICP-MS	EPA 6020/200.8	Chromium
ICP-MS	EPA 6020/200.8	Copper
ICP-MS	EPA 6020/200.8	Manganese
ICP-MS	EPA 6020/200.8	Molybdenum
ICP-MS	EPA 6020/200.8	Nickel
ICP-MS	EPA 6020/200.8	Lead
ICP-MS	EPA 6020/200.8	Antimony
ICP-MS	EPA 6020/200.8	Selenium
ICP-MS	EPA 6020/200.8	Thallium
ICP-MS	EPA 6020/200.8	Uranium
ICP-MS	EPA 6020/200.8	Vanadium
ICP-MS	EPA 6020/200.8	Zinc
CVAAS	EPA 7470A/245.1	Mercury
ICP-AES	EPA 7195/6010B	Hexavalent Chromium
GC/MS	EPA 8260B/624	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/624	1,1,1-Trichloroethane
GC/MS	EPA 8260B/624	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/624	1,1,2-Trichloroethane
GC/MS	EPA 8260B/624	1,1-Dichloroethane
GC/MS	EPA 8260B/624	1,1-Dichloroethene
GC/MS	EPA 8260B/624	1,1-Dichloropropene
GC/MS	EPA 8260B/624	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/624	1,2,3-Trichloropropane
GC/MS	EPA 8260B/624	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/624	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/624	1,2-Dibromo-3-Chloropropane
GC/MS	EPA 8260B/624	1,2-Dichlorobenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B/624	1,2-Dichloroethane
GC/MS	EPA 8260B/624	1,2-Dichloropropane
GC/MS	EPA 8260B/624	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/624	1,3-Dichlorobenzene
GC/MS	EPA 8260B/624	1,3-Dichloropropane
GC/MS	EPA 8260B/624	1,4-Dichlorobenzene
GC/MS	EPA 8260B/624	2,2-Dichloropropane
GC/MS	EPA 8260B/624	2-Chlorotoluene
GC/MS	EPA 8260B/624	2-Hexanone
GC/MS	EPA 8260B/624	4-Chlorotoluene
GC/MS	EPA 8260B/624	4-Isopropyltoluene
GC/MS	EPA 8260B/624	Acetone
GC/MS	EPA 8260B/624	Benzene
GC/MS	EPA 8260B/624	Bromobenzene
GC/MS	EPA 8260B/624	Bromodichloromethane
GC/MS	EPA 8260B/624	Bromoform
GC/MS	EPA 8260B/624	Bromomethane
GC/MS	EPA 8260B/624	Carbon disulfide
GC/MS	EPA 8260B/624	Carbon tetrachloride
GC/MS	EPA 8260B/624	Chlorobenzene
GC/MS	EPA 8260B/624	Chlorobromomethane
GC/MS	EPA 8260B/624	Chlorodibromomethane
GC/MS	EPA 8260B/624	Chloroethane
GC/MS	EPA 8260B/624	Chloroform
GC/MS	EPA 8260B/624	Chloromethane
GC/MS	EPA 8260B/624	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/624	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/624	Dibromomethane
GC/MS	EPA 8260B/624	Dichlorodifluoromethane
GC/MS	EPA 8260B/624	Ethylbenzene
GC/MS	EPA 8260B/624	Ethylene Dibromide
GC/MS	EPA 8260B/624	Hexachlorobutadiene
GC/MS	EPA 8260B/624	Isopropylbenzene
GC/MS	EPA 8260B/624	Methyl Ethyl Ketone
GC/MS	EPA 8260B/624	Methyl Isobutyl Ketone
GC/MS	EPA 8260B/624	Methyl tert-butyl ether
GC/MS	EPA 8260B/624	Methylene Chloride
GC/MS	EPA 8260B/624	m-Xylene & p-Xylene
GC/MS	EPA 8260B/624	Naphthalene
GC/MS	EPA 8260B/624	n-Butylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B/624	N-Propylbenzene
GC/MS	EPA 8260B/624	o-Xylene
GC/MS	EPA 8260B/624	sec-Butylbenzene
GC/MS	EPA 8260B/624	Styrene
GC/MS	EPA 8260B/624	tert-Butylbenzene
GC/MS	EPA 8260B/624	Tetrachloroethene
GC/MS	EPA 8260B/624	Toluene
GC/MS	EPA 8260B/624	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/624	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/624	Trichloroethene
GC/MS	EPA 8260B/624	Trichlorofluoromethane
GC/MS	EPA 8260B/624	Vinyl chloride
GC/MS	EPA 8270C/625	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/625	1,2-Dichlorobenzene
GC/MS	EPA 8270C/625	1,3-Dichlorobenzene
GC/MS	EPA 8270C/625	1,4-Dichlorobenzene
GC/MS	EPA 8270C/625	bis(2-chloroisopropyl)ether
GC/MS	EPA 8270C/625	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/625	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/625	2,4-Dichlorophenol
GC/MS	EPA 8270C/625	2,4-Dimethylphenol
GC/MS	EPA 8270C/625	2,4-Dinitrophenol
GC/MS	EPA 8270C/625	2,4-Dinitrotoluene
GC/MS	EPA 8270C/625	2,6-Dinitrotoluene
GC/MS	EPA 8270C/625	2-Chloronaphthalene
GC/MS	EPA 8270C/625	2-Chlorophenol
GC/MS	EPA 8270C/625	2-Methylnaphthalene
GC/MS	EPA 8270C/625	2-Methylphenol
GC/MS	EPA 8270C/625	2-Nitroaniline
GC/MS	EPA 8270C/625	2-Nitrophenol
GC/MS	EPA 8270C/625	3 & 4 Methylphenol
GC/MS	EPA 8270C/625	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/625	3-Nitroaniline
GC/MS	EPA 8270C/625	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/625	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/625	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/625	4-Chloroaniline
GC/MS	EPA 8270C/625	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/625	4-Nitroaniline
GC/MS	EPA 8270C/625	Acenaphthene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/625	Acenaphthylene
GC/MS	EPA 8270C/625	Anthracene
GC/MS	EPA 8270C/625	1,2-Diphenylhydrazine as Azobenzene
GC/MS	EPA 8270C/625	Benzo[a]anthracene
GC/MS	EPA 8270C/625	Benzo[a]pyrene
GC/MS	EPA 8270C/625	Benzo[b]fluoranthene
GC/MS	EPA 8270C/625	Benzo[g,h,i]perylene
GC/MS	EPA 8270C/625	Benzo[k]fluoranthene
GC/MS	EPA 8270C/625	Benzoic acid
GC/MS	EPA 8270C/625	Benzyl alcohol
GC/MS	EPA 8270C/625	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/625	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/625	Bis(2-ethylhexyl) phthalate
GC/MS	EPA 8270C/625	Butyl benzyl phthalate
GC/MS	EPA 8270C/625	Carbazole
GC/MS	EPA 8270C/625	Chrysene
GC/MS	EPA 8270C/625	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/625	Dibenzofuran
GC/MS	EPA 8270C/625	Diethyl phthalate
GC/MS	EPA 8270C/625	Dimethyl phthalate
GC/MS	EPA 8270C/625	Di-n-butyl phthalate
GC/MS	EPA 8270C/625	Di-n-octyl phthalate
GC/MS	EPA 8270C/625	Fluoranthene
GC/MS	EPA 8270C/625	Fluorene
GC/MS	EPA 8270C/625	Hexachlorobenzene
GC/MS	EPA 8270C/625	Hexachlorobutadiene
GC/MS	EPA 8270C/625	Hexachloroethane
GC/MS	EPA 8270C/625	Indeno[1,2,3-cd]pyrene
GC/MS	EPA 8270C/625	Isophorone
GC/MS	EPA 8270C/625	Naphthalene
GC/MS	EPA 8270C/625	Nitrobenzene
GC/MS	EPA 8270C/625	N-Nitrosodimethylamine
GC/MS	EPA 8270C/625	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/625	N-Nitrosodiphenylamine
GC/MS	EPA 8270C/625	Pentachlorophenol
GC/MS	EPA 8270C/625	Phenanthrene
GC/MS	EPA 8270C/625	Phenol
GC/MS	EPA 8270C/625	Pyrene
GC/MS SIM	EPA 8270C SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM	Acenaphthene

Non-Potable Water		
Technology	Method	Analyte
GC/MS SIM	EPA 8270C SIM	Acenaphthylene
GC/MS SIM	EPA 8270C SIM	Anthracene
GC/MS SIM	EPA 8270C SIM	Benzo[a]anthracene
GC/MS SIM	EPA 8270C SIM	Benzo[a]pyrene
GC/MS SIM	EPA 8270C SIM	Benzo[b]fluoranthene
GC/MS SIM	EPA 8270C SIM	Benzo[g,h,i]perylene
GC/MS SIM	EPA 8270C SIM	Benzo[k]fluoranthene
GC/MS SIM	EPA 8270C SIM	Chrysene
GC/MS SIM	EPA 8270C SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C SIM	Fluoranthene
GC/MS SIM	EPA 8270C SIM	Fluorene
GC/MS SIM	EPA 8270C SIM	Indeno[1,2,3-cd]pyrene
GC/MS SIM	EPA 8270C SIM	Naphthalene
GC/MS SIM	EPA 8270C SIM	Phenanthrene
GC/MS SIM	EPA 8270C SIM	Pyrene
GC-ECD	EPA 8011/504.1	1,2-Dibromoethane
GC-ECD	EPA 8011/504.1	1,2-Dibromo-3-Chloropropane
GC-ECD	EPA 8081A/608	4,4'-DDD
GC-ECD	EPA 8081A/608	4,4'-DDE
GC-ECD	EPA 8081A/608	4,4'-DDT
GC-ECD	EPA 8081A/608	Aldrin
GC-ECD	EPA 8081A/608	alpha-BHC
GC-ECD	EPA 8081A/608	alpha-Chlordane
GC-ECD	EPA 8081A/608	beta-BHC
GC-ECD	EPA 8081A/608	delta-BHC
GC-ECD	EPA 8081A/608	Dieldrin
GC-ECD	EPA 8081A/608	Endosulfan I
GC-ECD	EPA 8081A/608	Endosulfan II
GC-ECD	EPA 8081A/608	Endosulfan sulfate
GC-ECD	EPA 8081A/608	Endrin
GC-ECD	EPA 8081A/608	Endrin aldehyde
GC-ECD	EPA 8081A/608	Endrin ketone
GC-ECD	EPA 8081A/608	gamma-BHC (Lindane)
GC-ECD	EPA 8081A/608	gamma-Chlordane
GC-ECD	EPA 8081A/608	Heptachlor
GC-ECD	EPA 8081A/608	Heptachlor epoxide
GC-ECD	EPA 8081A/608	Methoxychlor
GC-ECD	EPA 8081A/608	Technical Chlordane
GC-ECD	EPA 8081A/608	Toxaphene
GC-ECD	EPA 8082/608	PCB-1016

Non-Potable Water		
Technology	Method	Analyte
GC-ECD	EPA 8082/608	PCB-1221
GC-ECD	EPA 8082/608	PCB-1232
GC-ECD	EPA 8082/608	PCB-1242
GC-ECD	EPA 8082/608	PCB-1248
GC-ECD	EPA 8082/608	PCB-1254
GC-ECD	EPA 8082/608	PCB-1260
GC-IT/MS	EPA 8151A mod.	2,4,5-T
GC-IT/MS	EPA 8151A mod.	2,4-D
GC-IT/MS	EPA 8151A mod.	2,4-DB
GC-IT/MS	EPA 8151A mod.	4-Nitrophenol
GC-IT/MS	EPA 8151A mod.	Dalapon
GC-IT/MS	EPA 8151A mod.	Dicamba
GC-IT/MS	EPA 8151A mod.	Dichlorprop
GC-IT/MS	EPA 8151A mod.	Dinoseb
GC-IT/MS	EPA 8151A mod.	MCPA
GC-IT/MS	EPA 8151A mod.	Mecoprop
GC-IT/MS	EPA 8151A mod.	Pentachlorophenol
GC-IT/MS	EPA 8151A mod.	Silvex (2,4,5-TP)
GC-FID	EPA 8015B/AK101/ NWTPH-Gx/NWVPH	Gasoline and Volatile Petroleum Hydrocarbons
GC-FID	EPA 8015B/AK102/ NWTPH-Dx/NWEPH	Diesel and Extractable Petroleum Hydrocarbons
GC-FID	EPA 8015B/AK103/ NWTPH-Dx/NWEPH	Motor Oil and Extractable Petroleum Hydrocarbons
Gravimetric	EPA 1664A	Oil & Grease
Colorimetric/RFA	9012A	Total Cyanides
Ion Chromatography	EPA 300.0/9056A	Bromide
Ion Chromatography	EPA 300.0/9056A	Chloride
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Sulfate
Ion Chromatography	EPA 300.0/9056A	Nitrate
Ion Chromatography	EPA 300.0/9056A	Nitrite
TOC Analyzer (IR)	EPA 415.1/9060	TOC
Probe	EPA 9040/9045/150.1	pH
Conductivity meter	EPA 9050/120.1 SM2510B	Specific Conductance
Pensky-Martens closed-cup tester/ Setaflash	EPA 1010/1020	Ignitability/Flashpoint
Preparation	Method	Type
Separatory Funnel Liquid- Liquid Extraction	EPA 3510C	Semivolatile and Nonvolatile Organics

Non-Potable Water		
Preparation	Method	Type
Continuous Liquid-Liquid Extraction	EPA 3520	Semivolatile and Nonvolatile Organics
Solvent Dilution	EPA 3580	Semivolatile and Nonvolatile Organics
Waste Dilution	EPA 3585	Volatile Organic Compounds
Purge and Trap	EPA 5030	Volatile Organic Compounds
Purge and Trap	EPA 5035	Volatile Organic Compounds
Acid Digestion (Aqueous)	EPA 3005/3010	Inorganics
Acid Digestion (Sediments, Sludges, and Soils)	EPA 3050	Inorganics
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure
Florisol Cleanup	EPA 3620B	Cleanup of pesticide residues and other chlorinated hydrocarbons
Silica Gel Cleanup	EPA 3630C	Column Cleanup
Gel Permeation Cleanup	EPA 3640A	Separation of Synthetic Macromolecules
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup Reagent
Sulfuric Acid Cleanup	EPA 3665A	Cleanup for Quantitation of PCBs
Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010B	Silver
ICP-AES	EPA 6010B	Aluminum
ICP-AES	EPA 6010B	Arsenic
ICP-AES	EPA 6010B	Boron
ICP-AES	EPA 6010B	Barium
ICP-AES	EPA 6010B	Beryllium
ICP-AES	EPA 6010B	Calcium
ICP-AES	EPA 6010B	Cadmium
ICP-AES	EPA 6010B	Cobalt
ICP-AES	EPA 6010B	Chromium
ICP-AES	EPA 6010B	Copper
ICP-AES	EPA 6010B	Iron
ICP-AES	EPA 6010B	Potassium
ICP-AES	EPA 6010B	Magnesium
ICP-AES	EPA 6010B	Manganese
ICP-AES	EPA 6010B	Molybdenum
ICP-AES	EPA 6010B	Sodium
ICP-AES	EPA 6010B	Nickel
ICP-AES	EPA 6010B	Lead
ICP-AES	EPA 6010B	Antimony
ICP-AES	EPA 6010B	Selenium

Solid and Chemical Materials		
Technology	Method	Analyte
ICP-AES	EPA 6010B	Silicon
ICP-AES	EPA 6010B	Tin
ICP-AES	EPA 6010B	Titanium
ICP-AES	EPA 6010B	Strontium
ICP-AES	EPA 6010B	Thallium
ICP-AES	EPA 6010B	Vanadium
ICP-AES	EPA 6010B	Zinc
ICP-MS	EPA 6020	Silver
ICP-MS	EPA 6020	Arsenic
ICP-MS	EPA 6020	Barium
ICP-MS	EPA 6020	Beryllium
ICP-MS	EPA 6020	Cadmium
ICP-MS	EPA 6020	Cobalt
ICP-MS	EPA 6020	Chromium
ICP-MS	EPA 6020	Copper
ICP-MS	EPA 6020	Iron
ICP-MS	EPA 6020	Manganese
ICP-MS	EPA 6020	Molybdenum
ICP-MS	EPA 6020	Nickel
ICP-MS	EPA 6020	Lead
ICP-MS	EPA 6020	Antimony
ICP-MS	EPA 6020	Selenium
ICP-MS	EPA 6020	Thallium
ICP-MS	EPA 6020	Uranium
ICP-MS	EPA 6020	Vanadium
ICP-MS	EPA 6020	Zinc
CVAAS	EPA 7471A	Mercury
ICP-AES	EPA 7195/6010B	Hexavalent Chromium
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethene
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,2-Dibromo-3-Chloropropane
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Isopropyltoluene
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon disulfide
GC/MS	EPA 8260B	Carbon tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chlorodibromomethane
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Ethylene Dibromide
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Isopropylbenzene
GC/MS	EPA 8260B	Methyl Ethyl Ketone
GC/MS	EPA 8260B	Methyl Isobutyl Ketone
GC/MS	EPA 8260B	Methyl tert-butyl ether
GC/MS	EPA 8260B	Methylene Chloride
GC/MS	EPA 8260B	m-Xylene & p-Xylene
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	N-Propylbenzene
GC/MS	EPA 8260B	o-Xylene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene
GC/MS	EPA 8260B	Trichlorofluoromethane
GC/MS	EPA 8260B	Vinyl chloride
GC/MS	EPA 8270C	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C	1,2-Dichlorobenzene
GC/MS	EPA 8270C	1,3-Dichlorobenzene
GC/MS	EPA 8270C	1,4-Dichlorobenzene
GC/MS	EPA 8270C	bis(2-chloroisopropyl)ether
GC/MS	EPA 8270C	2,4,5-Trichlorophenol
GC/MS	EPA 8270C	2,4,6-Trichlorophenol
GC/MS	EPA 8270C	2,4-Dichlorophenol
GC/MS	EPA 8270C	2,4-Dimethylphenol
GC/MS	EPA 8270C	2,4-Dinitrophenol
GC/MS	EPA 8270C	2,4-Dinitrotoluene
GC/MS	EPA 8270C	2,6-Dinitrotoluene
GC/MS	EPA 8270C	2-Chloronaphthalene
GC/MS	EPA 8270C	2-Chlorophenol
GC/MS	EPA 8270C	2-Methylnaphthalene
GC/MS	EPA 8270C	2-Methylphenol
GC/MS	EPA 8270C	2-Nitroaniline
GC/MS	EPA 8270C	2-Nitrophenol
GC/MS	EPA 8270C	3 & 4 Methylphenol
GC/MS	EPA 8270C	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C	3-Nitroaniline
GC/MS	EPA 8270C	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C	4-Chloro-3-methylphenol
GC/MS	EPA 8270C	4-Chloroaniline
GC/MS	EPA 8270C	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C	4-Nitroaniline
GC/MS	EPA 8270C	Acenaphthene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C	Acenaphthylene
GC/MS	EPA 8270C	Anthracene
GC/MS	EPA 8270C	1,2-Diphenylhydrazine as Azobenzene
GC/MS	EPA 8270C	Benzo[a]anthracene
GC/MS	EPA 8270C	Benzo[a]pyrene
GC/MS	EPA 8270C	Benzo[b]fluoranthene
GC/MS	EPA 8270C	Benzo[g,h,i]perylene
GC/MS	EPA 8270C	Benzo[k]fluoranthene
GC/MS	EPA 8270C	Benzoic acid
GC/MS	EPA 8270C	Benzyl alcohol
GC/MS	EPA 8270C	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C	Bis(2-ethylhexyl) phthalate
GC/MS	EPA 8270C	Butyl benzyl phthalate
GC/MS	EPA 8270C	Carbazole
GC/MS	EPA 8270C	Chrysene
GC/MS	EPA 8270C	Dibenz(a,h)anthracene
GC/MS	EPA 8270C	Dibenzofuran
GC/MS	EPA 8270C	Diethyl phthalate
GC/MS	EPA 8270C	Dimethyl phthalate
GC/MS	EPA 8270C	Di-n-butyl phthalate
GC/MS	EPA 8270C	Di-n-octyl phthalate
GC/MS	EPA 8270C	Fluoranthene
GC/MS	EPA 8270C	Fluorene
GC/MS	EPA 8270C	Hexachlorobenzene
GC/MS	EPA 8270C	Hexachlorobutadiene
GC/MS	EPA 8270C	Hexachloroethane
GC/MS	EPA 8270C	Indeno[1,2,3-cd]pyrene
GC/MS	EPA 8270C	Isophorone
GC/MS	EPA 8270C	Naphthalene
GC/MS	EPA 8270C	Nitrobenzene
GC/MS	EPA 8270C	N-Nitrosodimethylamine
GC/MS	EPA 8270C	N-Nitrosodi-n-propylamine
GC/MS	EPA 8270C	N-Nitrosodiphenylamine
GC/MS	EPA 8270C	Pentachlorophenol
GC/MS	EPA 8270C	Phenanthrene
GC/MS	EPA 8270C	Phenol
GC/MS	EPA 8270C	Pyrene
GC/MS SIM	EPA 8270C SIM	2-Methylnaphthalene
GC/MS SIM	EPA 8270C SIM	Acenaphthene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS SIM	EPA 8270C SIM	Acenaphthylene
GC/MS SIM	EPA 8270C SIM	Anthracene
GC/MS SIM	EPA 8270C SIM	Benzo[a]anthracene
GC/MS SIM	EPA 8270C SIM	Benzo[a]pyrene
GC/MS SIM	EPA 8270C SIM	Benzo[b]fluoranthene
GC/MS SIM	EPA 8270C SIM	Benzo[g,h,i]perylene
GC/MS SIM	EPA 8270C SIM	Benzo[k]fluoranthene
GC/MS SIM	EPA 8270C SIM	Chrysene
GC/MS SIM	EPA 8270C SIM	Dibenz(a,h)anthracene
GC/MS SIM	EPA 8270C SIM	Fluoranthene
GC/MS SIM	EPA 8270C SIM	Fluorene
GC/MS SIM	EPA 8270C SIM	Indeno[1,2,3-cd]pyrene
GC/MS SIM	EPA 8270C SIM	Naphthalene
GC/MS SIM	EPA 8270C SIM	Phenanthrene
GC/MS SIM	EPA 8270C SIM	Pyrene
GC-ECD	EPA 8081A	4,4'-DDD
GC-ECD	EPA 8081A	4,4'-DDE
GC-ECD	EPA 8081A	4,4'-DDT
GC-ECD	EPA 8081A	Aldrin
GC-ECD	EPA 8081A	alpha-BHC
GC-ECD	EPA 8081A	alpha-Chlordane
GC-ECD	EPA 8081A	beta-BHC
GC-ECD	EPA 8081A	delta-BHC
GC-ECD	EPA 8081A	Dieldrin
GC-ECD	EPA 8081A	Endosulfan I
GC-ECD	EPA 8081A	Endosulfan II
GC-ECD	EPA 8081A	Endosulfan sulfate
GC-ECD	EPA 8081A	Endrin
GC-ECD	EPA 8081A	Endrin aldehyde
GC-ECD	EPA 8081A	Endrin ketone
GC-ECD	EPA 8081A	gamma-BHC (Lindane)
GC-ECD	EPA 8081A	gamma-Chlordane
GC-ECD	EPA 8081A	Heptachlor
GC-ECD	EPA 8081A	Heptachlor epoxide
GC-ECD	EPA 8081A	Methoxychlor
GC-ECD	EPA 8081A	Technical Chlordane
GC-ECD	EPA 8081A	Toxaphene
GC-ECD	EPA 8082	PCB-1016
GC-ECD	EPA 8082	PCB-1221
GC-ECD	EPA 8082	PCB-1232

Solid and Chemical Materials		
Technology	Method	Analyte
GC-ECD	EPA 8082	PCB-1242
GC-ECD	EPA 8082	PCB-1248
GC-ECD	EPA 8082	PCB-1254
GC-ECD	EPA 8082	PCB-1260
GC-IT/MS	EPA 8151A mod.	2,4,5-T
GC-IT/MS	EPA 8151A mod.	2,4-D
GC-IT/MS	EPA 8151A mod.	2,4-DB
GC-IT/MS	EPA 8151A mod.	4-Nitrophenol
GC-IT/MS	EPA 8151A mod.	Dalapon
GC-IT/MS	EPA 8151A mod.	Dicamba
GC-IT/MS	EPA 8151A mod.	Dichlorprop
GC-IT/MS	EPA 8151A mod.	Dinoseb
GC-IT/MS	EPA 8151A mod.	MCPA
GC-IT/MS	EPA 8151A mod.	Mecoprop MCPP
GC-IT/MS	EPA 8151A mod.	Pentachlorophenol
GC-IT/MS	EPA 8151A mod.	Silvex (2,4,5-TP)
GC-FID	EPA 8015B/AK101/ NWTPH-Gx/NWVPH	Gasoline and Volatile Petroleum Hydrocarbons
GC-FID	EPA 8015B/AK102/ NWTPH-Dx/NWEPH	Diesel and Extractable Petroleum Hydrocarbons
GC-FID	EPA 8015B/AK103/ NWTPH-Dx/NWEPH	Motor Oil and Extractable Petroleum Hydrocarbons
Colorimetric/RFA	EPA 9012A	Total Cyanides
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Chloride
Ion Chromatography	EPA 300.0/9056A	Fluoride
Ion Chromatography	EPA 300.0/9056A	Sulfate
Ion Chromatography	EPA 300.0/9056A	Nitrate
Ion Chromatography	EPA 300.0/9056A	Nitrite
TOC Analyzer (IR)	EPA 9060	TOC
Probe	EPA 9040/9045	pH/Corrosivity
Conductivity meter	EPA 9050	Specific Conductance
Pensky-Martens closed-cup tester/ Setaflash	EPA 1010/1020	Ignitability/Flashpoint
Preparation	Method	Type
Separatory Funnel Liquid-Liquid Extraction	EPA 3510C	Semivolatile and Nonvolatile Organics
Continuous Liquid-Liquid Extraction	EPA 3520	Semivolatile and Nonvolatile Organics
Ultrasonic Extraction	EPA 3550C	Semivolatile and Nonvolatile Organics
Solvent Dilution	EPA 3580	Semivolatile and Nonvolatile Organics



Solid and Chemical Materials		
Preparation	Method	Type
Waste Dilution	EPA 3585	Volatile Organic Compounds
Purge and Trap	EPA 5030	Volatile Organic Compounds
Purge and Trap	EPA 5035	Volatile Organic Compounds
Acid Digestion (Aqueous)	EPA 3005/3010	Inorganics
Acid Digestion (Sediments, Sludges, and Soils)	EPA 3050	Inorganics
TCLP Extraction	EPA 1311	Toxicity Characteristic Leaching Procedure
Florisil Cleanup	EPA 3620B	Cleanup of pesticide residues and other chlorinated hydrocarbons
Silica Gel Cleanup	EPA 3630C	Column Cleanup
Gel Permeation Cleanup	EPA 3640A	Separation of Synthetic Macromolecules
Sulfur Cleanup	EPA 3660B	Sulfur Cleanup Reagent
Sulfuric Acid Cleanup	EPA 3665A	Cleanup for Quantitation of PCBs

Notes:

- 1) This laboratory offers commercial testing service.

Approved By: _____

R. Douglas Leonard
Chief Technical Officer

Date: May 18, 2010

Issued: 01/19/10

Revised: 05/18/10

The State of
Department  Washington
of Ecology

TestAmerica Seattle
Tacoma, WA

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective February 18, 2010 and shall expire February 17, 2011.

Witnessed under my hand on February 18, 2010



Stewart M. Lombard
Lab Accreditation Unit Supervisor

Laboratory ID
C553

WASHINGTON STATE DEPARTMENT OF ECOLOGY
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

TestAmerica Seattle
Tacoma, WA

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. Accreditation for U.S. Environmental Protection Agency (EPA) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods" (SW-846) is for the latest version of the method. SM refers to "Standard Methods for the Examination of Water and Wastewater," 18th through 21st Editions and the Online Edition, unless otherwise indicated. ASTM is the American Society for Testing and Materials. Other references are described in notes.

Matrix/Analyte	Method	Notes
Drinking Water		
Alkalinity as CaCO ₃	SM 2320 B	
Chloride	EPA 300.0	8
Conductivity	SM 2510 B	
Fluoride	EPA 300.0	8
Hardness (calc.)	SM 2340 B	9
Nitrate	EPA 300.0	8
Nitrate	EPA 353.2	10
Nitrite	EPA 300.0	8
Nitrite	EPA 353.2	10
Residue-filterable (TDS)	SM 2540 C	8
Sulfate	EPA 300.0	8
Turbidity	EPA 180.1	8,9
Aluminum	EPA 200.7	8
Antimony	EPA 200.8	8
Arsenic	EPA 200.8	8
Barium	EPA 200.7	8
Barium	EPA 200.8	8
Beryllium	EPA 200.8	8
Cadmium	EPA 200.8	8
Chromium	EPA 200.7	8
Chromium	EPA 200.8	8
Copper	EPA 200.7	8

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Matrix/Analyte	Method	Notes
Copper	EPA 200.8	8
Iron	EPA 200.7	8
Lead	EPA 200.8	8
Manganese	EPA 200.7	8
Mercury	EPA 245.1	
Nickel	EPA 200.7	8
Nickel	EPA 200.8	8
Selenium	EPA 200.8	8
Silica as SiO ₂	EPA 200.7	1,8
Silver	EPA 200.7	8
Silver	EPA 200.8	8
Sodium	EPA 200.7	8
Thallium	EPA 200.8	8
Zinc	EPA 200.7	8
Zinc	EPA 200.8	8
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	10
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	10
Non-Potable Water		
Alkalinity as CaCO ₃	SM 2320 B	10
Amenable cyanide	SM 4500-CN G	
Amenable cyanide	SM 4500-CN I	
Ammonia as N	EPA 350.1	8
Biochemical oxygen demand	SM 5210 B	10
Bromide	EPA 300.0	8
Chemical oxygen demand	SM 5220 C	10
Chloride	EPA 300.0	8
Chromium VI	SM 3500-Cr D	8
Conductivity	EPA 120.1	10
Fluoride	EPA 300.0	8
Nitrate	EPA 300.0	8
Nitrate	EPA 353.2	10
Nitrate + Nitrite	EPA 353.2	10
Nitrate-nitrite	EPA 300.0	8
Nitrite	EPA 300.0	8
Nitrite	EPA 353.2	10
Oil & Grease	EPA 1664	8

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Matrix/Analyte	Method	Notes
Phosphorus, total	EPA 365.1	8
Residue-filterable (TDS)	SM 2540 C	10
Residue-nonfilterable (TSS)	SM 2540 D	10
Residue-settleable	SM 2540 F	8
Residue-total	SM 2540 B	
Residue-volatile	EPA 160.4	10
Salinity	SM 2520 B	
Sulfate	EPA 300.0	8
Total cyanide	EPA 335.4	10
Total hardness as CaCO ₃	SM 2340 C	10
Total organic carbon	SM 5310 B	9
Turbidity	EPA 180.1	9,10
Aluminum	EPA 200.7	8
Antimony	EPA 200.7	8
Antimony	EPA 200.8	8
Arsenic	EPA 200.7	8
Arsenic	EPA 200.8	8
Barium	EPA 200.7	8
Barium	EPA 200.8	8
Beryllium	EPA 200.7	8
Beryllium	EPA 200.8	8
Cadmium	EPA 200.7	8
Cadmium	EPA 200.8	8
Calcium	EPA 200.7	8
Chromium	EPA 200.7	8
Chromium	EPA 200.8	8
Cobalt	EPA 200.7	8
Cobalt	EPA 200.8	8
Copper	EPA 200.7	8
Copper	EPA 200.8	8
Iron	EPA 200.7	8
Lead	EPA 200.7	8
Lead	EPA 200.8	8
Magnesium	EPA 200.7	8
Manganese	EPA 200.7	8
Manganese	EPA 200.8	8

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Matrix/Analyte	Method	Notes
Mercury	EPA 245.1	8
Molybdenum	EPA 200.7	8
Molybdenum	EPA 200.8	8
Nickel	EPA 200.7	8
Nickel	EPA 200.8	8
Potassium	EPA 200.7	8
Selenium	EPA 200.7	8
Selenium	EPA 200.8	8
Silica as SiO ₂	EPA 200.7	8
Silver	EPA 200.7	8
Silver	EPA 200.8	8
Sodium	EPA 200.7	
Strontium	EPA 200.7	8,9
Strontium	EPA 200.8	9
Thallium	EPA 200.7	8
Thallium	EPA 200.8	8
Tin	EPA 200.7	8
Titanium	EPA 200.7	8
Titanium	EPA 200.8	9
Vanadium	EPA 200.7	8
Vanadium	EPA 200.8	8
Zinc	EPA 200.7	10
Zinc	EPA 200.8	8
Organochlorine Pesticides	EPA 608	8
Polychlorinated Biphenyls	EPA 608	7,8
BNA Extr (Semivolatile) Organics	EPA 625	8
BTEX	EPA 624	8
Organo-tins	Krone 1988	3
Volatile Organic Compounds	EPA 624	4,8
Solid and Chemical Materials		
Amenable cyanide	EPA 9012	8
Total cyanide	EPA 9012	8
Total organic carbon	EPA 9060	
Aluminum	EPA 6010	8
Antimony	EPA 6010	8
Antimony	EPA 6020	8

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Matrix/Analyte	Method	Notes
Arsenic	EPA 6010	8
Arsenic	EPA 6020	8
Barium	EPA 6010	8
Barium	EPA 6020	8
Beryllium	EPA 6010	8
Beryllium	EPA 6020	8
Cadmium	EPA 6010	8
Cadmium	EPA 6020	8
Calcium	EPA 6010	8
Chromium	EPA 6010	8
Chromium	EPA 6020	8
Chromium VI	EPA 6010	
Cobalt	EPA 6010	8
Cobalt	EPA 6020	8
Copper	EPA 6010	8
Copper	EPA 6020	8
Iron	EPA 6010	8
Lead	EPA 6010	8
Lead	EPA 6020	8
Magnesium	EPA 6010	8
Manganese	EPA 6010	8
Manganese	EPA 6020	8
Mercury, Liquid Waste	EPA 7470	8
Mercury, Solid Waste	EPA 7471	8
Molybdenum	EPA 6010	8
Molybdenum	EPA 6020	8
Nickel	EPA 6010	8
Nickel	EPA 6020	8
Potassium	EPA 6010	8
Selenium	EPA 6010	8
Selenium	EPA 6020	8
Silica as SiO ₂	EPA 6010	
Silver	EPA 6010	8
Silver	EPA 6020	8
Sodium	EPA 6010	8
Strontium	EPA 6010	8

TestAmerica Seattle

Matrix/Analyte	Method	Notes
Thallium	EPA 6010	8
Thallium	EPA 6020	8
Tin	EPA 6010 Mod	8
Titanium	EPA 6010 Mod	8
Vanadium	EPA 6010	8
Vanadium	EPA 6020	8
Zinc	EPA 6010	8
Zinc	EPA 6020	8
BTEX & MTBE	EPA 8021 Mod BTEX/MTBE	10
Chlorinated Herbicides	EPA 8151	8
EDB & DBCP	EPA 8011	10,14
Glycols	EPA 8015 Mod Gly	6
Organochlorine Pesticides	EPA 8081	8
Petroleum Hydrocarbons, Extractable	WDOE EPH	2
Petroleum Hydrocarbons, Volatile	WDOE VPH	2
Polychlorinated Biphenyls	EPA 8082	7,8,13
Total Pet Hydrocarbons - Diesel	WDOE NWTPH-Dx	2,8
Total Pet Hydrocarbons - Gasoline	WDOE NWTPH-Gx	2,8
BNA Extr (Semivolatile) Organics	EPA 8270	5,8
Organo-tins	Krone 1988	3
Polycyclic Aromatic HC	EPA 8270	8
Total Pet Hydrocarbons - Gasoline	WDOE NWTPH-Gx	2,11
Volatile Organic Compounds	EPA 8260	4,8
Corrosivity	EPA 9045	8
Ignitability	EPA 1010	8,9
Ignitability	EPA 1020	8
Particle Size Distribution	ASTM D 422	12
Particle Size Distribution	Plumb 1981	
Particle Size Distribution (Sed)	PSEP 1986 Wet Sieve	

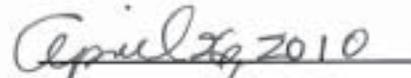
Matrix/Analyte	Method	Notes
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Accredited Parameter Note Detail

(1) Modified to use an ion trap MS detector instead of the method specified detector. (2) Washington Department of Ecology Analytical Methods for Petroleum Hydrocarbons, Publication Number ECY 97-602, June 1997. (3) Procedure is an Ion Trap method for determination of tetra-, tri-, di-, and monobutyltin in sediments and pore water. (4) Method modified to use tuning criteria required by EPA CLP. (5) For sediments, modifications are: 30-minute sonication at elevated temperature with a heated ultrasonic bath; use of 40-mL Surlyn coated vials; and extraction of 10 grams of sample with an initial solvent volume of 20 mL instead of extraction of 30 grams of sample with an initial solvent volume of 60 mL. (6) Method modified to determine glycols according to lab SOP. (7) When acid cleanup is not necessary, lab runs according to EPA 8081 protocol. (8) Accreditation based in part on recognition of Oregon NELAP accreditation. (9) Provisional pending receipt of an acceptable PT result. (10) Interim pending on-site audit to verify lab capability. (11) GC-MS. (12) Includes hydrometer and modified methods. (13) Includes oil matrix. (14) Limited to water only.



Authentication Signature



Date

Stewart M. Lombard, Lab Accreditation Unit Supervisor

Record of Management Decision

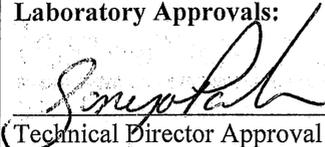
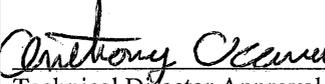
SOP: TA-___-___-R__ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00026

Effective Date: 11/2/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>11/01/10</u> Date
 Technical Director Approval (Ryan)	<u>10-28-10</u> Date
 Technical Director Approval (Ocana)	<u>11/1/10</u> Date
	Technical Director Approval/H & S Approval Date
 Quality Assurance Approval	<u>10-28-10</u> Date
	Laboratory Director Approval Date

1. **Description Of and Reason For Decision:**

As noted by a client, the columns identified in our SOP weren't the same as the columns identified on the package forms or raw data.

The chromatography column information typically provided in section 6 will be qualified with the following italicized text when appropriate:

(Column or primary column: Column ID and dimensions, as currently stated in the SOP) *or equivalent*
 (Secondary column: Column ID and dimensions, when applicable and as currently stated in the SOP) *or equivalent*
Note: Other columns may be used. These were the columns in place at the time the SOP was prepared.

**Column types must also be maintained by the analyst in TALS as previously noted.

2. **References:** AECOM/Port Heiden

3. **Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):**

AM 11-2-10 KST
 A. Ocana, A. Mattison, K. Teffeau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,
AO 11/1/10 11/1/10 Jil/10 OM 11/2/10 MAT 11/1/10 AT 11/1/10 EK 11/2/10
 M. Muir, S. Chambers, B. Hepner, T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
** 11/1/10 sv 11/1/10 BJA 11/1/10 10-28-10 SMC 10/28/10 J 11/1/10 MAT 11-1-10*

SOPs for Chromatography Methods

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0354-R01	PCBs in Transformer Oil
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)

Record of Management Decision

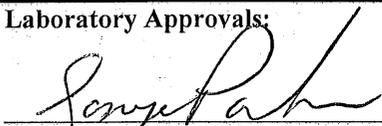
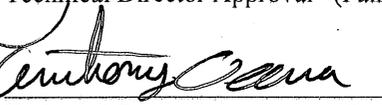
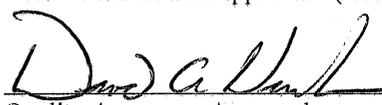
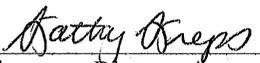
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00019

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
	10-15-10
Technical Director Approval (Palmer)	Date
	10/26/10
Technical Director Approval (Ocana)	Date
	10/28/10
Quality Assurance Approval	Date
	10-15-10
Technical Director Approval (Ryan)	Date
	10-15-10
Technical Director Approval/H & S Approval	Date
	10/28/10
Laboratory Director Approval	Date

1. **Description Of and Reason For Decision:**

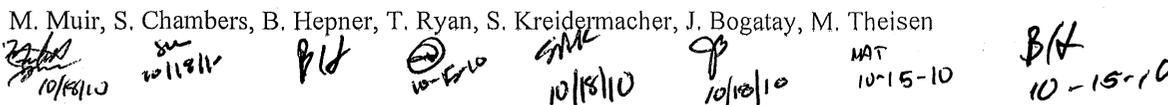
As noted in an L-A-B DOD audit finding, instrument records did not consistently include the identification of the chromatographic column.

There are two venues to address this. In the SOP, under the section on Equipment and Supplies, a column type could be designated with the disclaimer "or equivalent". Also, as part of maintenance procedures, the specific make and model of chromatography columns installed need to be documented in the instrument's maintenance logbook.

2. **References:** QSM 4.1, 4.12.2.5.3c

3. **Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):**

A. Ocana, A. Mattison, K. Tefteau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,

SOPs for Chromatography Methods

(Add Column Types)

SOP No	SOP Title	Column Type 1	Column Type 2
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A	Zebrom MR-2	Zebrom MR-1
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)	Phenom. ZB-1	
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod	↓	
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)	↓	
TA-GS-0351-R17	PCBs by EPA 8082	Zebrom MR-2	Zebrom MR-1
TA-GS-0354-R01	PCBs in Transformer Oil	↓	↓
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology	Phenom. ZB-1	
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)	↓	
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)	Roshek RTX 5ms	Roshek RTX XLB
TA-GS-0380-R09	Pesticides and PCBs by EPA 608	Zebrom MR-2	Zebrom MR-1
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology	Roshek VRX	
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)	↓	
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)	Zebrom 5ms	
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)	ZB- 5MS	
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)	ZB-5ms	
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)	ZB624	
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)	Roshek VRX	ZB624
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)	Dionet AG-18/ AS 18	AG14/ AS14

TAC044 TAC038

Record of Management Decision

SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00020

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10-15-10</u> Date
 Technical Director Approval (Ocana)	<u>10/26/10</u> Date
 Quality Assurance Approval	<u>10/28/10</u> Date
 Technical Director Approval (Ryan)	<u>10-15-10</u> Date
 Technical Director Approval/H & S Approval	<u>10-15-10</u> Date
 Laboratory Director Approval	<u>10/28/10</u> Date

1. **Description Of and Reason For Decision:**

As noted in an L-A-B DOD audit finding, instrument and equipment records did not consistently include the operating conditions of the instruments and equipment.

There are two venues to address this. In the SOP, under the section on Procedures, operating conditions could be specified or generalized. Also, as part of maintenance procedures, the specific operating conditions need to be documented in the instrument's or equipment's maintenance logbook. This documentation could be in the form of a written summary or a printout.

2. **References:** QSM 4.1, 4.12.2.5.3c

3. **Others Notified (date and initial below your name AFTER verifying/updating your maintenance logs):**

- A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway
 AM 10-20-10 KET
 DO 10/20/10 10/24/10
- S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner
 10/26/10 CA 10/26/10 10/26/10 AP 10/22/10 EK 10/22/10
- K. Johnson, D. Brechler, MJ. Tangora
 KES 10/25/10 10/25/10
- T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 10-15-10 SAK 10/18/10 10/10/10 MAT 10-15-10

SOPs for Methods with Operating Conditions

(Document Operating Conditions in Maintenance Manual if Different Than Those Described in SOP)

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391-R07	Analysis of Volatile Organic Compounds by Method 802.1B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MT-0200-R19	✓ Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	✓ Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	✓ Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-OP-0367-R00	Microwave Extraction Procedure
TA-OP-0388-R07	Gel Permeation Chromatography Extract Clean-up Procedure (3600C and 3640A)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0158-R10	Total Halogens EPA 9076)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0186-R01	Total Halogens
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP delineates the specific requirements for qualitative and semi-quantitative hydrocarbon identification and analysis. This method is applicable to both soil and water matrices. Because the soil and water extraction is identical to that found in the water portion of the NWTPH-Dx method, products may be quantitated using this extract.

1.1.2 Reporting Limits:

1.1.2.1 Soil: <20 mg/kg (gasoline), <50 mg/kg (diesel), and <100 mg/kg (oil)

1.1.2.2 Water: <0.10 mg/L (gasoline), <0.25 mg/L (diesel), and <0.50 mg/L (oil)

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 13.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

This method is a qualitative and semi-quantitative procedure that is used to identify petroleum fuel hydrocarbons containing components in the toluene through nC₃₆ range. Soil samples are dried, surrogate added and extracted with methylene chloride and then analyzed by GC-FID. Water samples are acidified, extracted with methylene chloride, concentrated and analyzed by GC-FID. Results are reported as less than (<) or greater than (>) the State specified limits for the gasoline range (toluene through nC₁₂), diesel range (>nC₁₂ through nC₂₄), and oil range (>nC₂₄ through nC₃₂).

3.0 Definitions

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Seattle Quality Assurance Manual (QAM).

4.0 Interferences

Non-petroleum hydrocarbons (polar and nonpolar) will also be extracted using this procedure. Hydrocarbons eluting in the ranges described above for fuel hydrocarbons will be detected and reported as false positives. Follow up confirmation analyses (NWTPH-Gx and NWTPH-Dx) with appropriate cleanup techniques will eliminate most of the false positives.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

- 5.1.2** There are areas of high voltage in both the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Gas chromatograph and detector: Hewlett Packard 6890 or equivalent w/wo autosampler
- Chromatographic column types:
 - i.) Phenomenex ZB-1 ms 20 meters x 0.18 mm ID x 0.18 um film thickness
 - ii.) Phenomenex ZB-5: 30 meters x 0.25 mm ID x 0.25 um film thickness, cut in half to give two (2) 15 meter columns for dual column applicable instruments.
 - iii.) Restek Rtx-5MS: 30 meters x 0.25 mm ID x 0.5 um film thickness, cut in half to give two (2) 15 meter columns for dual column applicable instruments
 - iv.) Restek Rtx-5MS: 15 meters x 0.25 mm ID x 0.5 um film thickness
- Data acquisition system: Hewlett Packard ChemStation or equivalent
- Analytical balance, 0.0001 g accuracy, **analysts must ensure the balance is calibrated each day before use and that the calibration brackets the weights to be determined.**

6.2 Supplies

- Gastight syringe, 10 uL
- Volumetric flasks, 10 mL
- Sample bottles with Teflon-lined screw caps, 4 oz

- Drying oven
- Glass standard vials with screw caps and Teflon-coated septum
- Autosampler vials, 1.5 mL, crimp top or equivalent
- 20 ml scintillation vials, with Teflon lined lids

7.0 Reagents and Standards

7.1 *Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP TA-QA-0619.*

7.2 Methylene Chloride (CH₂Cl₂), analytical reagent grade or equivalent.

7.3 Sodium Sulfate, anhydrous powder, reagent grade or equivalent.

7.4 Retention time window standard. The retention time standard is prepared by diluting the EPH aliphatic stock standard (EPH AL calstk_0000X), and TPH surrogates (AK1023_SR_0000X) to a final concentration of 20 ug/mL each, with a final volume of 25 mL. The standard contains nC8-nC40, except nC39, plus surrogates. (See chromatogram, Attachment 1)

7.5 Reference standards. Individual petroleum product reference standards are analyzed annually to generate a library of approximately 25 petroleum products. This ensures accurate identification of petroleum product contamination by fingerprinting techniques.

7.6 Gasoline stock standard. A 50,000 mg/L unleaded gasoline standard purchased from Restek (part #30206) is used. Alternatively, equal portions of three grades of non-oxygenated gasoline (regular, unleaded regular, and unleaded supreme) are mixed together to form a composite gasoline. From this composite a stock standard is prepared by placing approximately 9 mL of methylene chloride in a 10 mL volumetric flask. The flask is allowed to stand unstoppered until the alcohol-wetted surfaces have dried (about 10 minutes), then the flask and methylene chloride are placed on the analytical balance and the balance tarred to zero. Add about 10 drops of the composite gasoline standard to the flask such that the liquid drops directly into the methanol. Reweigh and dilute to volume (10 mL) with methanol. The gasoline standard concentration (approximately 10,000 mg/L) is calculated as follows:

$$\text{Concentration ug/mL} = \frac{(\text{Final wt., mg}) - (\text{Tare wt., mg})}{(10 \text{ ml})} \times \frac{1000 \text{ ug}}{1 \text{ mg}}$$

7.7 Diesel stock standard. A 50,000 mg/L #2 diesel standard purchased from Restek (part #31259) is used. Alternatively, equal portions of #2 diesel oil from at least three different oil companies are mixed together to yield a composite diesel fuel. From this composite fuel a stock standard is prepared by adding about 5 drops of the composite diesel to a tarred 10 mL volumetric flask. The flask is reweighed and brought to volume with methylene chloride, then thoroughly mixed to disperse the diesel. The diesel standard concentration is calculated (approximately 10,000 mg/L) in the same manner as the gasoline standard, using the equation in 7.5.

7.8 Motor Oil stock standard. A 20,000 mg/L SAE 30W motor oil standard purchased from Accustandard. Alternatively, a 0.1 gram portion of Penzoil SAE 30 W brand motor oil (or equivalent) is added to methylene chloride to a final volume of 10 mL. This 10,000-mg/L standard is diluted 1:100 for analysis.

7.9 Surrogate stock standard. Approximately 0.2 g (weighted to the nearest 0.0001 g) of 1-bromo-4-fluorobenzene (99% Aldrich) and 0.2g of o-Terphenyl (99%, Aldrich) are diluted to a final volume of 100 mL with acetone, providing a stock spiking solution of

approximately 2,000 mg/L. An alternate vendor may be used providing the standard is equivalent.

- 7.10** Composite calibration working standard. Two separate mixtures are prepared containing 20 ug/ml gasoline and 50 ug/mL diesel, from the stock standards described in Sections 7.0 and 7.5. For soils, add the appropriate volume of surrogate stock standard (described in section 7.9 above) to obtain a final concentration of 20 ug/mL; for waters, the final concentration of 40 ug/mL. The working standard corresponds to 20 mg/kg gasoline and 50 mg/kg diesel in soil and 0.10 mg/L gasoline and 0.25 mg/L #2 diesel for water, based off of the following initial/final volume ratios: soils: 10g/10mL; waters: 1000mL/5mL.
- 7.11** Motor oil working standard. A working standard is prepared from the stock standard at 1:200 dilution, corresponding to 100 mg/kg motor oil in soil and 0.50 mg/L in water, based off of the following initial/final volume ratios: soils: 10g/10mL; waters: 1000mL/5mL.
- 7.12** 1+1 HCl, reagent grade, for water preservation and extraction. Dilute 250 ml of hydrochloric acid with 250 ml or reverse osmosis water to make 1+1 HCl.
- 7.13** Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP TA-EHS-0036.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	1 Liter	Cool 0-6°C	7 Days	28 Days from collection	40 CFR Part 136.3
Soils	Glass	30 grams	Cool 0-6°C	14 Days	28 Days from collection	N/A

9.0 Quality Control

- 9.1** The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.
- 9.1.1** The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in SOP TA-QA-0620, *Quality Control Program*.
- 9.1.2** Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- 9.1.3** Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client

can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. This is in addition to the corrective actions described in the following sections.

9.2 Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed together using the same reagents and standards. Each quality control batch must contain a method blank (MB), a laboratory control sample (LCS) and a duplicate pair. *Per the method, a duplicate must be prepared and analyzed for every 10 samples. For this method, a matrix spike/matrix spike duplicate (MS/MSD) pair is only performed upon request. For more details see SOP TA-QA-0620.*

9.3 Method Blank (MB)

One method blank is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The method blank consists of either 1 liter of organic-free water (for batches of aqueous samples) or 10 grams of Ottawa sand (for batches of soil samples). The method blank is processed exactly as samples in the batch, and is used to assess whether the laboratory processes have contaminated the samples in the batch.

Acceptance Criteria: Results for the method blank must be less than or equal to the reporting limit concentration or less than 5% of the lowest concentration found in the associated samples.

Corrective Action: If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.

9.4 Laboratory Control Sample (LCS)

If running NWTPH-Dx simultaneously with this HCID method, one LCS is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The LCS (and LCSD as appropriate) consists of either 1 liter of organic-free water (for batches of aqueous samples) or 10 grams of Ottawa sand (for batches of soil samples), to which 100 µL of spike solution is added. The LCS is processed exactly as samples in the batch and is used to assess the accuracy of the analytical system.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. The control limits are set at ± 3 standard deviations around the calculated mean of the historical LCS recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy TA-QA-0620 for further details.

Corrective Action: If LCS acceptance limits are not met, the LCS should be reanalyzed once to confirm that the original analysis is reliable. If the results are still outside control limits, the associated samples must be re-extracted and reanalyzed. If the LCS recovery is above the upper control limit, and the associated samples are all below reportable concentrations, the deviation may be described in an NCM, if this is acceptable to the client or allowed by the specific program or project.

9.5 Surrogate Spikes

The o-terphenyl and 1-bromo-4-fluorobenzene surrogates have chemistry similar to the analytes of interest, but are not expected to be found in environmental samples.

Surrogate results are used to assess the performance of the analytical system for each field and QC sample (*including instrument blanks*).

Acceptance Criteria: The percent recovery of the surrogates must fall within 50-150% as established by WA DOE.

Corrective Action: If surrogate recoveries are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. High recoveries may be due to a co-eluting matrix interference and the chromatogram should be examined for evidence of this. Low recoveries may be due to adsorption by the sample matrix (e.g., clay particles, peat, or organic material in the sample). Recalculate the results and/or reanalyze the extract if the checks reveal a problem.

If the surrogate recovery is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative. As with matrix spike failures, some programs (e.g., USACE) may require additional analyses to confirm suspected matrix interferences. The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that a matrix effect is reproducible.

NOTE: Percent solids less than 80% may cause the 1-bromo-4-fluorobenzene surrogate to fail.

NOTE: For BP samples, if the surrogate percent recovery fails, the recovery must be confirmed by re-extraction and reanalysis with the following exceptions:

- *The lab has unequivocally demonstrated a sample matrix effect and informed the BP representative.*
- *The recovery exceeds control limits and all target analytes in the sample are non-detect.*

9.6 RT Reference Standard

The retention time window is established by injecting a mixture of n-alkanes from Toluene to n-hexatriacontane (C₃₆) three times over a 72 hour period. The mean and standard deviation for the three retention times are calculated. The width of the RT window is set at ± 3 times the standard deviations of the mean RT. If the resulting RT window is less than 0.03 minutes, then a window of 0.03 minutes is used.

Acceptance Criteria: Toluene must be resolved from the solvent peak.

Corrective Action: If the acceptance criteria are not met, check instrument conditions and calibration materials, correct as necessary and repeat analysis of the reference standard before proceeding with the analysis of samples.

9.7 Instrument QC**9.8 Initial Calibration (ICAL)**

9.8.1 *The 20/50 gas/diesel and 20 or 40 surrogate working standard, and the 100 motor oil standard are analyzed and updated every 24 hours for sample comparison as a single point calibration.*

9.9 *Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.*

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

10.1 Percent Solids

10.1.1 For solid samples, the percent solids (dry weight) of an approximately 10 gram portion of the sample is determined for use in determination of the concentration of analyte in the sample, following procedures described in SOP TA-WC-0160.

10.2 Calibration

10.2.1 The gas chromatograph is set up to initial conditions as follows:

Injector: 280°C

Detector: 300°C

Oven ramping profile for 6890 GC systems with 15 meter column(s): Initial column temperature is set to 45-60°C, and held for 0.5 minutes, ramped to 340°C at 30°C/min and held for 2-3 minutes. Flow is set to constant flow at 1.5-3.5 mL/min. A post run is initialized at 340°C at a flow rate of 5.0 mL/min, and held for 2-4 minutes to clean out the system of contaminants.

Oven ramping profile for 5890 GC systems with 15 meter column(s): The oven temperature program parameters for these systems are similar to that of the 6890 systems, however, the final holding time is increased to 4-6 minutes due to the instrument software not having a post run capability.

Note: the oven ramping profile will vary from instrument to instrument, as each does not perform exactly like one another. In addition, actual column lengths and types vary as well.

10.2.2 The FID is stabilized at manufacturer recommended makeup and carrier gas flows prior to analysis.

10.2.3 Standard and surrogate solutions are allowed to come to room temperature prior to use.

10.2.4 *The 20/50 gas/diesel and 20 or 40 surrogate working standard, and the 100 motor oil standard are analyzed and updated every 24 hours for sample comparison as a single point calibration.*

10.3 Sample Analysis

10.3.1 Extracts are analyzed by injection of 1 uL on the GC by autosampler; the chromatograms are compared to the daily 20/50 standard and motor oil standard for identification of petroleum products. For samples containing petroleum products other than gasoline, diesel or motor oil, if the product is known by site characterization or by the client, the analyst may prepare calibration standards of the product by the methods listed above or analyze the sample by a fully qualitative method such as NWTPH-Gx.

10.3.2 Gasoline is indicated if compounds are detected from toluene through dodecane (nC₁₂).

10.3.3 Diesel and related products are indicated if compounds are detected eluting after dodecane (nC₁₂) through tetracosane (nC₂₄).

10.3.4 Oil is indicated by the presence of a basically unresolved chromatographic envelope generally originating or extending beyond tetracosane (nC₂₄). Bunker-C and related products are indicated by a diesel-like pattern that extends beyond diesel and an unresolved chromatographic envelope extending beyond nC₂₄.

10.4 An example instrument analysis sequence is shown in Attachment 2.

10.5 Sample Hydrocarbon Identification and Calculations

10.5.1 Petroleum products are to be identified by pattern matching with reference product chromatograms generated the same day as the sample analysis. The terms "gasoline range" or "diesel range" hydrocarbons, or derivations of them, should only be used when the analyst is unable to identify the petroleum product present. When these terms are used, it is to indicate the presence of compounds eluting in the corresponding carbon ranges. Motor oils, hydraulic fluids and similar petroleum products originating or extending beyond tetracosane (nC₂₄), may be reported using the collective term lube oil unless specific identification is possible. Heavy fuel oils, e.g. fuel oil #6 or Bunker Crude, which contain diesel range components as well as a lube oil range, may be reported using the collective term, heavy fuel oil, unless specific identification is possible. These products should not be confused with mixtures of #2 diesel and motor oils. **Note:** The actual identification of the grade or type of lube oil and heavy fuel oil may require equipment and techniques beyond the scope of this method.

10.5.2 Gasoline. The calibration standard area of the components from toluene through dodecane is integrated to the baseline as a group. The samples are integrated in the same manner and the areas compared. Raw area counts in the gasoline range are corrected for surrogate area. If the sample area exceeds the calibration standard area, ">20 mg/kg" or ">0.10 mg/L" is reported for the sample for soil or water, respectively. If the sample area does not exceed the calibration standard area, "< 20 mg/kg" or "<0.10 mg/L" is reported for the soil or water sample.

10.5.3 Diesel. The calibration standard area of the components from dodecane through tetracosane is integrated to the baseline as a group. The samples are integrated in the same manner and the areas compared. Raw area counts in the diesel range are corrected for surrogate area. If the sample area exceeds the calibration

standard area, ">50 mg/kg" or ">0.25 mg/L" is reported for the soil or water sample, respectively. If the sample area does not exceed the calibration standard area, "< 50 mg/kg" or "<0.25 mg/L" is reported for the soil or water sample. If the diesel range area exceeds the calibration standard area for a water sample, the NWTPH-Dx Method may be performed.

10.5.4 Oil. The calibration standard area of the components after tetracosane is integrated to the baseline as a group. The samples are integrated in the same manner and the areas compared. If the sample area exceeds the calibration standard area, ">100 mg/kg" or ">0.50 mg/L" is reported for the sample for soil or water, respectively. If the sample area does not exceed the calibration standard area, "< 100 mg/kg" or "<0.50 mg/L" is reported for the soil or water sample.

10.6 Accuracy

% Recovery = $\frac{\text{observed concentration}}{\text{known concentration}} \times 100$

10.7 Precision (RPD)

Sample Duplicate (DUP) = $\frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$

11.0 Method Performance

11.1 Method Detection Limit Study (MDL)

A method detection limit (MDL) study is not performed for this analysis.

11.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

11.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

12.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

13.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP TA-EHS-0036.

13.1 Waste Streams Produced by the Method

13.1.1 Acidic extracted sample and QC wastewater. After the extraction has been completed the spent water is neutralized and then collected into the organics extraction water conical reservoir. The collected wastewater is then purged with air to remove any remaining methylene chloride. The air-purged wastewater is

then tested for methylene chloride. When the concentration levels are at or below local discharge limits, the wastewater can be discarded down the drain.

13.1.2 Methylene chloride waste. Any waste methylene chloride, i.e. KD rinses, syringe rinses, etc., from the extraction procedure is collected in beakers and then poured into a 4-liter amber bottle (appropriately labeled) located in the hood. After the extraction has been completed the MeCl₂ collected in the 4 L bottles is emptied into the MeCl₂ waste barrel located in the waste disposal room.

13.1.3 Vial extract waste. Sample extracts that have been placed in vials for analysis are discarded into plastic waste buckets located underneath the bench top. Once the buckets are full the GC vials are discarded into the non-PCB GC vial waste barrel located in the waste room.

13.1.4 Extract waste. Unused sample extracts are held for at least 40 days, in case further testing is deemed necessary. After at least 40 days has passed these extracts are transported to the waste room in racks of 100 were they are disposed of by the sample waste technician.

14.0 References / Cross-References

14.1 Analytical Methods for Petroleum Hydrocarbons, WSDOE, Toxics Cleanup Program and the Ecology Environmental Laboratory, Publication No. ECY 97-602, June 1997.

14.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8015B.

15.0 Method Modifications:

Item	Method xx	Modification
1	NWTPH-HCID	A full liter is used for water extraction. Final extract volume for waters is 5.0 mL due to the fact that a larger sample volume is used for extraction.

16.0 Attachments

Attachment 1: n-Alkane Retention Time Standard Chromatogram Example Instrument Sequence

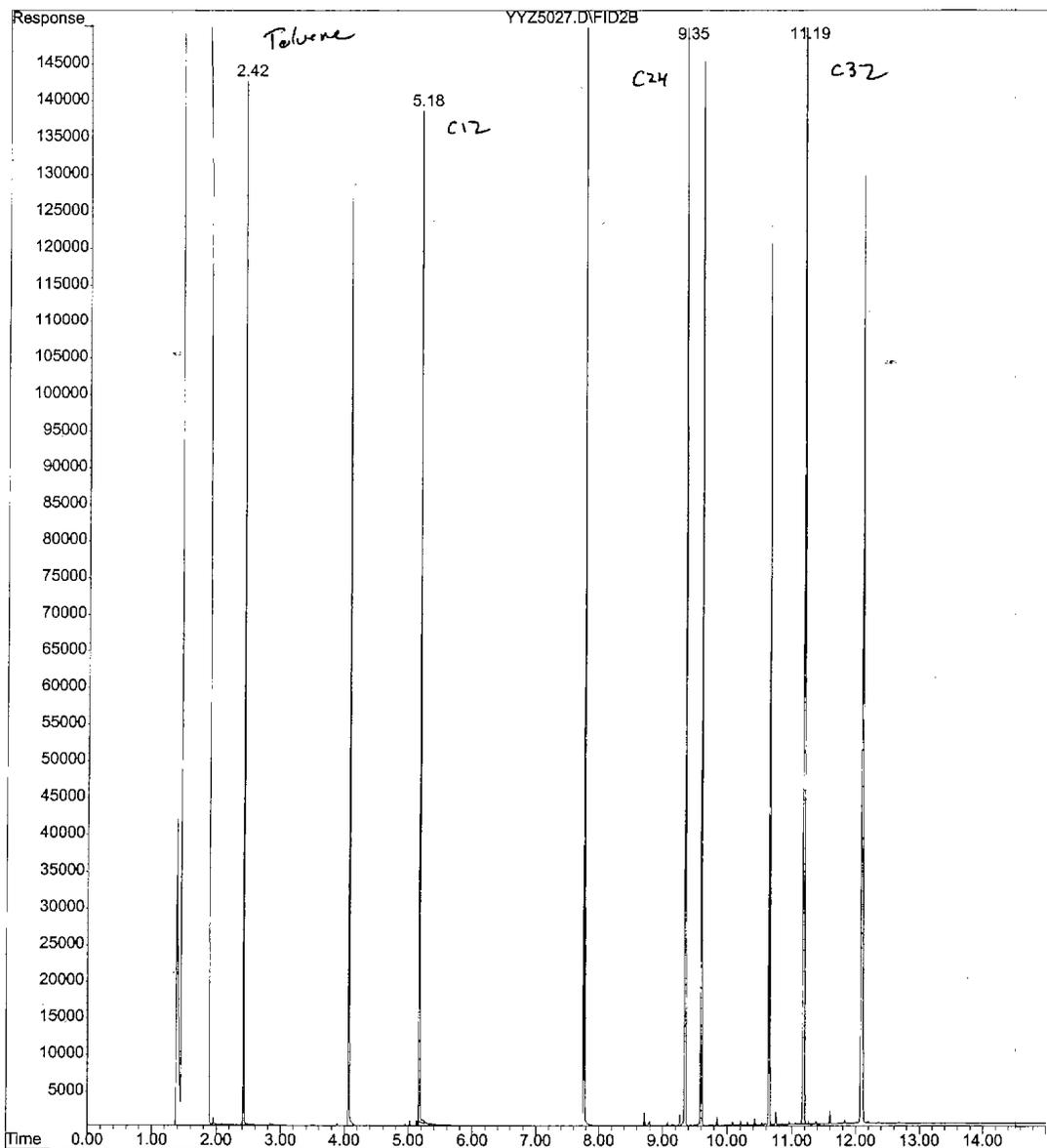
Attachment 2: Example Instrument Sequence

17.0 Revision History

- Revision 8, dated 26 March 2010
 - Added documentation of standards/reagents and standard/reagent preparation Section 7.1
 - Added removal of expired standards Section 7.13.
 - Added BP surrogate requirements, Section 9.5.
 - Added criteria for additional QC, Section 9.9.
 - Integration for TestAmerica Bothell and TestAmerica Seattle operations.
- Revision 7, dated 9 May 2008
 - Integration for TestAmerica and STL operations.

Attachment 1. n-Alkane Retention Time Standard Chromatogram

File : F:\DATA\051208_A\YYZ5027.D
Operator : TMR
Acquired : 12 May 2008 10:51 using AcqMethod RACQ.M
Instrument : SEA014
Sample Name: 1166-95-4 n-alkane rt std
Misc Info : BT=S014051208
Vial Number: 2



Attachment 2. Example Instrument Sequence

Directory: F:\DATA\051208_A				Injection Log		
Line	Vial	FileName	Multiplier	SampleName	Misc Info	Injected
1	1	Yyz5026.d	1.	RINSE	BT=S014051208	12 May 2008 10:28
2	2	Yyz5027.d	1.	1166-95-4 n-alkane rt std	BT=S014051208	12 May 2008 10:51
3	3	Yyz5028.d	1.	267109 2050gd HCID	BT=S014051208	12 May 2008 11:14
4	4	Yyz5029.d	1.	267110 100m HCID	BT=S014051208	12 May 2008 11:37
5	5	Yyz5030.d	1.	580-9817-A-61-B	BT=S014050908	12 May 2008 12:05
6	6	Yyz5031.d	1.	580-9817-A-61-C DU	BT=S014050908	12 May 2008 12:28
7	7	Yyz5032.d	1.	MB 580-31160/1-A	BT=S14051208	12 May 2008 16:16
8	8	Yyz5033.d	1.	580-9882-A-1-A	BT=S14051208	12 May 2008 16:39
9	9	Yyz5034.d	1.	580-9882-A-1-B MS	BT=S14051208	12 May 2008 17:02
10	10	Yyz5035.d	1.	580-9882-A-1-C MSD	BT=S14051208	12 May 2008 17:32
11	11	Yyz5036.d	1.	580-9882-A-2-A	BT=S14051208	12 May 2008 18:01
12	12	Yyz5037.d	1.	580-9817-A-67-D	BT=S14051208	12 May 2008 18:29

INST: SEA014
 METH: NWTPH-HC
 5/13/8

Record of Management Decision

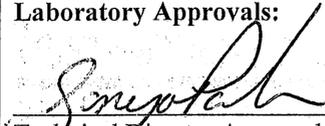
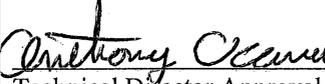
SOP: TA-___-___-R__ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00026

Effective Date: 11/2/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 _____ Technical Director Approval (Palmer) Date	 _____ Technical Director Approval (Ryan) Date
 _____ Technical Director Approval (Ocana) Date	_____ Technical Director Approval/H & S Approval Date
 _____ Quality Assurance Approval Date	_____ Laboratory Director Approval Date

1. **Description Of and Reason For Decision:**

As noted by a client, the columns identified in our SOP weren't the same as the columns identified on the package forms or raw data.

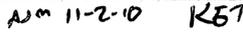
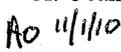
The chromatography column information typically provided in section 6 will be qualified with the following italicized text when appropriate:

(Column or primary column: Column ID and dimensions, as currently stated in the SOP) *or equivalent*
 (Secondary column: Column ID and dimensions, when applicable and as currently stated in the SOP) *or equivalent*
Note: Other columns may be used. These were the columns in place at the time the SOP was prepared.

**Column types must also be maintained by the analyst in TALS as previously noted.

2. **References:** AECOM/Port Heiden

3. **Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):**

 11-2-10 KBT
 A. Ocana, A. Mattison, K. Teffeau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,
 11/1/10  11/1/10  11/1/10  11/2/10  11/1/10  11/1/10  11/2/10
 M. Muir, S. Chambers, B. Hepner, T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 11/1/10  11/1/10  11/1/10  10-28-10  10/28/10  11/1/10  11-1-10

SOPs for Chromatography Methods

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0354-R01	PCBs in Transformer Oil
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)

Record of Management Decision

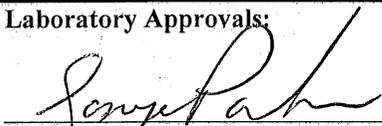
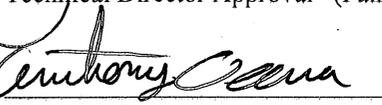
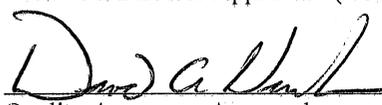
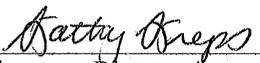
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00019

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
	10-15-10
Technical Director Approval (Palmer)	Date
	10/26/10
Technical Director Approval (Ocana)	Date
	10/28/10
Quality Assurance Approval	Date
	10-15-10
Technical Director Approval (Ryan)	Date
	10-15-10
Technical Director Approval/H & S Approval	Date
	10/28/10
Laboratory Director Approval	Date

1. **Description Of and Reason For Decision:**

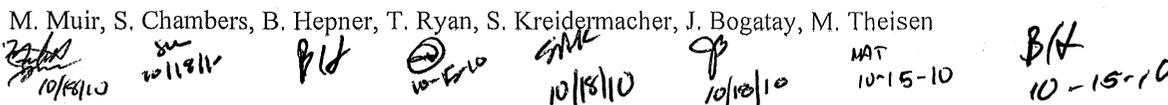
As noted in an L-A-B DOD audit finding, instrument records did not consistently include the identification of the chromatographic column.

There are two venues to address this. In the SOP, under the section on Equipment and Supplies, a column type could be designated with the disclaimer "or equivalent". Also, as part of maintenance procedures, the specific make and model of chromatography columns installed need to be documented in the instrument's maintenance logbook.

2. **References:** QSM 4.1, 4.12.2.5.3c

3. **Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):**

A. Ocana, A. Mattison, K. Tefteau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,

SOPs for Chromatography Methods

(Add Column Types)

SOP No	SOP Title	Column Type 1	Column Type 2
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A	Zebrom MR-2	Zebrom MR-1
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)	Phenom. ZB-1	
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod	↓	
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)	↓	
TA-GS-0351-R17	PCBs by EPA 8082	Zebrom MR-2	Zebrom MR-1
TA-GS-0354-R01	PCBs in Transformer Oil	↓	↓
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology	Phenom. ZB-1	
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)	↓	
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)	Roshek RTX 5ms	Roshek RTX XLB
TA-GS-0380-R09	Pesticides and PCBs by EPA 608	Zebrom MR-2	Zebrom MR-1
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology	Roshek VRX	
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)	↓	
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)	Zebrom 5ms	
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)	ZB- 5MS	
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)	ZB-5ms	
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)	ZB624	
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)	Roshek VRX	ZB624
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)	Dionet AG-18/ AS 18	AG14/ AS14

TAC044 TAC038

Record of Management Decision

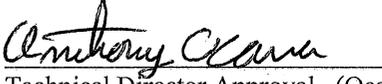
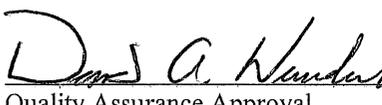
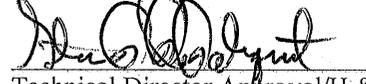
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00020

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10-15-10</u> Date
 Technical Director Approval (Ocana)	<u>10/26/10</u> Date
 Quality Assurance Approval	<u>10/28/10</u> Date
 Technical Director Approval (Ryan)	<u>10-15-10</u> Date
 Technical Director Approval/H & S Approval	<u>10-15-10</u> Date
 Laboratory Director Approval	<u>10/28/10</u> Date

1. **Description Of and Reason For Decision:**

As noted in an L-A-B DOD audit finding, instrument and equipment records did not consistently include the operating conditions of the instruments and equipment.

There are two venues to address this. In the SOP, under the section on Procedures, operating conditions could be specified or generalized. Also, as part of maintenance procedures, the specific operating conditions need to be documented in the instrument's or equipment's maintenance logbook. This documentation could be in the form of a written summary or a printout.

2. **References:** QSM 4.1, 4.12.2.5.3c

3. **Others Notified (date and initial below your name AFTER verifying/updating your maintenance logs):**

- A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway
 AM 10-20-10 KET
 DO 10/20/10 10/24/10
- S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner
 Bole 10/26/10 CA 10/26/10 AP 10/22/10 EK 10/22/10
- K. Johnson, D. Brechler, MJ. Tangora
 KES 10/25/10 M 10/25/10
- T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 10-15-10 SAK 10/18/10 MAT 10-15-10

SOPs for Methods with Operating Conditions

(Document Operating Conditions in Maintenance Manual if Different Than Those Described in SOP)

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391-R07	Analysis of Volatile Organic Compounds by Method 802.1B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MT-0200-R19	✓ Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	✓ Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	✓ Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-OP-0367-R00	Microwave Extraction Procedure
TA-OP-0388-R07	Gel Permeation Chromatography Extract Clean-up Procedure (3600C and 3640A)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0158-R10	Total Halogens EPA 9076)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0186-R01	Total Halogens
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

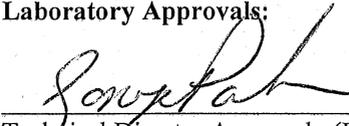
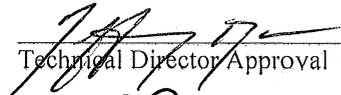
SOP: TA-___-___-R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00022

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10/26/10</u> Date
 Technical Director Approval (Ocana)	<u>10/26/10</u> Date
 Quality Assurance Approval	<u>10/28/10</u> Date
 Technical Director Approval (Ryan)	<u>10-27-10</u> Date
 Technical Director Approval/H & S Approval	<u>10/26/10</u> Date
 Laboratory Director Approval	<u>10/28/10</u> Date

1. Description Of and Reason For Decision:

As noted in an L-A-B DOD audit finding, the lab's instrumentation SOPs typically do not include sufficient detail pertaining to initial instrument calibration procedures as algorithms for all of the possible calibration models are not featured in those SOPs.

In addition to including the step-by step procedures employed to prepare and analyze the calibration standards, instrument SOPs also need to include a discussion of the possible calibration models as well as the applicable calibration algorithms or include a reference to corporate SOP CA-Q-S-005, Calibration Curves.

2. References: QSM 4.1, 5.5.2.2.1a

3. Others Notified (date and initial below your name):

Ao 10/26/10 ASM 10-26-10 KET 10/26/10 qd 10/26/10 AB 10-26-10
 A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway
 S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner, B/K
 10-27-10 KJS OM 10/27/10 MAT 10/27/10 22 10/27/10 22 10/27/10 22 10/27/10
 K. Johnson, D. Brechler, MJ, Tangora
 T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 10-27-10 SPK 10/27/10 10/27/10 MAT 10-29-10

Instrument SOPs with Inadequate Calibration Information

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0124-R09	Analysis of Total Phosphorus (EPA 365.1)
TA-WC-0156 R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0175-R09	Hexavalent Chromium (SM 3500 Cr-D)
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

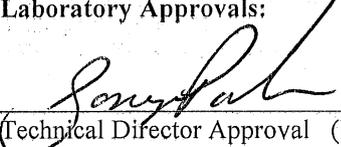
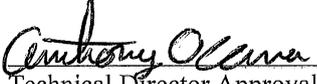
SOP: TA-____-____-R____ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00024

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 _____ Technical Director Approval (Palmer)	<u>10/29/10</u> Date
 _____ Technical Director Approval (Ryan)	<u>10-26-10</u> Date
 _____ Technical Director Approval (Ocana)	<u>10/27/10</u> Date
 _____ Technical Director Approval/H & S Approval	<u>10/28/10</u> Date
 _____ Quality Assurance Approval	<u>10/29/10</u> Date
 _____ Laboratory Director Approval	<u>10/28/10</u> Date

1. **Description Of and Reason For Decision:**

As noted in a L-A-B DOD audit finding, Gray Box 37 and Appendix F Tables state that for CCV failures, the laboratory shall reanalyze CCVs and all samples analyzed since the last successful CCV.

The applicable QC sections and tables in the attached SOPs need to reflect that CCV recoveries for DOD projects must fall within the DOD QSM-specified acceptance limits, regardless of observed bias or associated sample results and that for all CCV failures the lab shall reanalyze CCVs and all samples analyzed since the last successful CCV.

2. **References:** QSM 4.1, GB 37

3. **Others Notified (date and initial below your name):**

- AO* 10/27/10 *ASR* 10-27-10 *KBT* 10/27/10 *AP* 10/29/10 *F* 10/29/10 *PB* 10-29-10
A. Ocana, A. Mattison, K. Tefreau, S. Palmquist, F. Woo, P. Boardway
- J* *CM* 10/29/10 *AK* 10/29/10 *F* 10/29/10
S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner.
- KJS* 10-29-10 *CB* 10/29/10 *MJT* *AP* 10/29/10 *CK* 10/29/10 *SC* 10/29/10
K. Johnson, D. Brechler, MJ, Tangora
- T* Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
- 10-29-10* *SMW* 10/26/10 *MAT* 10-26-10

SOPs for DOD Certified Methods with CCV Requirements

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0122-R15	pH & Corrosivity (EPA 150.1, 9040B, 9045D and SM 4500-H+B)
TA-WC-0129-R11	Analysis of Conductivity (EPA 9050A and 120.1, Standard Method 2510B)
TA-WC-0138-R08	Corrosivity (EPA 9041A)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon In Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

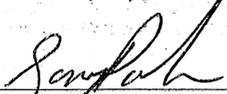
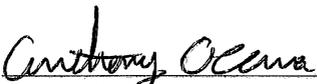
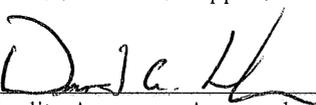
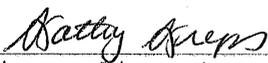
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00025

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10/29/10</u> Date
 Technical Director Approval (Ryan)	<u>10-26-10</u> Date
 Technical Director Approval (Ocana)	<u>10/27/10</u> Date
 Technical Director Approval/H & S Approval	<u>10/28/10</u> Date
 Quality Assurance Approval	<u>10/29/10</u> Date
 Laboratory Director Approval	<u>10/29/10</u> Date

1. **Description Of and Reason For Decision:**

As noted in an L-A-B DOD audit finding, Section 5.2.9d and Appendix D require measures to assess precision on a batch basis.

The applicable QC sections and tables in the attached SOPs need to reflect that a LCSD will be prepared, analyzed and evaluated when adequate sample volumes for MS/MSD are not provided.

2. **References:** QSM 4.1, 5.2.9d

3. **Others Notified (date and initial below your name):**

AO 10/27/10 AM 10-27-10 KB 10/27/10 J 10/28/10 RB 10-29-10
A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway

SP 10/29/10 AP 10/29/10 SK 10/29/10 BH
S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner.

KKJ 10-24-10 J 10/29/10 MJT
K. Johnson, D. Brechler, MJ, Tangcora

10-26-10 SK 10/26/10 MAT 10-26-10
T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen

SOPs for DOD Certified Methods

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0354-R01	PCBs in Transformer Oil
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504 1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-IP-0179-R11	Hexavalent Chromium Sample Preparation by Coprecipitation (EPA 7195 and 218 5)
TA-IP-0205-R16	Water Digestion Procedure for Total and Dissolved Metals Analysis (EPA 3005A, 3010A, 200 7, and 200 8)
TA-IP-0220-R06	Acid Digestion of Sediments, Sludges, and Soils (EPA 3050B)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-OP-0301-R12	Separatory Funnel Extraction
TA-OP-0302-R11	Sonication Extraction Procedure
TA-OP-0314-R01	Waste Dilution by EPA 3580A
TA-OP-0323-R15	Continuous Liquid-Liquid Extraction
TA-OP-0334-R11	High Temperature Sonication Extraction (Method 3550B Modified)
TA-OP-0367-R00	Microwave Extraction Procedure
TA-QA-0620-R02	Quality Control Program
TA-WC-0101-R10	Alkalinity by Titration (EPA 310.1 and SM 2320B)
TA-WC-0102-R14	5-Day Biochemical Oxygen Demand
TA-WC-0103-R11	High and Low Level Chemical Oxygen Demand (EPA 410.1, 410.2 and SM 5220C)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500-CN-E, G, and I)
TA-WC-0109-R11	Hardness Analysis (EPA 1302 and SM 2340 C)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0121-R10	n-Hexane Extractable Material (HEM) and Silica Gel Treated n-Hexane Extractable material (SGT-HEM) (EPA 1664)
TA-WC-0122-R15	pH & Corrosivity (EPA 150.1, 9040B, 9045D and SM 4500-H+B)
TA-WC-0125-R09	Determination of Solids in Waters and Wastes (SM 2540B, SM 2540C, SM 2540D, and SM 2540E)
TA-WC-0129-R11	Analysis of Conductivity (EPA 9050A and 120.1, Standard Method 2510B)
TA-WC-0138-R08	Corrosivity (EPA 9041A)
TA-WC-0154-R13	Static Flash Ignitability (EPA 1020A)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0186-R01	Total Halogens
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)



Title: Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology

Approvals:			
SIGNATURES ON FILE			
_____ Sonya Palmer Semivolatile Organics Department Manager	Date	_____ Stan Palmquist Health & Safety Manager / Coordinator	Date
_____ Dave Wunderlich Quality Assurance Manager	Date	_____ Kathy Kreps Laboratory Director	Date

This SOP was previously identified as SOP No. B-SOP-FLS-010.

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1.0 Scope and Application

- 1.1 This method is designed to measure the collective concentrations of different carbon ranges of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. The carbon ranges used throughout this document are given in equivalent carbon (EC) numbers which are related to the boiling point of a chemical normalized to the boiling point of the n-alkanes and its retention time in a boiling point gas chromatographic column.

Extractable aliphatic and aromatic hydrocarbons are collectively quantitated separately within five ranges:

C₈-C₁₀
>C₁₀-C₁₂
>C₁₂-C₁₆
>C₁₆-C₂₁
>C₂₁-C₃₄

These aliphatic and aromatic hydrocarbons ranges correspond to a boiling point range between approximately 150°C and 500°C.

- 1.2 Petroleum products suitable for evaluation by this method include, but are not limited to, kerosene and jet fuels, diesel and fuel oils, and hydraulic, insulating and lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, and other petroleum products which contain a significant percentage of hydrocarbons lighter than C₁₀. When samples are known or suspected to contain petroleum hydrocarbons of these or similar types, the Volatile Petroleum Hydrocarbon (VPH) method should also be employed to fully evaluate the hydrocarbons present.
- 1.3 The practical quantitation limits (PQLs), based upon a 1000mL or 30g initial sample amount, 2 mL final extract volume and assuming a minimum of 50% solids for soil/sediments are:

Water -50.0 ug/L for each aliphatic or aromatic carbon range
Soils - 5.0 mg/Kg for each aliphatic or aromatic carbon range

If lower quantitation limits are desired, the analyst is allowed to extract larger sample amounts and/or to concentrate the extracts to smaller volumes prior to analysis. Sample amounts greater than 50g are not recommended when using the sonication procedure. Concentration of extracts to final volumes of less than 1.0 ml is also not recommended.

- 1.4 This method is based on a solvent extraction, silica gel fractionation process and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in

extractable organics analysis. Analysts using this method should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

- 1.5 Like all GC procedures, this method is subject to a "false positive" bias, in that non-hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated with the respective carbon ranges. While the cleanup procedure specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analyses by gas chromatography/mass spectrometry (GC/MS) analysis (i.e. EPA method 8270C) or other suitable techniques are recommended in cases where significant concentrations of non-hydrocarbon compounds are known or suspected. Non-petroleum compounds identified and quantitated by GC/MS may be subtracted from the carbon ranges affected as long as the quantity and identities of the compounds are reported along with the carbon range data.
- 1.6 On occasion, clients may request slight modifications to this SOP. These modifications are addressed on a case by case basis with the supporting demonstration of sensitivity and accuracy (e.g. MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into project special instructions (e.g. Quality Assurance Project Plans), authorized by the laboratory, and mentioned in the report narrative.

2.0 Method Summary

- 2.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated. Sample cleanup and separation into aliphatic and aromatic fractions is conducted using a modification of EPA Method 3630 Silica Gel Cleanup (SW-846) (SOP TA-OP-0364). The two extracts produced (an aliphatic extract and an aromatic extract) are then concentrated to final volumes for analysis and separately analyzed using a gas chromatograph equipped with a capillary column and a flame ionization detector (FID). The resulting aliphatic and aromatics chromatograms are integrated in sections based upon the beginning and ending points of the targeted carbon ranges.
- 2.2 The beginning and ending points of the targeted equivalent carbon ranges are determined by analysis of retention time reference standards containing normal alkanes.
- 2.3 Average calibration factors or response factors determined using an aliphatic hydrocarbon standard mixture are used to calculate the collective concentrations of the different aliphatic hydrocarbons ranges. Average calibration or response factors determined using an aromatic hydrocarbon standard mixture are used to calculate the collective concentrations of the aromatic hydrocarbon ranges.
- 2.4 This method is suitable for the analysis of waters, soils, and sediments.

3.0 Definitions

- 3.1 Extractable Petroleum Hydrocarbons (EPH) - All hydrocarbon compounds eluting from toluene through benzo(g,h,i)perylene. EPH is comprised of C₈ through C₁₀, >C₁₀ through C₁₂, >C₁₂ through C₁₆, >C₁₆ through C₂₁ and >C₂₁ through C₃₄ Aliphatic Hydrocarbons and C₈ through C₁₀, >C₁₀ through C₁₂, >C₁₂ through C₁₆, >C₁₆ through C₂₁ and >C₂₁ through C₃₄ Aromatic Hydrocarbons. EPH concentration data are reported as the aggregate concentration of the aliphatic and aromatic hydrocarbon ranges.
- 3.2 Volatile Petroleum Hydrocarbons (VPH) - All hydrocarbon compounds eluting just prior to n-pentane (n-C₅) through 1-methylnaphthalene. VPH is comprised of C₅ through C₆, >C₆ through C₈, >C₈ through C₁₀, and >C₁₀ through C₁₂ Aliphatic Hydrocarbons, >C₈ through C₁₀, >C₁₀ through C₁₂, and >C₁₂ through C₁₃ Aromatic Hydrocarbons, and benzene and toluene. VPH concentration data are reported as the aggregate concentrations of the aliphatic and aromatic hydrocarbon ranges and as selected targeted analytes.
- 3.3 Equivalent Total Petroleum Hydrocarbons (E-TPH) - The summation of the EPH value and the VPH value with correction for overlapping carbon ranges:
- 3.3.1 For samples contaminated only with petroleum products heavier than C₁₀, the E-TPH value is equivalent to the EPH value.
- 3.3.2 For samples contaminated only with petroleum products lighter than C₁₂, the E-TPH value is equivalent to the VPH value.
- 3.3.3 For samples contaminated with petroleum hydrocarbons containing significant concentrations of hydrocarbons in both the EPH and VPH ranges (i.e. contaminated with both gasoline and diesel fuel) the E-TPH value is equal to the sum of the EPH and VPH values *minus* the lower of the two values in the overlapping (C₁₀ to C₁₂) aliphatic and aromatic range. In other words, the *higher* value is used from each fraction in the overlapping range when summing the EPH and VPH results.
- 3.4 C₈ through C₁₀ Aromatic Hydrocarbons – All aromatic hydrocarbon compounds eluting from (and including) toluene through 1,2,3-trimethylbenzene.
- 3.5 >C₁₀ through C₁₂ Aromatic Hydrocarbons – All aromatic hydrocarbon compounds eluting after 1,2,3-trimethylbenzene through naphthalene.
- 3.6 >C₁₂ through C₁₆ Aromatic Hydrocarbons – All aromatic hydrocarbon compounds eluting after naphthalene through acenaphthene.
- 3.7 >C₁₆ through C₂₁ Aromatic Hydrocarbons – All aromatic hydrocarbon compounds eluting after acenaphthene through pyrene.

- 3.8 >C₂₁ through C₃₄ Aromatic Hydrocarbons – All aromatic hydrocarbon compounds eluting after pyrene through benzo(g,h,i)perylene.
- 3.9 C₈ through C₁₀ Aliphatic Hydrocarbons – All aliphatic hydrocarbon compounds eluting from (and including) n-octane (n-C₈) through n-decane (n-C₁₀).
- 3.10 >C₁₀ through C₁₂ Aliphatic Hydrocarbons – All aliphatic hydrocarbon compounds eluting after n-decane through n-dodecane (n-C₁₂).
- 3.11 >C₁₂ through C₁₆ Aliphatic Hydrocarbons – All aliphatic hydrocarbon compounds eluting after n-dodecane through n-hexadecane (n-C₁₆).
- 3.12 >C₁₆ through C₂₁ Aliphatic Hydrocarbons – All aliphatic hydrocarbon compounds eluting after n-hexadecane through n-henicosane (n-C₂₁).
- 3.13 >C₂₁ through C₃₄ Aliphatic Hydrocarbons – All aliphatic hydrocarbon compounds eluting after n-henicosane through tetratriacontane (n-C₃₄).
- 3.14 Aromatic Hydrocarbon Standard – A mixture (plus surrogate) of aromatic hydrocarbons used to define and establish the windows for the Aromatic Hydrocarbon ranges and determine chromatographic response factors that can in turn be used to calculate the collective concentration of aromatic hydrocarbons in environmental samples within those hydrocarbon ranges.
- 3.15 Aliphatic Hydrocarbon Standard – A mixture (plus surrogate) of the normal alkanes used to define and establish the windows for the Aliphatic Hydrocarbon ranges and determine chromatographic response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.
- 3.16 Analytical Batch – A group of 20 or less samples extracted and processed together within the same shift using the same reagents. Each batch must contain a minimum QC of a method blank, laboratory control sample, matrix spike and sample duplicate. If insufficient sample is available, a laboratory control sample duplicate should be included in each analytical batch.
- 3.18 Field Duplicates - Two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures. The analysis of field duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.
- 3.19 Calibration Standards - A series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compounds of interest.
- 3.20 Calibration Check Standard (CCS) - A calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is

prepared from the same stock standard solution as the calibration standards and is generally one of the mid-range calibration standard dilutions.

- 3.21 Matrix Spiking Solution - A solution which is prepared independently from the calibration standards and which contains known concentrations of method analytes.
- 3.22 Laboratory Method Blank – Depending on the matrix of the samples, either reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is prepared and analyzed along with all associated samples in a single batch. It is exposed to all glassware, solvents, reagents and equipment. At least one laboratory method blank is analyzed with every batch of samples to determine if method analytes or other interferences are present in the laboratory environment, reagents or equipment.
- 3.23 Laboratory Fortified Blank/Blank Spike/Laboratory Control Sample (LFB/LCS/LCS) - Depending on the matrix of the samples, either reagent water or clean sand blank fortified with a matrix spiking solution. This control sample is prepared and analyzed along with all associated samples in a single batch. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required practical quantitation limits.
- 3.24 Laboratory Fortified Matrix/Matrix Spike (LFM/MS) - An environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. This control sample is prepared and analyzed along with all associated samples in a batch. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the LFM/MS sample corrected for background concentrations.
- 3.25 Calibration Verification Standard (CVS): A commercially prepared and certified quality control standard from a source other than that used to prepare the Calibration standards. This second source standard is used as a quality control check to verify the accuracy of the external calibration.
- 3.26 Fractionation Check Solution: The Fractionation Check Solution is used to monitor the fractionation efficiency of different batches of silica gel. See SOP *TA-OP-0364*
- 3.27 Fractionation Surrogate Check Standard: 5,6,7,8-Tetrahydrol-1-naphthol. Used to monitor the fractionation process of the samples and batch QC. Recovery of 5,6,7,8-Tetrahydrol-1-naphthol should be less than 10% in the aliphatic and aromatic extracts.

4.0 Interferences and Comments

- 4.1 In order to eliminate contaminants from glassware, all glassware must be cleaned in accordance with SOP *TA-QA-0010* prior to being utilized.
- 4.2 High purity reagents must be used to minimize interference problems.
- 4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of a solvent blank to check for cross-contamination.
- 4.4 Matrix interferences may be caused by contaminants that are co-extracted along with the analytes of interest from the sample. The type and extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled and may include certain solvents, halogenated hydrocarbons and phthalate esters.
- 4.5 Alumina is also frequently used to separate petroleum distillates into aliphatic and aromatic fractions. An alternate method for performing the fractionation is given in SW-846 Method 3611, *Alumina Column Cleanup and Separation of Petroleum Wastes*. Use of the alumina fractionation technique is an acceptable alternative to the silica gel technique listed in this method.
- 4.6 The capacity of silica gel can be overloaded by high concentrations of petroleum or non-petroleum hydrocarbon mixtures. 10 grams of silica gel can normally handle more than 50 mg of neat petroleum hydrocarbons. Comparison of column capacity against existing TPH data, however, can be misleading if high levels of interfering compounds or non-petroleum hydrocarbons are present. Overloading yields inadequate fractionation and tends to produce high variability between the various aliphatic and aromatic carbon ranges for multiple analyses of a given sample. Laboratories performing fractionation should be able to demonstrate that the sample extract is not overloading the fractionation column during the procedure.
- 4.7 The laboratory analyst must perform the method in accordance with this SOP. The analyst will resolve non-conformances in methods and data, either individually, or with the assistance of the Department Supervisor or Operations Manager. Deviations from this SOP must be documented. Bench sheets and raw data must capture information related to a deviation. The laboratory analyst or supervisor will report deviations or non-conforming events to the operations, project and/or QA manager via a non-conformance report.
- 4.8 The Department Supervisor, Operations Manager, and/or QA Manager will assist the laboratory analyst in resolving non-conformances.
- 4.9 The Department Supervisor or designee will review and approve data, data qualifiers, non-conformance reports, methodology, logbooks and final reports for all analyses performed in his/her department.

- 4.10 The QA Manager shall verify adherence to this SOP through annual audits, non-conformance reports, and performance evaluation studies.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum. Specific safety concerns or requirements as they related to this procedure include:

- 5.1 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.2 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degrades the skin. May be absorbed through skin.

Methanol	Flammable Poison Irritant	200 ppm- TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm- TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas Chromatography

6.1.1 Gas Chromatograph - Hewlett Packard 6890 or 5890 Series II Gas Chromatograph or equivalent

An analytical system complete with temperature programmable gas chromatograph for use with capillary columns. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

6.1.2 *Column: 30-m long x 0.25-mm I.D., 0.1- μ m film ZB-1 column (Zebron) or equivalent. This column will allow for the adequate resolution of alkanes from n-C₈ to n-C₃₄.*

6.1.3 Detector: Flame Ionization Detector (FID).

6.1.4 Autosampler: An autosampler capable of making 1 to 2 μ L injections is recommended.

6.1.5 *Data system: One component of the system, Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. Since no processing is done by Chemstation and since there are no audit trail functions associated with data acquisition, the audit trail feature for Chemstation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.*

6.2 Analytical balance, capable of accurately weighing 0.1 mg.

6.3 Autosampler vials with seals

6.4 Microsyringes: 10- μ L, 100- μ L, 250- μ L, 500- μ L, 1000- μ L, accurate to \pm 5%

6.5 Volumetric flasks, Class A: 10, 50, 100, 500 mL

7.0 Reagents and Standards

All reagents and standards used in this procedure must conform to the requirements specified in *TA-QA-0030 Rev 11 Verification of Quality of Materials*. Preparation of reagents and standards will be documented in the LIMS and a label will be generated to include, as appropriate: the name and expected concentration of the material; the generated LIM's ID number; the date of preparation; the expiration date and the analyst's initials. All initial instrument calibrations must be verified with a standard obtained from a second manufacturer. If a standard from a second manufacturer is not available, verification may be achieved by using a different lot from the primary manufacturer. However, the supervisor must obtain written warranties that the two references were not prepared from the same reference material. *Certificates of Analysis or Traceability must be scanned and attached to the standard information in TALS.*

All standards prepared by the laboratory must be stored per manufacturer's recommendations. Stock standards and calibration standards must be replaced within 6 months of preparation. Standards that are purchased pre-made from commercial suppliers may be kept for the life, and under conditions, specified by the manufacturer. Standards should be brought to room temperature prior to use. **DO NOT PLACE VOLUMETRIC GLASSWARE IN AN OVEN.**

7.1 Calibration Standard Solutions

7.1.1 Aliphatic Hydrocarbon Standard: The Aliphatic Hydrocarbon Standard consists of all normal alkanes from Octane through Tetracontane (listed in Table 1). Pristane and Phytane may be included if desired. The aliphatic surrogates (see Table 3) may also be added to this mixture. Prepare individual stock standard solutions by accurately weighing pure (assayed to $\geq 96\%$ w/w) material to at least three significant digits and dissolving in hexane or 3:1 carbon disulfide/methylene chloride. Volumetrically combine individual stocks and dilute to volume as necessary to obtain a combined stock solution containing all compounds at appropriate concentrations (i.e. 500 to 1000 $\mu\text{g/mL}$ each). The use of commercially prepared stock solutions or combined mixtures is recommended (e.g. AccuStandard Cat. No. DRH-008S). Neats expire after two years. Prepared or purchased stock standards expire after one year (or on the expiration date provided by the manufacturer, if sooner). Dilute this combined stock solution as needed to produce the calibration standards for aliphatic hydrocarbons.

Table 1. Aliphatic Hydrocarbon Standard

Octane	Heptadecane	Tetracosane	Tritriacontane
Nonane	Pristane (optional)	Pentacosane	Tetratriacontane
Decane	Octadecane	Hexacosane	Pentatriacontane
Undecane	Phytane (optional)	Heptacosane	Hexatriacontane
Dodecane	Nonadecane	Octacosane	Heptatriacontane
Tridecane	Eicosane	Nonacosane	Octatriacontane
Tetradecane	Heneicosane	Triacontane	
Pentadecane	Docosane	n-Hentriacontane	Tetracontane
Hexadecane	Tricosane	Dotriacontane	

- 7.1.2 Aromatic Hydrocarbon Standard: The Aromatic Hydrocarbon Standard consists of the 16 aromatic compounds listed in Table 2. The aromatic surrogate (see Table 3) may also be included. Prepare individual stock standard solutions by accurately weighing pure (assayed to $\geq 96\%$ w/w) material to at least three significant digits and dissolving in methylene chloride. Volumetrically combine individual stocks and dilute to volume as necessary to obtain a combined stock solution containing all compounds at appropriate concentrations (i.e. 500 to 2000 $\mu\text{g/mL}$ each). The use of commercially prepared stock solutions or combined mixtures is recommended (i.e. PAH Mix AccuStandard Cat. No. Z-014G. Toluene Accustandard Cat. #M-502-46-10X). Neats expire after two years. Prepared or purchased stock standards expire after one year (or on the expiration date provided by the manufacturer, if sooner). Dilute this combined stock solution as needed to produce the calibration standards for aromatic hydrocarbons.

Table 2. Aromatic Hydrocarbon Standard

Acenaphthene	Benzo(g,h,i)perylene	Dibenz(a,h)anthracene
Acenaphthylene	Benzo(k)fluoranthene	Naphthalene
Anthracene	Chrysene	Phenanthrene
Benzo(a)anthracene	Fluoranthene	Pyrene
Benzo(a)pyrene	Fluorene	Toluene (Added when working standard is prepared.)
Benzo(b)fluoranthene	Indeno(1,2,3-cd)pyrene	

- 7.1.3 Surrogate Standards: This method requires the use of at least three surrogates. These surrogates must include a low-end *aromatic* compound, a mid-range aliphatic compound and a high-end aromatic compound. The recommended surrogate compounds are listed in Table 3. Other surrogate compounds may be used in place of the recommended compounds if they:

1. Elute within the same carbon range as the recommended surrogate
2. Separate ($>95\%$) into the same fraction as the recommended surrogate

Prepare individual stock surrogate solutions by accurately weighing pure (assayed to $\geq 96\%$ w/w) material to at least three significant digits and dissolving in methylene chloride. To simplify calibration standard preparation, it is recommended that the single *aliphatic* surrogate be combined with the *Aliphatic* Hydrocarbon Standard when preparing stock solutions. The *aromatic* surrogate compounds may be included in the *aromatic* calibration standard as well. The use of commercially prepared stock solutions or combined mixtures is recommended. Neats expire after two years. Prepared or purchased stock standards expire after one year (or on the expiration date provided by the manufacturer, if sooner).

Table 3. Surrogate Compounds

Aromatic	o-Terphenyl (<i>OTP</i>) p-Terphenyl-d ₁₄
Aliphatic	1-Chlorooctadecane (<i>COD</i>)

As samples for EPH analysis can be batched with samples for EPA 8270C High Volume analysis, the laboratory has combined the surrogates for the EPH analysis and the EPA 8270C HV analysis into a single surrogate spiking solution. The components for this solution include:

- 7.1.3.1 1-Chlorooctadecane (COD) (neat) – Available from ACROS or equivalent.
 - 7.1.3.2 o-Terphenyl (OTP) (neat) – Alfa Aesar Cat No A19680 or equivalent.
 - 7.1.3.3 p-Terphenyl-d₁₄ Stock Solution – 2000 ug/mL in MeCl₂. Ultra Scientific Cat No ATS-160 or equivalent.
 - 7.1.3.4 1-Methylnaphthalene-d₁₀ Stock Solution – 1000 ug/mL in Methanol. Absolute Cat No 71221 or equivalent.
 - 7.1.3.5 Benzo(a)pyrene-d₁₂ Stock Solution – 1000 ug/mL in MeCl₂. Absolute Cat No 71739.
- 7.2 Surrogate Spiking Solution: The recommended surrogate spiking solution is comprised of a mixture of the COD and OTP surrogate standards. The laboratory is currently combining the EPH and EPA 8270C HV surrogates into one spiking solution. Each sample, blank, and spike is fortified with the surrogate spiking solution.
- 7.2.1 Combine 0.25g of 1-Chlorooctadecane (7.1.3.1), 0.25 g of o-Terphenyl (7.1.3.2), 10 mLs of p-Terphenyl-d₁₄ Stock Solution (7.1.3.3), 0.5mL of 1-Methylnaphthalene-d₁₀ Stock Solution (7.1.3.4) and 0.5mL of Benzo(a)pyrene-d₁₂ Stock Solution (7.1.3.5) in MeCl₂ and bring to 500 mL final volume.

- 7.2.2 The final concentration of the surrogate components is 500 ppm each of COD and OTP, 40ppm of p-Terphenyl-d14, and 1ppm each of 1-Methylnaphthalene-d10 and Benzo(a)pyrene-d12).
- 7.3 Petroleum Performance Check Standard: The use of a Petroleum Performance Check Standard is recommended for demonstration of analytical accuracy and sensitivity following initial calibration. The Petroleum Performance Check Standard for routine sample analysis consists of a mixture of diesel fuel and motor oil combined at a ratio of 1:4 and diluted to final concentration in methylene chloride or hexane. This standard expires after one year. For routine application of this method to the standard carbon ranges, the final concentration of the Petroleum Performance Check Standard should be 250 µg/mL (50 µg/mL diesel / 200 µg/mL motor oil). Other products and/or combinations of products may be more appropriate for different projects or types of samples. The final summed concentration of the check standard should not be greater than 250 µg/mL.
- 7.4 Aliphatics Calibration Verification Standard (Second Source): A commercially prepared, certified quality control standard used to verify the accuracy of the external calibration. The Aliphatic Second Source consists of all normal alkanes from Octane through Tetracontane (listed in Table 1). Pristane and Phytane may be included if desired. The use of commercially prepared stock solutions or combined mixtures is recommended (e.g. Ultra Cat. No. SFL-601). Purchased stock standards expire after one year (or on the expiration date provided by the manufacturer, if sooner). Dilute this stock solution as needed to produce the CVS at the midpoint of the initial calibration.
- 7.5 Aromatics Calibration Verification Standard (Second Source): A commercially prepared, certified quality control standard used to verify the accuracy of the external calibration. The Aromatics Second Source consists of all aromatic compounds listed in Table 2. The use of commercially prepared stock solutions or combined mixtures is recommended (e.g. Ultra Cat. No. US-106N). Purchased stock standards expire after one year (or on the expiration date provided by the manufacturer, if sooner). Dilute this stock solution as needed to produce the CVS at the midpoint of the initial calibration.
- 7.6 Methylene Chloride, pesticide grade. Label all vials and squeeze bottles with Lot Number, chemical name and NFPA chemical hazard label.
- 7.7 *Analysts are expected to check those areas where standards are stored on a monthly basis and dispose of expired standards according to sec. 14.1.3.*
- 8.0 Sample Collection, Preservation, Shipment and Storage**
- 8.1 Aqueous samples are collected in 1 liter amber glass bottles with PTFE-lined screw caps.

- 8.2 Soil and sediment samples are collected in 4 oz. (120 mL) or 8 oz. (250 mL) wide-mouth glass jars with PTFE-lined screw caps.
- 8.3 Aqueous samples should be preserved at the time of sampling by the addition of an acid to reduce the pH of the sample to less than 2.0. This is accomplished by the addition of approximately 5 mL of 1:1 HCl to a 1 liter sample. Confirm pH preservation, after mixing, by transferring one or two drops of sample via disposable transfer pipette onto a strip of wide range pH paper. Following collection and the addition of acid, the sample must be cooled to 0-6°C.
- 8.4 Soil and sediment samples must be cooled to 0-6°C immediately after collection.
- 8.5 Preserved aqueous samples must be extracted within 14 days of collection. Unpreserved aqueous samples must be extracted within 7 days of collection. Soil and/or sediment samples must be extracted within 14 days of collection. Both aqueous and soil/sediment extracts must be analyzed within 40 days of extraction.
- 8.6 A summary of sample collection, preservation, and holding times is provided in Table 4.

Table 4. Holding Times and Preservatives for EPH Samples

Matrix	Container	Preservation	Holding Time
Aqueous Samples	1-Liter amber glass bottle with PTFE-lined screw cap	Add 5 mL of 1:1 HCl; cool to 0-6°C	Samples must be extracted within 14 days and extracts analyzed within 40 days
Soil/Sediment Samples	4-oz. (or larger) wide mouth glass jar with PTFE-lined screw cap	Cool to 0-6°C	Samples must be extracted within 14 days and extracts analyzed within 40 days

- 8.7 Water samples are prepared according to SOP TA-OP-0323 (EPA 3520C) or SOP TA-OP-0301 (EPA 3510C).
- 8.8 Soil samples are prepared according to SOP TA-OP-0302 (EPA 3550B)
- 8.9 Extracts are fractionated according to SOP TA-OP-0315.

9.0 Quality Control

- 9.1 Initial Calibration

- 9.1.1 The percent RSD for the initial calibration must be $\leq 20\%$. If the %RSD is $>20\%$, linear or nonlinear modeling may be attempted. The correlation coefficient (r) must be ≥ 0.995 for all linear or nonlinear calibration curves.
- 9.1.2 When linear regression is used, the intercept value should be checked to ensure that it is not at a level that is equivalent to or higher than the lowest calibration standard used.
- 9.1.3 A second source must be analyzed and have %D $\leq 20\%$ for hydrocarbon ranges.
- 9.1.4 If acceptance criteria are not achieved, corrective action must be taken and a new initial calibration completed.
- 9.1.5 Any samples associated with a failed initial calibration must be reanalyzed.
- 9.2 Continuing Calibration Verification
 - 9.2.1 Calibration verification must be completed prior to sample analysis, after every ten samples and at the end of the analytical sequence.
 - 9.2.2 The CFs for the respective hydrocarbon ranges in the continuing verification must not vary more than 20% for from those established by the initial calibration.
 - 9.2.3 In the event of calibration verification failure, corrective action must be taken prior to sample analysis. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications (using fresh calibration solutions, at low and high concentrations) or, alternatively, a new initial calibration must be established. If one of these calibration verification injections fails, a new initial calibration curve must be processed.
 - 9.2.3.1 Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present. Re-analysis is required for all other situations. If for some reason (i.e., lack of sample) re-analysis can't take place, a NCM needs to be initiated and the sample and QC results associated with the failing CCV need to qualified in the final report.

9.2.3.2 If the sequence of samples preceding a failing closing CCV includes samples suspected of matrix interference and interference-free samples such as blanks and blank spikes, then the samples analyzed prior to the failing closing calibration verification need to be segregated for re-analysis. The interference-free samples should be analyzed within one set of bracketing CCVs, and the samples with suspected interference should be analyzed within another set of bracketing CCVs.

9.2.4 Mass Discrimination Check: The first Aliphatic HydroCarbon Continuing Calibration Check Standard of an analytical sequence must be evaluated for mass discrimination. Mass discrimination is calculated as shown in Equation 4. If the mass discrimination is greater than 30%, the instrument parameters must be adjusted to obtain a more consistent response over the full calibration range. The mass discrimination objective must be met prior to further sample analysis.

Equation 4: Mass Discrimination Calculation

$$\text{Mass Disc. Value} = \left[\frac{(A_{C_{30}} - A_{C_{12}})}{(A_{C_{30}} + A_{C_{12}})/2} \right] \times 100$$

where: $A_{C_{30}}$ = Area of triacontane peak (n-C₃₀)
 $A_{C_{12}}$ = Area of dodecane peak (n-C₁₂)

9.3 Surrogates

9.3.1 Surrogate percent recovery is monitored to assess method performance on the particular matrix. Surrogates are added to all samples, blanks, and spikes prior to extraction. Surrogates are also added to all calibration and check standards.

9.3.2 Compare the %R of the surrogate recoveries to the method-specified limits of 60-140%.

9.3.3 If a surrogate recovery for a method blank is outside of control limits (low or high), but the blank is clean, the surrogate and target analyte recoveries in the LCS are acceptable and associated samples have acceptable surrogate recoveries and are non-detect for the target analyte, initiate an NCM and qualify the surrogate in the method blank. Otherwise, re-fractionate/re-extract/re-analyze some or the entire batch, depending on the exception.

9.3.4 If a surrogate recovery for a LCS is outside of control limits (low or high), initiate a NCM and consult with supervisor and quality assurance to determine the appropriate course of action.

- 9.3.5 If a surrogate recovery for a matrix spike is outside of control limits (low or high), but the method blank is clean and the surrogate and target analyte recoveries in the LCS are acceptable, initiate a NCM and qualify the surrogate in the matrix spike.
- 9.3.6 If surrogate recoveries are outside the control limits for a sample, and are not obviously due to matrix effects, (e.g. fractionation issues, high levels of target analytes, large dilutions, coeluting matrix interferences or high levels of non-target analytes), initiate a NCM and re-fractionate/re-prepare/re-analyze the sample. If a surrogate recovery is still outside control limits, qualify the surrogate in the sample. No re-analysis is required if the sample was chosen for the MS/MSD analysis and the MS and/or MSD are outside of limits. If additional sample is not available, the analyst must report the occurrence of the out-of-control surrogates with the analytical results.

If reanalysis solves the problem, then only submit the sample data from the analysis with surrogate spike recovery within acceptable limits. If, after appropriate corrective action, the surrogate recovery is still out of control, the data will be flagged as out of control due to matrix interference. Surrogate recoveries below the low cal point will be flagged as below the quantitation limit (and/or diluted out of range) and no value reported.

- 9.4 Retention Time Window Verification – Establish new retention time windows each after a new column is installed according to the procedures described in section 10.1.2.
- 9.5 Petroleum Performance Check Standard – Calculate the total concentration of all petroleum hydrocarbons within each range and add the values together. The concentration calculated is expected to be within $\pm 30\%$ of the known concentration.
- 9.6 Minimum Instrument QC
- 9.6.1 The instrument must be able to achieve adequate separation and resolution of peaks of interest.
- 9.6.2 The n-octane (n-C8) and toluene peaks must be adequately resolved from the solvent front of the chromatograms.
- 9.6.3 The surrogates used must be adequately resolved from any individual components in the Aliphatic HydroCarbon and Aromatic HydroCarbon standards.

9.7 Batch QC

At a minimum, for each analytical batch (up to 20 samples), a Method Blank, Blank Spike (LCS), Matrix Spike and Sample Duplicate must be analyzed. The blank, spikes and duplicate samples must be carried through all stages of the sample preparation and measurement process.

9.7.1 Method Blank

9.7.1.1 The method blank must be analyzed on each GC system used for analysis.

9.7.1.2 A *method blank* or an instrument blank *spiked with surrogate* needs to be analyzed after each CCV. If a method blank wasn't analyzed after the opening CCV, process an instrument blank to demonstrate the instrument is contamination-free.

9.7.1.3 Method and/or instrument blanks will be considered acceptable if:

9.7.1.3.1 No contaminants are found above the MRL , or

9.7.1.3.2 Contaminates are found and one of the following apply

- no associated analytes are found in the sample(s)
- The contamination is less than *10 times* the concentration found in the sample(s)

9.7.1.4 If a method or instrument blank is re-analyzed to confirm contamination, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (e.g. bent autosampler needle, etc.)

9.7.1.5 If method or instrument blank failure occurs, samples with results greater than the method reporting limit must be re-prepared and re-analyzed, unless the contamination present in the blank is less than 10 times of the concentration present in a sample. If the samples cannot be reprocessed, the blank and all associated samples must be flagged for blank contamination.

9.7.1.6 Blank subtraction must not be applied as a correction to sample analysis.

- 9.7.1.7 When analytes are detected in a method blank at levels between the method detection limit (MDL) and the method reporting limit (MRL), any results for the analyte in client samples should be qualified as potential laboratory contaminants if they are less than two times the level found in the blank.
- 9.7.2 Blank Spikes (LCS)
- 9.7.2.1 The LCS must be spiked with the surrogate solution and the spiking solution, and taken through the entire preparative process. Recovery of target analyte(s) should fall within an advisory range of 70 – 130% for all ranges except the C₈-C₁₀ aliphatic and C₈-C₁₀ aromatic. The blank spike recovery of the C₈-C₁₀ aliphatic and C₈-C₁₀ aromatic should fall within the advisory range of 50-150%.
- 9.7.2.2 If the recovery for a LCS is low and outside of control limits or high and outside of control limits with detections for the target analyte in the associated samples, initiate a NCM and re-fractionate/re-prepare/re-analyze the affected samples.
- 9.7.2.2.1 When reprocessing is not possible, *all QC and batch samples must be qualified for the analyte that failed in the LCS.*
- 9.7.2.3 If the recovery for a LCS is high and outside of control limits and the target analyte wasn't detected in the associated samples, initiate a NCM and qualify the analyte in the LCS, other QC and samples.
- 9.7.2.4 If a LCS/LCSD pair fails to meet acceptance criteria for precision (RPD), but recoveries for the LCS or LCSD are within control limits, initiate a NCM and qualify the analyte in the LCS, LCSD, other QC and samples.
- 9.7.2.5 If batch QC samples are re-analyzed to confirm recovery, and an improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this protocol would be if an obvious analytical problem occurred during the initial analysis (e.g. bent autosampler needle, etc.)
- 9.7.3 Matrix Spikes, Matrix Spike Duplicates (MS/MSD) and Sample Duplicates
- 9.7.3.1 TA-S policy expects that a Dup and MS be completed with every extraction batch if sufficient sample volume is available. Sample volume must not be split to facilitate a

- MS/MSD/duplicate analysis if sufficient volume has not been provided. If sufficient sample volume is available, prepare the matrix spike and the sample duplicate from the same source. If there is not enough of one sample for both QC samples, a second source can be used for either the matrix spike or the sample duplicate.
- 9.7.3.2 If sufficient volume is not available for a Matrix Spike or Duplicate, prepare and analyze a LCSD in its place and document the lack of sufficient sample volume for batch QC in a NCM.
- 9.7.3.3 The Dup and MS must be spiked with the surrogate solution and the MS must be spiked with the spiking solution, and taken through the entire preparative process.
- 9.7.3.4 Recovery of target analyte(s) in the Matrix Spikes must fall within 70-130%. Due to possible matrix interferences, acceptance failures should not be considered as grounds for data rejection.
- 9.7.3.5 The RPD must fall within $\pm 25\%$. Due to possible matrix interferences, acceptance failures should not be considered as grounds for data rejection.
- 9.7.3.6 Due to possible matrix interferences, accuracy failures relative to the matrix spike or precision failures relative to the duplicate should not be considered as grounds for data rejection. In these cases, the source sample and the QC sample (Dup and/or MS) should be qualified. Re-analysis of a Dup or MS for a failing recovery or RPD is not required and should not be undertaken.
- 9.7.4 Fractionation Check - To demonstrate the capability to properly fractionate aliphatic and aromatic hydrocarbons using this method, the analyst must prepare and analyze the Fractionation Check Solution along with each analytical batch of samples.
- 9.7.4.1 For each analyte within the Fractionation Check Solution, the percent recovery must be between 70 % and 130 %. Failure to meet this criteria indicates the need for review and/or re-development of the fractionation procedure. The aliphatic/aromatic fractionation is a critical component of this analytical method. Do not report analytical data for samples associated with a failed fractionation check.
- 9.8 Anomalous situations occurring during sample preparation and/or analysis must be documented on the bench sheet, and non-conformance reports must be issued if necessary. Refer to the SOP for Non-Conformances, TA-QA-0610.

9.9 The accuracy and precision of the bottle top dispenser (for methanol) must be checked at regular intervals according to SOP TA-QA-0016

9.10 Syringes must be calibrated every six months or replaced. See SOP TA-QA-0016

10.0 Procedure

10.1 Gas Chromatographic Analysis

10.1.1 Suggested Gas Chromatographic Conditions

10.1.1.1 *Oven Program: Set oven temperature to 45°C for 0.5 minutes, then 30°C/min to 330°C and hold for 6 minutes.*

10.1.1.2 Sample/autosampler injection volume is 1 µL.

10.1.1.3 Gas Flows: The recommended Carrier gas is nitrogen.

Nitrogen Carrier gas flow: 5 mL/min.

Air: 400 mL/min.

Hydrogen: 35 mL/min.

Make up gas flow: 30 mL/min.

10.1.1.4 Miscellaneous:

FID temperature: 300°C

Injection port temperature: 280°C

Injector mode: Splitless

Column head pressure: 15.0 psi at 50°C

Linear velocity: Approximately 50 cm/sec

10.1.2 Retention Time Windows

10.1.2.1 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Before establishing windows, make sure the GC system is within optimum operating conditions. Make at least three injections of the mid-level Aliphatic and Aromatic HydroCarbon Standards throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.

10.1.2.2 Calculate the standard deviation of the three absolute retention times for the surrogate compounds and the aliphatic and aromatic compounds which correspond to the start and end points of the equivalent Carbon ranges used in the method.

- 10.1.2.3 The retention time window for each compound is defined as the mean of the absolute retention time plus or minus three times the standard deviation of the retention times measured from the three (or more) standard injections. A default retention time window may be assigned to the individual compounds after calculating and documenting actual retention time windows which are less than or equal to the assigned default. Given the differences in capillary columns and separation technology (i.e. pressure control) on different gas chromatographs, the default retention time window should be chosen so that it is representative of the calculated windows for the reference compounds and not too broad. As a general rule, the default retention time window should not be greater than 1.0% of the absolute retention time of Tetracontane (n-C₄₀) under the conditions of the analysis.
- 10.1.2.4 EPH retention time windows for all fractions except the C8 through C10 are defined as beginning 0.1 minutes after the retention time of the beginning marker compound and ending 0.1 minutes after the retention time of the ending marker compound. Since the first fraction for both the aliphatics and aromatics includes the beginning marker compounds, the retention time windows for them are defined as beginning 0.1 minutes before the retention time of the beginning marker compound and ends 0.1 minutes after the ending marker compound. See Table 5.
- 10.1.2.5 Save the RT window calculations in the RT window folder for the instrument.

Table 5. Retention Time Windows for EPH Marker Compounds

Range/ HydroCarbon Standard	Beginning Marker Compound	Ending Marker Compound
C ₈ -C ₁₀ Aliphatic Hydrocarbons	Just before n-Octane	Just after n-Decane
>C ₁₀ -C ₁₂ Aliphatic Hydrocarbons	Just after n-Decane	Just after n-Dodecane
>C ₁₂ -C ₁₆ Aliphatic Hydrocarbons	Just after n-Dodecane	Just after n-Hexadecane
>C ₁₆ -C ₂₁ Aliphatic Hydrocarbons	Just after n-Hexadecane	Just after n-Heneicosane
>C ₂₁ -C ₃₄ Aliphatic Hydrocarbons	Just after n-Heneicosane	Just after n-Tetratriacontane
C ₈ -C ₁₀ Aromatic Hydrocarbons	Just before Toluene	Just after 1,2,3-Trimethylbenzene
C ₁₀ -C ₁₂ Aromatic Hydrocarbons	Just after 1,2,3-Trimethylbenzene	Just after Naphthalene
C ₁₂ -C ₁₆ Aromatic Hydrocarbons	Just after Naphthalene	Just after Acenaphthene
C ₁₆ -C ₂₁ Aromatic Hydrocarbons	Just after Acenaphthene	Just after Pyrene
C ₂₁ -C ₃₄ Aromatic Hydrocarbons	Just after Pyrene	Just after Benzo(g,h,i)perylene

10.1.3 Initial Calibration

Calibrate the GC system using the external standard procedure described below.

10.1.3.1 Working Calibration Standards

10.1.3.1.1 For each fraction, one of the calibration standards must be prepared at a concentration near, but above, the method detection limit and is used to determine the reporting PQL (5 µg/mL of each individual component). The other concentrations must correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

10.1.3.1.2 Prepare a high level calibration standard at 400ppm with an 8:2 ratio of the aliphatic standard and 1-chlorooctadecane. Add 8mL of aliphatic standard to a

10mL volumetric and 2mL of 1-chlorooctadecane standard. All other calibration levels are prepared, at the time of calibration in 1mL final volumes, by diluting this standard to achieve desired concentrations.

Level	Amount of standard used for dilution.	Final concentration of C8-C10 Aliphatics in 1mL	Final concentration of >C10-C12 Aliphatics in 1mL	Final concentration of >C12-C16 Aliphatics in 1mL	Final concentration of >C16-C21 Aliphatics in 1mL	Final concentration of >C21 to C34 Aliphatics 1mL
Level I 1ppm	100uL of 10ppm	3	2	4	7	13
Level II 10ppm	100uL of 100ppm	30	20	40	70	130
Level III 25ppm	500uL of 50ppm	75	50	100	175	325
Level IV 50ppm	125uL of 400ppm	150	100	200	350	650
Level V 100ppm	250uL of 400ppm	300	200	400	700	1300
Level VI 200ppm	500uL of 400ppm	600	400	800	1400	2600
Level VII 400ppm		1200	800	1600	2800	5200

10.1.3.1.3 For the aromatic calibration prepare a high level calibration standard at 400ppm. In a 10mL volumetric flask, using a gas tight syringe, add 2mL of the aromatic stock standard(accustandard Cat#z-014G), 2mL of the o-terphenyl standard(accustandard cat# DRH-006-SS-PAK), 2mL of the toluene standard(accustandard cat# M-502-46-10X) and 1mL of the p-terphenyl surrogate standard(ultra cat#ATS-160). From this the other 6 calibration levels will be prepared at a final volume of 1mL at the time of calibration.

Level	Amount of standard to be diluted	Final concentration of C8-C10 Aromatics in 1mLs (ug/mL)	Final concentration of >C10-C12 Aromatics in 1mLs (ug/mL)	Final concentration of >C12-C16 Aromatics in 1mLs (ug/mL)	Final concentration of >C16-C21 Aromatics in 1mLs (ug/mL)	Final concentration of >C21 to C34 Aromatics in 1mLs (ug/mL)
Level I 1ppm	100uL of 10ppm	1	1	2	5	8
Level II 10ppm	100uL of 100ppm	10	10	20	50	80
Level III 25ppm	500uL of 50ppm	25	25	50	125	200
Level IV 50ppm	125uL of 400ppm	50	50	100	250	400
Level V 100ppm	250uL of 400ppm	100	100	200	500	800
Level VI 200ppm	500uL of 400ppm	200	200	400	1000	1600
Level VII 400ppm		400	400	800	2000	3200

10.1.3.2 A collective calibration curve or factor must be established for each hydroCarbon range of interest within each fraction. Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (i.e. 1 to 2 μ L injections). Record the identification of the standards on the raw data.

For both the aliphatic and aromatic sets of standards, tabulate the summation of all peak areas in the calibration standard that fall within that range against the total mass represented by those peaks. Note that the surrogate compounds will each fall within a Carbon range. Surrogate areas should normally be subtracted from their respective Carbon ranges in both calibration standards and samples.

Only the highest or lowest calibration points may be rejected because of non-linearity. Rejection of such a point may affect the reporting limit or the linear range. Points within the curve may not be rejected [this represents a departure from the previous revision of this SOP that permitted a rejection for a valid analytical reason, because frequently no reasons were provided or the reasons were found to be invalid (e.g., when the deleted level was included, the curve would pass for the majority of the compounds) and the required documentation was lacking in completeness]. When rejecting an extreme end level, remember that there must be a minimum of five standards remaining for a linear model. Although a forced baseline projection is required for samples and blanks, the analyst is allowed to integrate only the areas of the specific calibration compounds to generate the calibration factors.

- 10.1.3.3 The ratio of the response to the amount injected, defined as the calibration factor (CF), or range CF, may be calculated for hydroCarbon ranges at each standard concentration using Equation 1. If the percent relative standard deviation (%RSD) of the calibration factor is equal to or less than 20% over the working range for the Carbon ranges of interest, as determined using Equation 2, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve. In the event that the 20% is exceeded, then linear regression or quadratic calibration is used. In either case, the correlation coefficient (r) must be ≥ 0.995 . Also, quadratic modeling requires a minimum of six calibration points.

Equation 1: Range Calibration Factor

$$\text{Range CF} = \frac{\text{Total Area of Peaks within the Range}}{\text{Total Mass Injected (ng) within the Range}}$$

Equation 2: Percent Relative Standard Deviation

$$\%RSD = \frac{\text{Standard Deviation of CFs}}{\text{Mean of the CFs}} \times 100$$

10.1.4 Always record the standard IDs on the raw data.

10.1.5 2nd Source Calibration Verification

10.1.6.1 Upon completion of the initial calibration, verify the calibration by analyzing a second source calibration standard. The source of this standard should be different than the source from which the calibration standards were obtained. In situations where a single vendor is used to provide the primary and secondary standards, the analyst must obtain written warranties that the two references were not prepared from the same reference material.

10.1.6.2 All hydroCarbon ranges should be within 20% of the expected value. In the event that this *criterion* is exceeded, corrective action must be taken and the instrument recalibrated.

10.1.6.3 Record the identification of the second source on the raw data.

10.1.6 Continuing Calibration Verification

10.1.6.1 The calibration curve (concentration or CF) must be verified prior to analysis of samples, after every ten (10) samples, and at the conclusion of analysis.

10.1.6.2 Several clients require that instrument contamination be monitored at the same frequency as CCV standards throughout the sequence.

10.1.6.3 Continuing verification may be completed using primary source calibration mixtures alternating between low and high concentrations every 10 samples throughout the analytical sequence and calculating the concentrations of all Carbon ranges against the calibration factor or linear regression curve.

10.1.6.4 The percent difference for the measured concentrations or CFs in the initial verification must be $\leq 20\%$ (see Equation 3) for hydroCarbon ranges. If acceptance criteria are not achieved, the verification process must be repeated using fresh calibration solution. If the suLCSequent calibration verification injection fails, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive calibration

verifications (using fresh calibration solutions, at low and high concentrations) or, alternatively, a new initial calibration must be established according to Section 9.1.3. If one of these calibration verification injections fails, a new initial calibration curve must be processed.

Equation 3: Percent Difference (%D)

$$\% D = \left| \frac{R_i - R_v}{R_i} \right| \times 100$$

where:

R_i = Standard concentration

R_v = Calculated concentration from verification check.

10.1.6.5 Record the identification of the standards and concentrations on the raw data.

10.1.6.6 Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present. Re-analysis is required for all other situations. If for some reason (i.e., lack of sample) re-analysis can't take place, a NCM needs to be initiated and the sample and QC results associated with the failing CCV need to be qualified in the final report.

10.1.6.7 Either a method or instrument blank spiked with surrogate must be analyzed immediately after each CCV standard. A batch number must be assigned to each method blank on the raw data, while instrument blanks may be designated as IBL- and continuing calibration blanks may be designated as CCB-. It is expressly prohibited to process consecutive blanks for the purpose of evaluating or reporting any of the blanks other than the initial blank (i.e., picking and choosing QC). If multiple blanks are analyzed consecutively, such as a CCB followed by a MB, the first Blank shall always be evaluated and corrective action taken, if necessary.

10.1.6.8 Record the identification of the standards and concentrations on the raw data.

10.1.7 Sample Analysis

- 10.1.7.1 Each analytical sequence must have a laboratory information management system (LIMS) sequence number.
- 10.1.7.2 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration, or verification of calibration, followed by samples interspersed with blanks and QC samples. The analytical sequence must end with an acceptable verification calibration standard. If the ending calibration standard is not acceptable, all samples analyzed after the last acceptable calibration verification must be reanalyzed. If, for whatever reason, reanalysis is not possible, then the data for those samples must be qualified as an "estimate."
- 10.1.7.3 Open an analytical sequence by injecting a solvent (methylene chloride) blank, followed by the Calibration Verification Standard. The solvent blank should contain no analyte(s) of interest at concentrations greater than the method reporting limit. If the concentration of a target analyte in the blank is greater than the method reporting limit, instrument maintenance must be performed. Try baking out the column and then analyze another solvent blank. If this fails, consult your supervisor.
- 10.1.7.4 Under the same instrument operating conditions employed for instrument calibration, verify the calibration by analyzing a continuing calibration standard. If the result for the continuing calibration standard falls within acceptance limits, proceed with sample analysis.
- 10.1.7.5 The hydroCarbon retention time windows for this method will be evaluated using the RTC run at the beginning of each sequence. If the retention times are off, ID the individual peaks and Chrom will update the ranges automatically.*
- 10.1.7.6 Transfer a small portion of the sample extract to a labeled auto-sampler vial. Analyze sample and QC extracts using the same instrument operating conditions employed for initial calibration and calibration verification. Aliphatic and aromatic extracts are introduced into the gas chromatograph by splitless injection. Inject 1 to 2 μL of the sample extract. Record the volume injected and the resulting peak size in area units. Client samples must be analyzed on the same instrument as the batch QC samples. Since instruments with two injectors, columns and detectors are effectively two separate instruments, this requirement is injector/column/detector specific in Semivolatiles Fuels. The exception to this is dilution re-analyses.

10.1.7.7 Validation of GC system qualitative performance is accomplished by the analysis standards within the analysis sequence. If any of the standards drifts outside the retention time window initially established for it on that day, the system is out of control. In such cases, the cause of the problem must be determined and corrected before continuing with analysis of samples.

10.1.7.8 Peak area integration for the aliphatic and aromatic Carbon ranges must be from the baseline (i.e. must include the unresolved complex mixture "hump" areas).

Note - The areas for the surrogates must be subtracted from the areas of the ranges in which they elute.

10.1.7.9 Because the quantitative technique involved in this method compares summed areas to discrete single peaks, the linear range of the calibration is defined in terms of peak height. The linear range of the calibration must be considered to have been exceeded if the chromatogram yields a response within any Carbon range window which is greater than the highest peak height found within that Carbon range during the initial calibration. Dilute and reanalyze sample extracts which exceed the linear range of the calibration. Record the lot number of the dilution solvent on the injection log. All detections which exceed the calibration range must be "E"-flagged on the raw data and in the LIMS (if uploaded).

10.1.7.9.1 Transfer a known amount of the original extract to a clean vial and add the appropriate amount of methylene chloride to achieve the desired dilution. Record the lot number of the methylene chloride on the injection log.

10.1.7.9.2 Analyze the diluted sample extract using the same instrument operating conditions employed for initial calibration and calibration verification. The response for the diluted extract should be kept in the upper range of the calibration curve. If the response falls in the lower range, a lesser dilution of the sample extract must be prepared and analyzed.

10.1.7.10 If batch QC samples are re-analyzed to confirm recovery failure or method blank contamination, and the improvement in results would cause the re-analysis to be reported, then the associated client samples must also be re-analyzed. The only exception to this

protocol would be an obvious analysis problem during the initial analysis (e.g. bent autosampler needle, etc.).

- 10.1.7.11 Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for instrument contamination and prevent carryover. If the blank shows contamination above the method reporting limit (one-half the MRL for DOD projects), the column must be baked out and subsequent blanks analyzed until the system is shown to be clean (less than the method reporting limit).
- 10.1.7.12 Analyze a continuing calibration standard every ten samples and at the conclusion of the analysis. Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present.
- 10.1.7.13 *Following each CCV, analyze a method blank or an instrument blank spiked with surrogate (see section 10.1.9.7).*

10.2 Quantitation and Data Reduction

10.2.1 Aliphatic Fraction

- 10.2.1.1 Separately determine the total area count for all peaks eluting 0.1 minutes before the RT for C8 to 0.1 minutes after the RT for C10 (C8 through C10 Aliphatics); for all peaks eluting 0.1 minutes after C10 through 0.1 minutes after C12 (>C10 through C12 Aliphatics); for all peaks eluting 0.1 minutes after C12 through 0.1 minutes after C16 (>C12 through C₁₆ Aliphatics); for all peaks eluting 0.1 minutes after C16 through 0.1 minutes after C21 (>C₁₆ through C₂₁ Aliphatics); and for all peaks eluting 0.1 minutes after C21 through 0.1 minutes after C34 (>C21 through C34 Aliphatics).
- 10.2.1.2 Determine the peak area count for the surrogate. Subtract these values from the collective area counts values within the appropriate hydroCarbon range(s).
- 10.2.1.3 Calculate the concentrations for the surrogate and the Aliphatic HydroCarbon ranges using the CFs determined from the calibration curves.

10.2.2 Aromatic Fraction

- 10.2.2.1 Separately determine the total area count for all peaks eluting 0.1 minutes before the RT for toluene to 0.1 minutes after the RT for 1,2,3-trimethylbenzene; for all peaks eluting 0.1 minutes after 1,2,3-trimethylbenzene through 0.1 minutes after naphthalene; for all peaks eluting 0.1 minutes after naphthalene through 0.1 minutes after acenaphthene; for all peaks eluting 0.1 minutes after acenaphthene through 0.1 minutes after pyrene; and for all peaks eluting 0.1 minutes after pyrene through 0.1 minutes after benzo(g,h,i)perylene.
- 10.2.2.2 Determine the peak area count for the surrogate. Subtract these values from the collective area counts values within the appropriate hydroCarbon range(s).
- 10.2.2.3 Calculate the concentrations for the surrogate and the Aromatic HydroCarbon ranges using the CFs determined from the calibration curves.

10.3 Manual Integrations

10.3.1 Manual integrations should be employed only in situations where the data system has:

10.3.1.1 incorrectly identified a signal, or

10.3.1.2 incorrectly quantitated a signal thereby producing an obvious bias

10.3.2 *Refer to corporate SOP CA-Q-S-002 for specifics on manual integration.* For any manual integrated peaks in blanks, blank spikes, matrix spikes, duplicate, initial and/or continuing calibration standards, the analyst is required to print the before and after chromatograms from the same scale where the manual integrations were performed. These chromatograms must be dated and initial by the analyst and retained in the calibration or daily sequence folder.

10.4 Reporting Format

10.4.1 Report the calculated concentrations of C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aliphatic HydroCarbon ranges and C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aromatic HydroCarbon ranges.

When reporting results on samples that were scheduled for both VPH and EPH, examine the results for the ranges that overlap both methods (C8-C10 Aliphatics, C8-C10 Aromatics, C10-C12 Aliphatics and C10-C12 Aromatics). Reporting only one result for each of these ranges, make sure that the highest result is set to "Reportable", while the lowest is turned Off. In the event that both are Nondetect, set the range with the lowest reporting limit to "Reportable" and turn Off the range with the highest reporting limit.

10.5 Data Review

10.5.1 Upon completion of the analytical run, the primary analyst must review all data for compliance with criteria documented in Section 9.0 and according to the procedures described in *SOP TA-QA-0635, Data Review.*

11.0 Calculations / Data Reduction

The concentration of each hydroCarbon range in a sample may be determined by calculating the amount of hydroCarbon range compounds injected, from the peak area response, using the calibration curve or the calibration factor determined in Section 10.1.3.

11.1 Aqueous samples:

Calculate the concentration of each hydroCarbon range in aqueous samples using Equation 6.

Equation 6: Aqueous Sample Calculation

$$\text{Range Concentration (ug / L)} = \frac{(Ax)(Vx)(D)}{(Vi)(Vs)(CF)}$$

where:

Ax = Area sum for a hydroCarbon range in the sample.

Vx = Volume of total extract, μL .

D = Dilution factor (if no dilution was made, $D = 1$).

Vi = Volume of extract injected, μL .

Vs = Volume of sample extracted, mL .

CF = Calibration Factor for hydroCarbon range from initial calibration, Area/ng .

11.2 Nonaqueous samples:

Calculate the concentration of each hydroCarbon range in soil/sediment samples using Equation 7.

Equation 7: Nonaqueous Sample Calculation

$$\text{Range Concentration (ug / Kg)} = \frac{(Ax)(Vx)(D)}{(Vi)(Sx)(CF)(DW)}$$

where:

Ax = Area sum for a hydroCarbon range in the sample.

Vx = Volume of total extract, μL .

D = Dilution factor (if no dilution was made, $D = 1$).

Vi = Volume of extract injected, μL .

Sx = Mass of sample extracted, grams

CF = Calibration Factor for hydroCarbon range from initial calibration, Area/ng .

DW = Fractional dry weight of the sample (i.e. 81.2 % solids = 0.812 fractional dry weight).

11.3 Standard Deviation

$$SD = \left(\frac{\sum_i^n (x_i - m)^2}{(n-1)} \right)^{1/2}$$

where: x = individual value within set
 m = mean of set
 n = population of set

11.4 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD * 100}{(\text{Mean RF})}$$

11.5 Percent Recovery (%R)

$$\% R = \frac{(C_s - C) * 100}{(C_a)}$$

where: C_s = OLC Served spiked sample concentration
 C_a = Spike Level
 C = Source Sample concentration

11.6 Relative Percent Difference (RPD)

$$RPD = \frac{|X_1 - X_2| * 100}{(X_1 + X_2)/2}$$

where: X_1 = Concentration of sample analyte
 X_2 = Concentration of duplicate analyte

12.0 Method Performance

12.1 Per the method, Method Detection Limit (MDL) studies are optional. If a MDL study is conducted, follow the procedure described *in SOP TA-QA-0602*.

12.2 An IDC must be completed according to the procedures specified in SOP SL-QAG-018.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., processing one set of MDLs on all applicable instruments, examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention." Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in

an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SL-SPL-006.

14.1 Waste Streams Produced by the Method.

The following waste streams are produced when this method is carried out.

14.1.1 Autosampler vials containing Methylene Chloride. After use, the 2.0 ml autosampler vials are discarded into properly labeled 1-gallon glass jars with screw cap located near any of the five GC's (GC-1, 3, 7, 9, or 11). When the jar is full, the EH&S specialist is notified that the vials are ready for proper disposal.

14.1.2 Waste Methylene Chloride rinses. Methylene chloride rinse waste generated at the vialing station is collected in properly labeled 300 ml tall jars under the hood. When the jars become full, the analyst disposes of the contents into the 55-gallon satellite waste drum in the Extractions department.

14.1.3 Expired Standards. Expired standards in 10 ml/25 ml screw cap vials are discarded into the 300 ml rinse waste tall jars (14.1.2).

15.0 References

15.1 TestAmerica Seattle Quality Assurance Manual (QAM), Rev. 2.

15.2 TestAmerica Environmental Health and Safety Manual, Rev 1.

15.3 "Extractable Petroleum Hydrocarbons (EPH) Fractions", Analytical Methods for Petroleum Hydrocarbons, Manchester Environmental Laboratory, Dept. of Ecology, State of Washington, June 1997.

16.0 Attachments

None

17.0 Revision History

- Revision 8, dated 26 January 2010
 - General formatting and integration for TestAmerica Seattle and Tacoma combined operations.
 - Updated equipment list Section 6.1.2
 - Added data system/audit trail information Section 6.1.5
 - Added evaluation of retention time windows by Chrom Section 10.1.7.5
- Revision 7, dated 5 December 2008
 - General formatting and integration for integration of TestAmerica and STL operations

18.0 Discrepancies to the Method

18.1 *Internal standards are not used with this method.*

Record of Management Decision

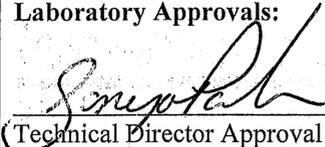
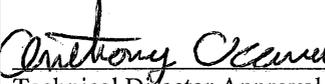
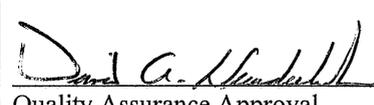
SOP: TA-___-___-R__ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00026

Effective Date: 11/2/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 _____ Technical Director Approval (Palmer) Date	 _____ Technical Director Approval (Ryan) Date
 _____ Technical Director Approval (Ocana) Date	_____ Technical Director Approval/H & S Approval Date
 _____ Quality Assurance Approval Date	_____ Laboratory Director Approval Date

1. **Description Of and Reason For Decision:**

As noted by a client, the columns identified in our SOP weren't the same as the columns identified on the package forms or raw data.

The chromatography column information typically provided in section 6 will be qualified with the following italicized text when appropriate:

(Column or primary column: Column ID and dimensions, as currently stated in the SOP) *or equivalent*
 (Secondary column: Column ID and dimensions, when applicable and as currently stated in the SOP) *or equivalent*
Note: Other columns may be used. These were the columns in place at the time the SOP was prepared.

**Column types must also be maintained by the analyst in TALS as previously noted.

2. **References:** AECOM/Port Heiden

3. **Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):**

AS 11-2-10 KBT
 A. Ocana, A. Mattison, K. Teffeau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,
 AO 11/1/10 11/1/10 Jil 11/10 OM 11/2/10 MAT 11/1/10 AT 11/1/10 EK 11/2/11
 M. Muir, S. Chambers, B. Hepner, T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 * 11/1/10 sv 11/1/10 BJA 11/1/10 10-28-10 SMC 10/28/10 J 11/1/10 MAT 11-1-10

SOPs for Chromatography Methods

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0354-R01	PCBs in Transformer Oil
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)

Record of Management Decision

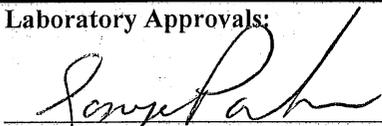
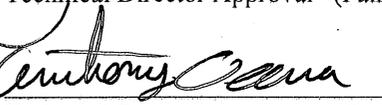
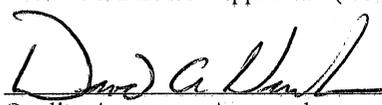
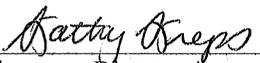
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00019

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
	10-15-10
Technical Director Approval (Palmer)	Date
	10/26/10
Technical Director Approval (Ocana)	Date
	10/28/10
Quality Assurance Approval	Date
	10-15-10
Technical Director Approval (Ryan)	Date
	10-15-10
Technical Director Approval/H & S Approval	Date
	10/28/10
Laboratory Director Approval	Date

1. Description Of and Reason For Decision:

As noted in an L-A-B DOD audit finding, instrument records did not consistently include the identification of the chromatographic column.

There are two venues to address this. In the SOP, under the section on Equipment and Supplies, a column type could be designated with the disclaimer "or equivalent". Also, as part of maintenance procedures, the specific make and model of chromatography columns installed need to be documented in the instrument's maintenance logbook.

2. References: QSM 4.1, 4.12.2.5.3c

3. Others Notified (date and initial below your name AFTER adding chromatography column information on the next page and verifying/updating the chromatography columns in your maintenance logs):

A. Ocana, A. Mattison, K. Tefteau, S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura,



M. Muir, S. Chambers, B. Hepner, T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen



SOPs for Chromatography Methods

(Add Column Types)

SOP No	SOP Title	Column Type 1	Column Type 2
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A	Zebrom MR-2	Zebrom MR-1
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)	Phenom. ZB-1	
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod	↓	
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)	↓	
TA-GS-0351-R17	PCBs by EPA 8082	Zebrom MR-2	Zebrom MR-1
TA-GS-0354-R01	PCBs in Transformer Oil	↓	↓
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology	Phenom. ZB-1	
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)	↓	
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)	Roshek RTX 5ms	Roshek RTX XL3
TA-GS-0380-R09	Pesticides and PCBs by EPA 608	Zebrom MR-2	Zebrom MR-1
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology	Roshek VRX	
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)	↓	
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)	Zebrom 5ms	
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)	ZB- 5MS	
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)	ZB-5ms	
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)	ZB624	
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)	Roshek VRX	ZB624
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)	Dionet AG-18/ AS 18	AG14/ AS14

TAC044 TAC038

Record of Management Decision

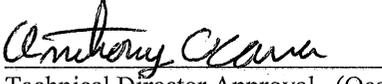
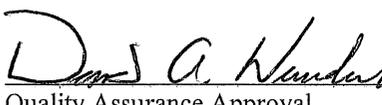
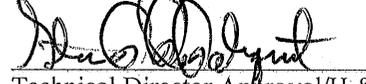
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00020

Effective Date: 10/25/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10-15-10</u> Date
 Technical Director Approval (Ocana)	<u>10/26/10</u> Date
 Quality Assurance Approval	<u>10/28/10</u> Date
 Technical Director Approval (Ryan)	<u>10-15-10</u> Date
 Technical Director Approval/H & S Approval	<u>10-15-10</u> Date
 Laboratory Director Approval	<u>10/28/10</u> Date

1. Description Of and Reason For Decision:

As noted in an L-A-B DOD audit finding, instrument and equipment records did not consistently include the operating conditions of the instruments and equipment.

There are two venues to address this. In the SOP, under the section on Procedures, operating conditions could be specified or generalized. Also, as part of maintenance procedures, the specific operating conditions need to be documented in the instrument's or equipment's maintenance logbook. This documentation could be in the form of a written summary or a printout.

2. References: QSM 4.1, 4.12.2.5.3c

3. Others Notified (date and initial below your name AFTER verifying/updating your maintenance logs):

A. Ocana, A. Mattison, K. Teffeau, S. Palmquist, F. Woo, P. Boardway
AM 10-20-10 KET
DO 10/20/10 *10/24/10* *9/20* *FB*

S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner,
Boledio CA 10/26/10 *MT 10/26/10* *AP 10/22/10* *EX 10/22/10* *11/2/10* *10/21/10* *10/21/10* *10/21/10*

K. Johnson, D. Brechler, MJ. Tangora
KJS 10/25/10 *10/25/10* *MT 10/25/10*

T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
10-15-10 *SAK 10/18/10* *10/10/10* *MAT 10-15-10*

SOPs for Methods with Operating Conditions

(Document Operating Conditions in Maintenance Manual if Different Than Those Described in SOP)

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0326-R08	Hydrocarbon Identification Method (NWTPH-HCID)
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391-R07	Analysis of Volatile Organic Compounds by Method 802.1B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MT-0200-R19	✓ Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	✓ Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	✓ Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-OP-0367-R00	Microwave Extraction Procedure
TA-OP-0388-R07	Gel Permeation Chromatography Extract Clean-up Procedure (3600C and 3640A)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0158-R10	Total Halogens EPA 9076)
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0186-R01	Total Halogens
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

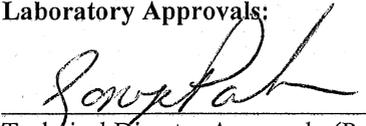
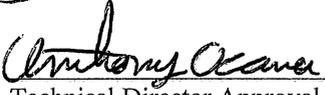
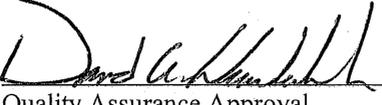
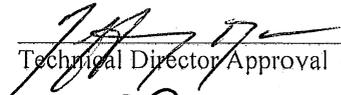
SOP: TA-___-___-R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00022

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10/26/10</u> Date
 Technical Director Approval (Ocana)	<u>10/26/10</u> Date
 Quality Assurance Approval	<u>10/28/10</u> Date
 Technical Director Approval (Ryan)	<u>10-27-10</u> Date
 Technical Director Approval/H & S Approval	<u>10/26/10</u> Date
 Laboratory Director Approval	<u>10/28/10</u> Date

1. Description Of and Reason For Decision:

As noted in an L-A-B DOD audit finding, the lab's instrumentation SOPs typically do not include sufficient detail pertaining to initial instrument calibration procedures as algorithms for all of the possible calibration models are not featured in those SOPs.

In addition to including the step-by step procedures employed to prepare and analyze the calibration standards, instrument SOPs also need to include a discussion of the possible calibration models as well as the applicable calibration algorithms or include a reference to corporate SOP CA-Q-S-005, Calibration Curves.

2. References: QSM 4.1, 5.5.2.2.1a

3. Others Notified (date and initial below your name):

Ao 10/26/10 ASM 10-26-10 KET 10/26/10 qd 10/26/10 AB 10-26-10
 A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway
 S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner, B/Kepner
 10-27-10 KES OM 10/27/10 MAT 10/27/10 22 10/27/10 22 10/27/10 22 10/27/10
 K. Johnson, D. Brechler, MJ, Tangora
 T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
 10-27-10 SPAN 10/27/10 10/27/10 MAT 10-29-10

Instrument SOPs with Inadequate Calibration Information

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-GV-0391 R07	Analysis of Volatile Organic Compounds by Method 8021B Utilizing Gas Chromatography with a Photoionization Detector (GC/PID)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MS-0346-R08	Organotin Compounds (Puget Sound Estuary Protocol)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0124-R09	Analysis of Total Phosphorus (EPA 365.1)
TA-WC-0156 R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0175-R09	Hexavalent Chromium (SM 3500 Cr-D)
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

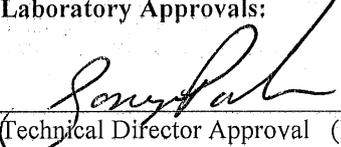
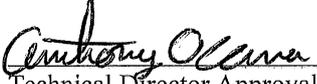
SOP: TA-____-____-R____ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00024

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 _____ Technical Director Approval (Palmer) Date	 _____ Technical Director Approval (Ryan) Date
 _____ Technical Director Approval (Ocana) Date	 _____ Technical Director Approval/H & S Approval Date
 _____ Quality Assurance Approval Date	 _____ Laboratory Director Approval Date

1. **Description Of and Reason For Decision:**

As noted in a L-A-B DOD audit finding, Gray Box 37 and Appendix F Tables state that for CCV failures, the laboratory shall reanalyze CCVs and all samples analyzed since the last successful CCV.

The applicable QC sections and tables in the attached SOPs need to reflect that CCV recoveries for DOD projects must fall within the DOD QSM-specified acceptance limits, regardless of observed bias or associated sample results and that for all CCV failures the lab shall reanalyze CCVs and all samples analyzed since the last successful CCV.

2. **References:** QSM 4.1, GB 37

3. **Others Notified (date and initial below your name):**

- A. Ocana, A. Mattison, K. Tefreau, S. Palmquist, F. Woo, P. Boardway
 - S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner.
 - K. Johnson, D. Brechler, MJ, Tangora
 - T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen
- Handwritten initials and dates:*
 KET 10-29-10, ASR 10-27-10, KET 10/29/10, CM 10/29/10, AM 10/29/10, PB 10-29-10, B. Hepner, KET 10-29-10, SAM 10/26/10, MAT 10-26-10, JCE 10/29/10, etc.

SOPs for DOD Certified Methods with CCV Requirements

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504.1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0122-R15	pH & Corrosivity (EPA 150.1, 9040B, 9045D and SM 4500-H+B)
TA-WC-0129-R11	Analysis of Conductivity (EPA 9050A and 120.1, Standard Method 2510B)
TA-WC-0138-R08	Corrosivity (EPA 9041A)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon In Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

Record of Management Decision

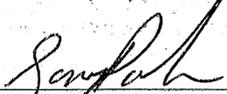
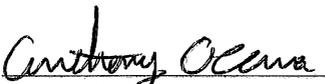
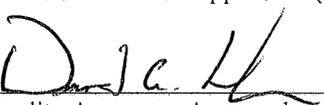
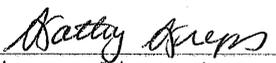
SOP: TA-___ - ___ -R___ (multiple SOPs - see attached list)

Title: (see attached list)

ROMD ID: 00025

Effective Date: 10/29/10

Expiration Date: When indicated SOPs are revised

Laboratory Approvals:	
 Technical Director Approval (Palmer)	<u>10/29/10</u> Date
 Technical Director Approval (Ryan)	<u>10-26-10</u> Date
 Technical Director Approval (Ocana)	<u>10/27/10</u> Date
 Technical Director Approval/H & S Approval	<u>10/28/10</u> Date
 Quality Assurance Approval	<u>10/29/10</u> Date
 Laboratory Director Approval	<u>10/29/10</u> Date

1. **Description Of and Reason For Decision:**

As noted in an L-A-B DOD audit finding, Section 5.2.9d and Appendix D require measures to assess precision on a batch basis.

The applicable QC sections and tables in the attached SOPs need to reflect that a LCSD will be prepared, analyzed and evaluated when adequate sample volumes for MS/MSD are not provided.

2. **References:** QSM 4.1, 5.2.9d

3. **Others Notified (date and initial below your name):**

AO 10/27/10 AM 10-27-10 KB 10/27/10 SB 10/28/10 RB 10-29-10
A. Ocana, A. Mattison, K. Tefteau, S. Palmquist, F. Woo, P. Boardway

SP 10/29/10 AP 10/29/10 SK 10/29/10
S. Palmer, C. McKean, B. Tadesse, A. Pham, E. Kimura, M. Muir, S. Chambers, B. Hepner.

KKJ 10-24-10 MJT 10/29/10 MAT 10-26-10
K. Johnson, D. Brechler, MJ, Tangcora

T. Ryan, S. Kreidermacher, J. Bogatay, M. Theisen

10-26-10 MAT 10-26-10

BH

SOPs for DOD Certified Methods

SOP No	SOP Title
TA-GS-0308-R19	Chlorinated Pesticides by EPA 8081A
TA-GS-0335-R14	Extractable Petroleum Fuel Hydrocarbons by EPA 8015B Mod
TA-GS-0339-R10	Semivolatile Petroleum Products Method for Soil and Water (NWTPH-Dx mod)
TA-GS-0351-R17	PCBs by EPA 8082
TA-GS-0354-R01	PCBs in Transformer Oil
TA-GS-0356-R08	Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions According to WDOE Methodology
TA-GS-0363-R13	Diesel and/or Residual Range Organics (AK102 & AK103)
TA-GS-0365-R08	Determination of EDB and DBCP in Aqueous Matrices (8011 and 504 1)
TA-GS-0380-R09	Pesticides and PCBs by EPA 608
TA-GV-0390-R07	Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions According to WDOE Methodology
TA-IP-0179-R11	Hexavalent Chromium Sample Preparation by Coprecipitation (EPA 7195 and 218 5)
TA-IP-0205-R16	Water Digestion Procedure for Total and Dissolved Metals Analysis (EPA 3005A, 3010A, 200 7, and 200 8)
TA-IP-0220-R06	Acid Digestion of Sediments, Sludges, and Soils (EPA 3050B)
TA-MS-0310-R11	Chlorinated Herbicides by GC/MS (EPA 8151A modified)
TA-MS-0313-R16	Semivolatile Organic Compound (Base/Neutrals and Acids) Analysis by GC/MS (EPA 8270C and 625)
TA-MT-0200-R19	Trace Metals Analysis by ICP (EPA 6010B and 200.7)
TA-MT-0202-R18	Mercury Analysis by CVAA (EPA 245.1, 7470A and 7471A)
TA-MT-0217-R21	Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)(EPA 6020 and 200.8)
TA-MV-0312-R19	Determination of Volatile Organic Compounds by GC/MS (EPA 8260B and 624)
TA-MV-0376-R08	Gasoline Range Organics/MBTEX Analysis (EPA 8015B/8260B, AK101 and NWTPH-Gx)
TA-OP-0301-R12	Separatory Funnel Extraction
TA-OP-0302-R11	Sonication Extraction Procedure
TA-OP-0314-R01	Waste Dilution by EPA 3580A
TA-OP-0323-R15	Continuous Liquid-Liquid Extraction
TA-OP-0334-R11	High Temperature Sonication Extraction (Method 3550B Modified)
TA-OP-0367-R00	Microwave Extraction Procedure
TA-QA-0620-R02	Quality Control Program
TA-WC-0101-R10	Alkalinity by Titration (EPA 310.1 and SM 2320B)
TA-WC-0102-R14	5-Day Biochemical Oxygen Demand
TA-WC-0103-R11	High and Low Level Chemical Oxygen Demand (EPA 410.1, 410.2 and SM 5220C)
TA-WC-0106-R10	Cyanide, Automated Colorimetric (EPA 335.4, SW 9010B/9013/9012A and SM 4500 CN-E, G, and I)
TA-WC-0109-R11	Hardness Analysis (EPA 1302 and SM 2340 C)
TA-WC-0111-R11	Ammonia (Methods EPA 350.1 and 350.2, SM 4500-NH3-B, and SM 4500-NH3-H)
TA-WC-0121-R10	n-Hexane Extractable Material (HEM) and Silica Gel Treated n-Hexane Extractable material (SGT-HEM) (EPA 1664)
TA-WC-0122-R15	pH & Corrosivity (EPA 150.1, 9040B, 9045D and SM 4500-H+B)
TA-WC-0125-R09	Determination of Solids in Waters and Wastes (SM 2540B, SM 2540C, SM 2540D, and SM 2540E)
TA-WC-0129-R11	Analysis of Conductivity (EPA 9050A and 120.1, Standard Method 2510B)
TA-WC-0138-R08	Corrosivity (EPA 9041A)
TA-WC-0154-R13	Static Flash Ignitability (EPA 1020A)
TA-WC-0156-R05	Determination of Total Organic Carbon in Liquid Matrices
TA-WC-0157-R12	Total Organic Carbon in Solids
TA-WC-0161-R17	Anions by Ion Chromatography (EPA 300.0 and 9056A)
TA-WC-0186-R01	Total Halogens
TA-WC-0187-R07	Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)

**Title: Determination of Volatile Petroleum Hydrocarbons (VPH)
Fractions According to WDOE Methodology
[Method No. WA-VPH]**

Approvals:

SIGNATURES ON FILE

Mike Theisen
Chemist

Date

Stan Palmquist
Health & Safety Manager / Coordinator

Date

Dave Wunderlich
Quality Assurance Manager

Date

Kathy Kreps
Laboratory Director

Date

This SOP was previously identified as SOP No. SL-FLS-008.

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method is designed to measure the collective concentrations of different carbon ranges of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. The carbon ranges used throughout this document are given in equivalent carbon (EC) numbers. The equivalent carbon number of a given compound is determined by normalizing its boiling point (or retention time on a boiling point gas chromatographic column) against the boiling points of normal alkanes.

Volatile aliphatic hydrocarbons are collectively quantitated within four ranges:

C_5-C_6
 $>C_6-C_8$
 $>C_8-C_{10}$
 $>C_{10}-C_{12}$

Volatile aromatic hydrocarbons are collectively quantitated within three ranges:

$>C_8-C_{10}$
 $>C_{10}-C_{12}$
 $>C_{12}-C_{13}$

1.1.2 This method is also designed to measure the individual concentrations of benzene, toluene, ethylbenzene, xylenes, methyl tert butyl ether (MTBE), and *naphthalene* in water and soil.

1.1.3 Petroleum products suitable for evaluation by this method include, but are not limited to, gasoline, mineral spirits, and certain petroleum naphthas. This method, in and of itself, is not suitable for the evaluation of samples contaminated with kerosene, jet fuel, heating oils, lubricating oils, or other petroleum products which contain a significant percentage of hydrocarbons larger than C_{10} . When samples are known or suspected to contain petroleum hydrocarbons of these or similar types, the Extractable Petroleum Hydrocarbon (EPH) method should also be employed to fully evaluate the hydrocarbons present.

1.1.4 The practical quantitation limits (PQLs) for this method are:

Water - 50.0 $\mu\text{g/L}$ for each aliphatic or aromatic carbon range and 5.0 $\mu\text{g/L}$ for individual target compounds based upon a 5.0 mL sample size. The laboratory's reporting limits are 50 $\mu\text{g/L}$ for each aliphatic or aromatic carbon range and 1.0 $\mu\text{g/L}$ for individual target compounds with the exception of *naphthalene* with a reporting limit of 2.5 $\mu\text{g/L}$.

Soils - 5.0 mg/Kg for each aliphatic or aromatic carbon range and 0.5 mg/Kg for individual target compounds based upon a 10.0 g to 10 mL methanol extraction with 100 μL of the extract purged and assuming a minimum of 50% solids for soil/sediments. The laboratory's reporting limits are 5.0 mg/Kg for each aliphatic or aromatic carbon range, 0.02 mg/kg for Benzene, 0.07mg/kg for MTBE and 0.5 mg/Kg for Ethylbenzene, naphthalene, Toluene, o-Xylene and m,p-Xylenes.

The analyst is allowed to extract a larger sample amount, provided that the solvent volume is increased proportionally to prevent the potential reduction of extraction

efficiency. Due to its impact on representativeness, smaller sample amounts are not allowed. Evaporative concentration of the methanol extract is also not allowed.

- 1.1.5** This method is based on a purge-and-trap, gas chromatography (GC) procedure. This method should be used by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.
- 1.1.6** Like all GC procedures, this method is subject to a "false positive" bias in the reporting of targeted analytes, in that non-petroleum compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated with the respective carbon ranges. Confirmatory analysis by a GC/MS, EPA Method 8260B, or other suitable procedures is recommended in cases where significant concentrations of non-hydrocarbon compounds are known or suspected. If the results of these analyses lead to identification and quantitation of non-petroleum compounds, the analyst may subtract those values from the affected carbon ranges as long as the identities and quantities of subtracted compounds are provided in the analytical report.
- 1.1.7** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 13.3.1 in the Quality Assurance Manual.

2.0 Summary of Method

- 2.1** Samples are analyzed using purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved using a photo-ionization detector (PID) and a flame ionization detector (FID) in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons.

The FID chromatogram is integrated in sections based upon the beginning and ending points of the equivalent carbon ranges to determine the concentrations of the total hydrocarbons (aliphatic and aromatic) within each carbon range.

The PID chromatogram is integrated in sections based upon the beginning and ending points of the equivalent carbon ranges to determine the concentrations of only the aromatic hydrocarbons within each carbon range.

The concentrations of the aromatic hydrocarbons (PID) are subtracted from the corresponding total hydrocarbons (FID) to calculate the aliphatic hydrocarbons within each carbon range.

- 2.2** The beginning and ending points of the equivalent carbon ranges are determined by analysis of retention time reference standards containing normal alkanes.
- 2.3** Average calibration or response factors, determined using both the aliphatic and aromatic compounds from a combined aliphatic/aromatic hydrocarbon standard mixture, are used to calculate the collective concentrations of the total hydrocarbons within each equivalent carbon range.

Average calibration or response factors, determined using only the aromatic hydrocarbons from the combined aliphatic/aromatic hydrocarbon standard mixture, are used to calculate the collective concentrations of the aromatic hydrocarbons within each equivalent carbon range.

2.4 This method is suitable for the analysis of waters, soils, and sediments.

3.0 Definitions

3.1 Volatile Petroleum Hydrocarbons (VPH): All hydrocarbon compounds eluting just prior to n-pentane (n-C₅) through 1-methylnaphthalene. VPH is comprised of C₅ through C₆, >C₆ through C₈, >C₈ through C₁₀, and >C₁₀ through C₁₂ Aliphatic Hydrocarbons, >C₈ through C₁₀, >C₁₀ through C₁₂, and >C₁₂ through C₁₃ Aromatic Hydrocarbons, and benzene and toluene. VPH concentration data are reported as the aggregate concentrations of the aliphatic and aromatic hydrocarbon ranges and as selected targeted analytes.

3.2 Extractable Petroleum Hydrocarbons (EPH): All hydrocarbon compounds eluting from toluene through benzo(g,h,i)perylene. EPH is comprised of C₈ through C₁₀, >C₁₀ through C₁₂, >C₁₂ through C₁₆, >C₁₆ through C₂₁ and >C₂₁ through C₃₄ Aliphatic Hydrocarbons and C₈ through C₁₀, >C₁₀ through C₁₂, >C₁₂ through C₁₆, >C₁₆ through C₂₁ and >C₂₁ through C₃₄ Aromatic Hydrocarbons. EPH concentration data are reported as the aggregate concentration of the aliphatic and aromatic hydrocarbon ranges.

3.3 Equivalent Total Petroleum Hydrocarbons (E-TPH): The summation of the EPH value and the VPH value with correction for overlapping carbon ranges:

For samples contaminated only with petroleum products heavier than C₁₀, the E-TPH value is equivalent to the EPH value.

For samples contaminated only with petroleum products lighter than C₁₂, the E-TPH value is equivalent to the VPH value.

For samples contaminated with petroleum hydrocarbons containing significant concentrations of hydrocarbons in both the EPH and VPH ranges (e.g. contaminated with both gasoline and diesel fuel) the E-TPH value is equal to the sum of the EPH and VPH values minus the lower of the two values in the overlapping (C₁₀ to C₁₂) aliphatic and aromatic range. In other words, the higher value is used from each fraction in the overlapping range when summing the EPH and VPH results.

3.4 C₅ through C₆ Aliphatic Hydrocarbons: All aliphatic hydrocarbon compounds which elute on the FID chromatogram from (and including) n-pentane through n-hexane.

3.5 C₆ through C₈ Aliphatic Hydrocarbons: All aliphatic hydrocarbon compounds which elute on the FID chromatogram after n-hexane through n-octane.

3.6 C₈ through C₁₀ Aliphatic Hydrocarbons: All aliphatic hydrocarbon compounds which elute on the FID chromatogram after n-octane through n-decane.

3.7 C₁₀ through C₁₂ Aliphatic Hydrocarbons: All aliphatic hydrocarbon compounds which elute on the FID chromatogram after n-decane through n-dodecane.

3.8 C₈ through C₁₀ Aromatic Hydrocarbons: All hydrocarbon compounds which elute on the PID chromatogram after toluene through 1,2,3-trimethylbenzene. The range is established by identifying the C₈ and C₁₀ Aliphatic hydrocarbons on the PID signal and using them as the marker compounds for the C₈ through C₁₀ aromatic range.

3.9 C₁₀ through C₁₂ Aromatic Hydrocarbons: All hydrocarbon compounds which elute on the PID chromatogram after 1,2,3-trimethylbenzene through naphthalene. The range is established by identifying the C₁₀ and C₁₂ Aliphatic hydrocarbons on the PID signal and using them as the marker compounds for the C₁₀ through C₁₂ aromatic range.

3.10 C₁₂ through C₁₃ Aromatic Hydrocarbons: All hydrocarbon compounds which elute on the PID chromatogram after naphthalene through 1-methylnaphthalene. The range is

established by identifying the C₁₂ Aliphatic hydrocarbon on the PID signal and using it as the marker compound for the beginning of the C₁₂ through C₁₃ aromatic range. This is a hybrid range which is designed to acquire the methylnaphthalenes associated with petroleum products like gasoline and is only used when VPH is run without an accompanying EPH method request.

- 3.11** Targeted VPH Analytes: Benzene, Toluene, Ethylbenzene, m,p,o-Xylenes, *Naphthalene* and MTBE.
- 3.12** VPH Component Standard: A 15-component mixture of aliphatic and aromatic compounds, plus surrogate (Table 1). The compounds are used to: a) define the individual retention times and chromatographic response factors for each of the Targeted VPH Analytes, b) define and establish the windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and c) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges.

Table 1. VPH Component Standard

Compound	Equivalent Carbon Number
n-Pentane	5.0
MTBE	N/A
n-Hexane	6.0
Benzene	6.5
Toluene	7.6
n-Octane	8.0
Ethylbenzene	8.5
m- & p- Xylene	8.6
o-Xylene	8.8
4-Bromofluorobenzene (surrogate)	N/A
n-Decane	10.0
1,2,3-Trimethylbenzene	10.1
n-Dodecane	12.0
Naphthalene	11.7
1-Methylnaphthalene	13.0

- 3.13** Batch: A group of 20 or less samples extracted and processed together within the same shift using the same reagents. Each batch must contain the minimum QC of a method blank and a laboratory control sample. If sufficient sample is available, a matrix spike and matrix spike duplicate should be included in each analytical batch.
- 3.14** Field Duplicates: Two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures.

The analysis of field duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

- 3.15** VPH Calibration Standards: A series of standard solutions prepared from dilutions of a stock standard solution containing known concentrations of response reference compounds and surrogate compounds of interest.
- 3.16** Calibration Check Standard (Continuing Calibration Standard) (CCS): A calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock standard solution as the calibration standards and is generally one of the mid-range calibration standard dilutions.
- 3.17** Matrix Spiking Solution: A solution which is prepared independently from the calibration standards and which contains known concentrations of method analytes.
- 3.18** Laboratory Method Blank: Depending on the matrix of the samples, either reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is prepared and analyzed along with all associated samples in a single batch. It is exposed to all glassware, solvents, reagents and equipment. At least one laboratory method blank is analyzed with every batch of samples to determine if method analytes or other interferences are present in the laboratory environment, reagents or equipment.
- 3.19** Laboratory Fortified Blank/Blank Spike/Laboratory Control Sample (LFB/BS/LCS): Depending on the matrix of the samples, either reagent water or clean sand blank fortified with a matrix spiking solution. This control sample is prepared and analyzed along with all associated samples in a single batch. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required practical quantitation limits.
- 3.20** Laboratory Fortified Matrix/Matrix Spike (LFM/MS): An environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. This control sample is prepared and analyzed along with all associated samples in a batch. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the LFM/MS sample corrected for background concentrations.
- 3.21** Calibration Verification Standard (CVS): A VPH quality control standard (Certified, or equivalent) from a source other than that used to prepare the VPH Calibration Standards. This standard serves as a quality control check to verify the accuracy of calibration. (Second source standard.)

4.0 Interferences

- 4.1** Some sources of purge and trap grade methanol have occasionally been shown to contain contaminant levels of the target analytes for this method. A methanol blank should be analyzed before using any new lot of methanol for client samples.
- 4.2** Sample injection line carryover contamination can be prevented by daily rinsing of the sample injection lines with sequential washes of approximately 0.5 mL of methanol and reagent water.
- 4.3** Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water or solvent. The trap and other parts of the purge and trap system are also subject to contamination. Frequent trap bake out at 240° for 12 minutes may be required to prevent this contamination. When a suspected

high-level sample is analyzed, it should be followed by an analysis of reagent water (purge blank) to check for carryover contamination.

- 4.4 Reagent water may become contaminated with low-level volatile organic compounds due to background laboratory contamination. Reagent water is continuously purged with helium to minimize this interferent.
- 4.5 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. Trip blanks prepared from reagent water should be carried through sampling and subsequent storage and handling to serve as a check on such contamination.
- 4.6 Storage blanks are used to monitor contamination in refrigerators used to store samples for volatile analysis. These blanks are prepared, logged in and transferred to the appropriate refrigerators by sample control or QA personnel according to SOP TA-QA-0616.
- 4.7 A number of low-level contaminants may be encountered. These include Benzene, Toluene, Ethylbenzene and Xylenes. Contamination sources may include breakdown of trap material, basic laboratory contamination (glassware, lab air, etc.) or the reagent water. These are monitored on a daily basis through the use of calibration and method blanks.
- 4.8 Certain organic compounds not associated with releases of petroleum products, including chlorinated solvents, ketones, and ethers, will be quantitated as Volatile Petroleum Hydrocarbons. Some samples may require additional analytical procedures to be employed, e.g. GC/MS, to document the presence and quantity of such compounds.
- 4.9 The response selectivity of a photo-ionization detector (PID) is used in this method to differentiate aromatic hydrocarbons from aliphatic hydrocarbons. All compounds eluting on the PID chromatogram within the defined equivalent carbon ranges are identified by the method as aromatic hydrocarbons. This will lead to an overestimation of aromatic hydrocarbons within samples, as certain aliphatic compounds will elicit a response on the PID, particularly unsaturated compounds such as alkenes.

5.0 **Safety**

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 **Specific Safety Concerns or Requirements**

- 5.1.1 Eye protection that satisfies ANSI Z87.1 (per the Corporate Safety Manual), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex, nitrile and vinyl gloves may be worn while handling samples, standards, solvents, and reagents for this procedure. Cut resistant gloves must be worn when using sharp tools or when washing glassware. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

- 5.1.2** The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- 5.1.3** There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.
- 5.1.4** GC VOA instruments use an ultraviolet (UV) light source, which must be shielded from view. There should also be a warning label/sticker on each instrument that identifies it as a UV light source.
- 5.1.5** Spills of USDA regulated soil and water residue are to be handled as follows:
Spray generously with bleach, before attempting to clean the spill up to prevent the release of airborne contaminants. The area should be cleaned with bleach again after the spill has been removed. Place all clean up material, spilled sample and PPE into a sealable container for autoclaving.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Hewlett Packard 5890 Series II Gas Chromatograph or equivalent with OI model 4430 Photoionization Detector (PID) or equivalent and OI Flame Ionization Detector (FID) or equivalent
- Data System: Hewlett Packard ChemStation for Windows 95 (version G1701AA) or equivalent. Agilent's ChemStation, is used for data acquisition and storage on machine-readable media. *Since no processing is done by Chemstation and since there are no audit trail functions associated with data acquisition, the audit trail feature for Chemstation may be either enabled or disabled. The other component, Chrom, is used for data processing such as the measurement of peak area or peak height. By design, the audit trail feature for Chrom is always enabled.*
- Column: Restek RTX-VRX (75 m x 0.45 mm x 2.5 µm) or equivalent
- Supelco M Trap or equivalent

- Analytical balance - Capable of accurately weighing 0.0001 g (for weighing standards), calibrated per SOP TA-QA-0014.
- Top-loading balance - Capable of accurately weighing to 0.01 g (for weighing soil samples), calibrated per SOP TA-QA-0014.
- Ultrasonic bath

6.2 Supplies

- Gas tight syringes: 10 μ L, 25 μ L, 50 μ L, 100 μ L, 250 μ L, 500 μ L, 1 mL, 2.5 mL, 5 mL, 10 mL and 25 mL - accurate to \pm 3%
- 5 mL Luer Lock syringe - accurate to \pm 3%
- Bottle Top Dispenser, for methanol - accurate to \pm 3%
- Sample Containers:
 - Soil: 40 mL Scintillation vials.
 - Water: 40 mL VOA vials (single use) with PTFE/silicone septum caps
- 40 mL VOA vials (single use), 5 mL and 2 mL gas-tight vials
- Volumetric flasks: 10, 25, 50, 100, 250 and 1,000-mL with gas-tight stoppers, accurate to \pm 1%
- Disposable Pasteur pipettes
- Spatula: Stainless steel.
- pH paper (pHydrion)
- Vortex mixer
- Centrifuge

7.0 Reagents and Standards

7.1 All reagents and standards used in this procedure must conform to the requirements specified in SOP TA-QA-0619 Preparation, Storage, and Verification of Standards. All initial instrument calibrations must be verified with a standard obtained from a second manufacturer. If a standard from a second manufacturer is not available, verification may be achieved by using a different lot from the primary manufacturer. However, the supervisor must obtain written warranties that the two references were not prepared from the same reference material. *Reagents/standards and reagent/standard preparation must be documented in TALS using the reagent module as described in SOP TA-QA-0619.*

All standards prepared by the laboratory must be stored without headspace at -10°C to -25°C and protected from light. Laboratory prepared standards must be replaced within 6 months of preparation. Standards that are purchased pre-made from commercial suppliers may be kept for the life, and under conditions, specified by the manufacturer. Standards should be brought to room temperature prior to use. Volumetric flasks are cleaned by rinsing with methanol *or reagent water*. **DO NOT PLACE VOLUMETRIC GLASSWARE IN AN OVEN.**

7.2 Reagents

7.2.1 Deionized water: *continuously* purged with helium

7.2.2 Purge and Trap grade methanol (*MeOH*): Store in a flammable-safe cabinet away from other solvents. Methanol is usually dispensed using a bottle-top type dispenser

7.2.3 Ottawa and/or masonry sand: Baked, free of volatile petroleum hydrocarbons

7.3 Standards

7.3.1 *Document standards and standard preparation in TALS using the reagent module as*

described in SOP TA-QA-0619.

7.4 Calibration Standard Solutions

7.4.1 WA VPH Stock Standard: Contains all compounds listed in Table 1 (except the surrogate) at a concentration of 1000 µg/mL in methanol. (Restek Cat No 30451 or equivalent).

7.4.1.1 VPH Calibration Standard: Dilute 1.25mL of WA VPH Stock Standard (7.2.1) to 25mL of MeOH for a final concentration of 50 ppm (ng/mL) of each compound. This standard is used to prepare initial and verifications standards for instruments equipped with autosamplers. Working standards expire after six months (or on the expiration date of the parent, if sooner).

7.4.2 Second Source VPH Custom Stock Standard: Contains all compounds listed in Table 1 (except the surrogate) at a concentration of 1,000 µg/mL in methanol. The second source standard may contain extra compounds including n-Nonane and n-Heptane. (Ultra Scientific, Restek Cat No 559222 or equivalent).

7.4.3 Internal Standard Stock Solution: Contains 1,2,3-Trifluorobenzene at 50,000 ug/mL in methanol. (AccuStandard Cat No S-12830 or equivalent).

7.4.4 Surrogate Stock Standard: 4-Bromofluorobenzene at a concentration of 10,000 µg/mL. (Restek Cat No 30082 or equivalent)

7.5 Working Surrogate Spike Solutions for Samples

7.5.1 Combined Internal standard/surrogate solution for water and soil samples: 1.2mLs of Internal Standard Stock Solution (7.2.2) and 7.5mLs of Surrogate Stock Standard (7.2.3) diluted to a final volume of 500 mLs in methanol. Contains 1,2,3-TFB at a concentration of 120 µg/mL and 4-BFB at a concentration of 150ug/mL in methanol. Working standards expire after six months (or on the expiration date of the parent, if sooner.)

7.6 Working Matrix Spike Solution

7.6.1 Water and Soil Intermediate: 1250 µL of Second Source VPH Custom Stock Standard (7.2.2) and 1.25 mL Nonane 2nd source stock diluted to a final volume of 25 mLs with methanol. All target compounds present at a concentration of 50 µg/mL in methanol. Working standards expire after six months (or on the expiration date of the parent, if sooner).

7.7 70% alcohol (or bleach) for disinfecting.

7.8 *Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to sec. 13.1.7.*

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Aqueous samples should be collected in 40 mL VOA vials with Teflon lined septa and screw caps. Samples should be preserved with 1:1 HCl sufficient to lower sample pH to < 2. If the testing schedule includes aromatic compounds such as BTEX, the samples must be preserved to pH < 2. Sample vials should be filled to overflowing with no visible headspace or air bubbles. All samples should be stored refrigerated at a temperature of 0-6°C. Samples suspected or confirmed to contain high levels of contaminants (> 1200 ppb) must be stored in a separate designated refrigerator. Highly buffered samples may require more acid to obtain the desired reduction in pH.

- 8.2** Solid samples should be subsampled in the field using an EasyDraw syringe and then dispensed into a pre-weighed 40 mL VOA vial with 10 ml methanol preservative. A second vial for each sample should be prepared in the same manner and a separate jar should be used to collect sample for dry weight determination. All samples should be stored refrigerated at a temperature of 0-6°C. Samples suspected or confirmed to contain high levels of contaminants (> 1200 ppb) must be stored in a separate designated refrigerator.

Unpreserved bulk soil samples received from BP, Shell or BNSF must be extracted in methanol within 48 hours of collection.

- 8.3** Preservation of water samples should be confirmed and documented by the laboratory analyst immediately after opening the sealed VOA vial and obtaining the necessary aliquots for analysis.
- 8.4** Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	VOA Vial	50 mLs	HNO ₃ , pH < 2; Cool 0-6°C	14 Days (7 Days if not preserved)	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 0-6°C	14 Days	N/A

9.0 Quality Control

- 9.1 Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) ¹	1 in 20 or fewer samples	Statistical Limits ⁴
Matrix Spike (MS) ²	1 in 20 or fewer samples	Statistical Limits ⁴
MS Duplicate (MSD) ²	1 in 20 or fewer samples	Statistical Limits ⁴
Surrogates	every sample ³	Statistical Limits ⁴

¹ LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

² The sample selection for MS/MSD are randomly selected, unless specifically requested by a client...predetermined by the extraction lab.

³ Analytical and QC samples (MB, LCS, MS/MSD)

⁴ Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV)

9.2.2 Continuing Calibration Verification (CCV)

9.2.3 Calibration Acceptance Summary

Include a line for each appropriate parameter you used above.

Step	Standards	Type	Control Limit	Frequency
<i>Method #</i>				
<i>Initial Cal</i>	<i>Conc and # of stds</i>	<i>Type of Cal: Linear, ...</i>		<i>How often performed?</i>
<i>ICV</i>				
<i>CCV</i>				

9.3 *Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.*

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # TA-QA-0610. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Sample Preparation

10.2 Soil and Sediment Preparation

Soil samples should arrive at the lab preserved in methanol. After determining the final weight of each sample container, the field-preserved sample can be analyzed by the procedures that begin in section 10.4.3. For soil samples that weren't field-preserved in methanol, process according to the procedures described below.

Unpreserved bulk soil samples received from BP, Shell or BNSF must be extracted in methanol within 48 hours of collection.

Note: Prior to preparing USDA regulated soils, review the procedures described in Section 9.4 of SOP TA-QA-0531, Handling, Storing, Safeguarding, Transporting and Disposing of Foreign and Regulated Domestic Soil Samples and Associated Aqueous Residues

10.2.1 *Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in TA-QA-0014.*

10.2.2 Prescreen samples using historical data or a portable PID meter in order to estimate the existing contamination level and any necessary dilutions. Batch samples into groups of not more than 20 samples each.

10.2.3 Tare a labeled 40 mL scintillation vial on a top loading balance.

10.2.4 Open a sample container and quickly transfer at least 10 + 1 grams of sample to the vial. Reseal and determine the mass of the soil/sediment sample. Record the mass to 0.05 g in the VOA Soil Extraction Log and provide the other requested information.

(Autosamplers add 2uL of combined BFB and IS standards to the 5 mL aliquot to be purged. Also, all site specific QC derived from initial sample extraction.)

10.2.5 Quickly add 10 mL of methanol to each vial. Recap the vial.

10.2.6 Vortex until no pellets remain and then place on shaker for at least 5 min.

10.2.7 Allow sediment to settle or centrifuge the vials until a clear layer of methanol is apparent.

10.2.8 *For the extraction blank, LCS and LCSD, weigh out a 10 g aliquot of prepared Ottawa sand into a VOA vials. Quickly add 10 mL of methanol to the vial. Mix (10.6.2.2.5 and 10.6.2.2.6). The LCS and LCSD are spiked directly into the Ottawa sand just prior to the addition of methanol. The surrogate is added at the instrument level, by the auto sampler.*

10.2.8.1 Determine the percent solids on soil and sediment samples using a separate aliquot of sample as described in SOP TA-WC-0160. Upload the results into the laboratory information management system (LIMS).

10.3 Initial Calibration

10.3.1 The percent RSD for the initial calibration must be $\leq 20\%$. If the %RSD is $>20\%$, linear or nonlinear modeling may be attempted. The correlation coefficient (r) or coefficient of determination (r^2) must be ≥ 0.995 for all linear or nonlinear calibration curves.

When linear regression is used, the intercept value should be checked to ensure that it is not at a level that is equivalent to or higher than the lowest calibration standards being used.

10.3.2 A second source must be analyzed and have %D $<20\%$ for individual target compounds and %D $<30\%$ for hydrocarbon ranges.

10.3.3 If acceptance criteria are not achieved, corrective action must be taken and a new initial calibration completed.

10.3.4 Any samples associated with a failed initial calibration must be reanalyzed.

10.4 Continuing Calibration Verification

10.4.1 Calibration verification must be completed prior to sample analysis.

10.4.2 Calibration verification must be completed after every 10 samples.

10.4.3 Calibration verification must be completed at the end of the analytical sequence.

10.4.4 The RFs for the respective analyte(s) in the continuing verification must not vary more than 20% for individual target compounds or hydrocarbon ranges from those established by the initial calibration.

10.4.5 In the event of calibration verification failure, corrective action must be taken prior to sample analysis. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications (using fresh calibration solutions, at low and high concentrations) or, alternatively, a new initial calibration must be established according to Section 10.3. If one of these calibration verification injections fails, a new initial calibration curve must be processed.

10.4.5.1 Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present. Re-analysis is required for all other situations. If for some reason (i.e., lack of sample) re-analysis can't take place, a NCM needs to be initiated and the sample and QC results associated with the failing CCV need to be qualified in the final report.

10.4.5.2 If the sequence of samples preceding a failing closing CCV includes samples suspected of matrix interference and interference-free samples such as blanks and blank spikes, then the samples analyzed prior to the failing closing calibration verification need to be segregated for re-analysis. The interference-free samples should be analyzed within one set of bracketing CCVs, and the samples with suspected interference should be analyzed within another set of bracketing CCVs.

10.4.5.3 Sample results associated with a CCV failure need to be qualified at the analyte level as appropriate. Additional information related to the CCV failure or corrective actions taken should be summarized in a NCM.

10.5 Sample Analysis

Daily start-up tasks are summarized in Attachment-02. Analysis of standards and sample extracts is completed using a GC/PID/FID equipped with a purge and trap. The following recommended conditions apply to instrument configurations listed in Section 6.0.

10.6 Recommended purge and trap conditions are:

Table 3

Purge gas:	<i>Nitrogen</i>
Purge gas flow rate:	40 mL/minute
Purge time:	11.0 minutes
Purge temperature:	Ambient
Dry purge	4 minutes
Desorb time	1.5 minutes
Bake	11 minutes
Desorb and bake	250°C
Backflush inert gas flow:	15-20 mL/minute

10.7 Recommended Gas Chromatographic conditions are:

Column:	Restek RTX-VRX (75 m x 0.45 mm x 2.55 μ m)
Injection Port:	220°C
Injection volume:	100 μ l MeOH or 5 mLs water
Injector:	Low volume injector
Flow:	10 mL/min. @ 40°C
Liner:	L.V.I.
Transfer Line:	100°C
GC Program:	Initial temp 40°C Ramp to 65°C @ 15°C per minute for 2 minutes Hold 6 minutes Ramp 15°C per minute to 230°C for 11 minutes Hold 3.5 minutes
Total time:	22.5 minutes

Note: These conditions can vary by instrument. This is only a guideline.

10.8 Initial Calibration**10.8.1** Retention Time Windows

10.8.1.1 VPH retention time windows are defined as beginning 0.1 minutes after the retention time of the beginning marker compound and ending 0.1 minutes after the retention time of the ending marker compound. The exception to this is the C₅ through C₆ Aliphatic Hydrocarbon ranges, where its retention time window is defined as beginning 0.1 minutes prior to the beginning marker compound and ending 0.1 minutes after the ending marker compound. The retention time windows are defined and summarized in Table 4.

10.8.1.2 *The hydrocarbon retention time windows for this method will be evaluated using the opening CCV.*

10.8.1.2.1 The retention time of the CCV will updated using the update retention times function of Chrom.

Table 4. VPH Hydrocarbon Ranges

VPH Hydrocarbon Range	Range Begins	Range Ends
C ₅ -C ₆ Aliphatic Hydrocarbons (FID)	Just before n-Pentane	Just after n-Hexane
>C ₆ -C ₈ Aliphatic Hydrocarbons (FID)	Just after n-Hexane	Just after n-Octane
>C ₈ -C ₁₀ Aliphatic Hydrocarbons (FID)	Just after n-Octane	Just after n-Decane
>C ₁₀ -C ₁₂ Aliphatic Hydrocarbons (FID)	Just after n-Decane	Just after n-Dodecane
>C ₈ -C ₁₀ Aromatic Hydrocarbons (PID)	Just after n-Octane as identified on PID (corresponding to just after Toluene)	Just after n-Decane as identified on PID (corresponding to just after 1,23-Trimethylbebezene)
>C ₁₀ -C ₁₂ Aromatic Hydrocarbons (PID)	Just after n-Decane as identified on PID (corresponding to just after 1,23-Trimethylbebezene)	Just after n-Dodecane as identified on PID (corresponding to just after Naphthalene)
>C ₁₂ -C ₁₃ Aromatic Hydrocarbons (PID)	Just after n-Dodecane as identified on PID (corresponding to just after Naphthalene)	Just after 1-Methylnaphthalene

10.8.2 Working Calibration Standards**10.8.2.1 Instruments Equipped with Autosamplers**

Using the calibration standard identified in section 7.2.1.1, the following calibrations levels are made up to a final volume of 100 mL.

	Amt. of VPH (μ L)	Conc. of VPH Cal Std	Final Conc. Of Cal Std Working Std.
Level I	0.8	50 ng/mL per analyte	0.4 ng/mL each
Level II	4	50 ng/mL per analyte	2 ng/mL each
Level III	20	50 ng/mL per analyte	10 ng/mL each
Level IV	60	50 ng/mL per analyte	30 ng/mL each
Level V	100	50 ng/mL per analyte	50 ng/mL each
Level VI	160	50 ng/mL per analyte	80 ng/mL each
Level VII	200	50 ng/mL per analyte	100 ng/mL each
Level VIII	240	50 ng/mL per analyte	120 ng/mL each

(Note: Additional levels may be added at the analyst's discretion to extend the calibration range. Always record the standard IDs on the raw data.)

10.8.3 (instruments attached to autosamplers) Start the sequence with a surrogated solvent blank, to demonstrate the absence of contamination. Starting with the lowest level standard, analyze each calibration mixture using the same conditions employed for the analysis of the retention time standard. The method prohibits the analysis of additional standards for the purpose of choosing a set of results that meet the calibration acceptance criteria

10.8.4 A collective calibration curve or factor must be established for each hydrocarbon range of interest on both the FID and the PID and a calibration curve or factor must be established for each individual target compound on the PID. Record the identification of the standards on the raw data.

For hydrocarbon ranges on both the FID and the PID, tabulate the summation of the peak areas of all components in the calibration standard which fall within the VPH Hydrocarbon ranges (see Table 4) against the total mass represented by those peaks. For the FID chromatogram, the total areas and masses of both aliphatic and aromatic compounds are summed within each range. For the PID chromatogram, only the areas and masses of the aromatic compounds are summed within each range. Do not include the area of any surrogate or internal standard in calculating a Range Calibration Factor.

For individual target compounds on the PID, tabulate area responses against mass injected.

Only the highest or lowest calibration points may be rejected because of non-linearity. Rejection of such a point may affect the reporting limit or the linear range. Points within the curve may not be rejected [this represents a departure from the previous revision of this SOP that permitted a rejection for a valid analytical reason, because frequently no reasons were provided or the reasons were found to be invalid (e.g., when the deleted level was included, the curve would pass for the majority of the compounds) and the required documentation was lacking in completeness]. When rejecting an extreme end level, remember that there must be a minimum of five standards remaining for a linear model and six points for a quadratic.

10.8.5 The ratio of the response to the amount injected, defined as the response factor (RF) or range RF, may be calculated for individual target compounds or hydrocarbon ranges at each standard concentration using Equation 1. An acceptable calibration is one in which the relative standard deviation (%RSD) of the response factor, as determined using Equation 2, does not exceed 20% over the working range for each individual compound or carbon range of interest. In the event that the 20% is exceeded, then linear regression or quadratic calibration is used. In either case, the correlation coefficient (r) or coefficient of determination (r²) must be ≥ 0.995 . Also, quadratic modeling requires a minimum of six calibration points.

Equation 1: Range Calibration Factor

$$\text{Range RF} = \frac{\text{Total Area of Peaks within a Range} \times \text{Conc}_{\text{IS}}}{\text{Peak Area}_{\text{IS}} \times \text{Total Mass Injected (ng) within a Range}}$$

Equation 2: Percent Relative Standard Deviation

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Mean of the RFs}} \times 100$$

Note: Non-linear calibrations should not be employed for methods or instruments previously shown to exhibit linear calibration for the analytes of interest. In other words, you can't employ a quadratic model to compensate for detector saturation or lapses in instrument maintenance.

10.8.6 2nd Source Calibration Verification

10.8.6.1 Upon completion of the initial calibration, verify the calibration by analyzing a second source calibration standard (7.2.2). The source of this standard should be different than the source from which the calibration standards were obtained. In situations where a single vendor is used to provide the primary and secondary standards, the analyst must obtain written warranties that the two references were not prepared from the same reference material.

10.8.6.2 All individual analytes should be within 20% of the expected value. Hydrocarbon ranges should be within 30% of the expected value.

10.8.7 Continuing Calibration Verification

10.8.7.1 The calibration curve (concentration or RF) must be verified prior to analysis of samples, after every ten (10) samples, and at the conclusion of analysis.

10.8.7.2 Several clients require that instrument contamination be monitored at the same frequency as CCV standards throughout the sequence.

10.8.7.3 Continuing verification may be completed using primary source calibration mixtures alternating between low and medium concentrations every 10 samples throughout the analytical sequence. Record the identification of the standards on the raw data.

10.8.7.4 The percent difference (Equation 3) for the measured concentrations or RFs in the initial verification must be < 20% for individual target analytes and hydrocarbon ranges. If acceptance criteria are not achieved, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the lab has to demonstrate acceptable performance after corrective action with two consecutive calibration verifications (using fresh calibration solutions, at low and high concentrations) or, alternatively, a new initial calibration must be established according to Section 10.3. If one of these calibration verification injections fails, a new initial calibration curve must be processed.

Equation 3: Percent Difference (%D)

$$\%D = \left| \frac{R_i - R_v}{R_i} \right| \times 100$$

where:

R_i = Standard concentration

R_v = Calculated concentration from verification check.

10.8.7.5 Any samples associated with failed closing calibration verifications where the response for an analyte in the calibration verification standard is above the acceptance limit and the analyte was not detected in any of the samples analyzed since the previous passing verification, do not need to be reanalyzed as the verification standard has demonstrated that the analyte would have been detected were it present. Re-analysis is required for all other situations. If for some reason (i.e., lack of sample) re-analysis can't take place, a NCM needs to be initiated and the sample and QC results associated with the failing CCV need to be qualified in the final report.

10.8.7.6 Internal standard areas should fall within 50% - 200% and the RRT should be within 30 seconds, as established by the initial calibration.

10.8.7.7 **Either a method or instrument blank spiked with surrogate must be analyzed immediately after each CCV standard. A batch number must be assigned to each method blank on the raw data, while instrument blanks may be designated as IBL- and continuing calibration blanks may be designated as CCB- (followed by a number to indicate their position in the sequence).** Acceptance criteria for Method and instrument blanks are found in section 9.7 of this SOP

10.8.7.8 The Targeted VPH Analytes and Aromatic Hydrocarbons are quantitated on the PID chromatogram.

10.8.7.9 The Aliphatic Hydrocarbons are quantitated on the FID chromatogram, after subtraction of the collective concentrations of MTBE, BTEX compounds and other aromatic compounds identified on the PID chromatogram.

10.9 Calculations / Data Reduction

10.10 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

10.11 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

10.12 Concentration = mg/kg or L =
$$\frac{C \times V \times D}{W}$$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.0 Method Performance**11.1 Method Detection Limit Study (MDL)**

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure (see SOP TA-QA-0602). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

11.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP TA-QA-0617 for details.

11.3 Training Requirements

See SOP TA-QA-0608 for detailed training requirements.

12.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

13.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner.

Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP TA-EHS-0036.

13.1 Waste Streams Produced by the Method

- 13.1.1** Aqueous waste generated from analysis. This material may have a pH of less than 2.0. All liquid waste generated from analysis is deposited into a large satellite drum, which when close to being full, the laboratory EH&S specialist removes and disposes of in the appropriate, regulatory manner.
- 13.1.2** Aqueous acidic waste from sample analysis, which may contain small amounts of methanol and sodium bisulfate. All liquid waste is deposited into a large satellite drum, which when close to being full, the laboratory EH&S specialist removes and disposes of in the appropriate, regulatory manner.
- 13.1.3** VOA vials containing extracted acidic water and small amounts of methanol. All liquid waste is deposited into a large satellite drum, which when close to being full, the laboratory EH&S specialist removes and disposes of in the appropriate, regulatory manner. The empty voa vials themselves are placed into a broken glass disposal box and sealed and placed in the general waste depository.
- 13.1.4** Solvent waste generated from analysis. All liquid waste is deposited into a large satellite drum, which when close to being full, the laboratory EH&S specialist removes and disposes of in the appropriate, regulatory manner.
- 13.1.5** VOA vials containing extracted soil samples, which will contain small amounts of methanol. The laboratory EH&S specialist removes and disposes of all soil samples in the appropriate, regulatory manner.
- 13.1.6** Solid waste generated from analysis. Empty voa vials and broken syringes are placed into a broken glass disposal box and sealed and placed in the general waste depository.
- 13.1.7** Expired Standards. Expired standards are emptied in to the flammable liquid waste satellite container, which, when full, is removed by the EH&S specialist and disposed of in the appropriate, regulatory manner. The containers they were housed in are placed in the same containers as the used voa vials.
- 13.1.8** Methanol rinses. All liquid waste is deposited into a large satellite drum, which when close to being full, the laboratory EH&S specialist removes and disposes of in the appropriate, regulatory manner.
- 13.1.9** Used samples. The laboratory EH&S specialist removes and disposes of all samples in the appropriate, regulatory manner.

14.0 References / Cross-References

- 14.1** "Method for the Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions," WA State Dept. of Ecology, June 1997.

15.0 Attachments

Attachment 1: Dilution Factors

16.0 Revision History

- Revision 7, dated 26 January, 2010
 - Added Naphthalene to list of analytes, Section 1.1.2
 - Updated reporting limits for soil Section 1.1.4
 - Updated purge gas for DI water from Nitrogen to Helium in relevant sections.
 - Updated equipment to reflect current set-up Section 6.1
 - Added audit trail information Section 6.1
 - Updated container information Section 6.1 and 10.2.3
 - Added documentation of standards and standard preparation Section 7.1
 - Added removal of expired standards Section 7.8
 - Added criteria for extra QC Section 9.3
 - Added balance check requirement Section 10.2.1
 - Updated sample/solvent quantities Section 10.2.4
 - Updated addition of surrogates Section 10.2.8
 - Integration for TestAmerica Tacoma and TestAmerica Seattle operations.
 - Removed method modification Section 15.0
 - Removed Start up procedure, Attachment 2.

Attachment 1. Dilution Factors

WATER	DILUTION FACTOR
5 mL	1
2.5 mL	2
2 mL	2.5
1 mL	5
500 µL	10
250 µL	20
200 µL	25
100 µL	50
50 µL	100
25 µL	200
10 µL	500
5 µL	1000
2 µL	2500
1 µL	5000
0.5 µL	10000
0.2 µL	25000
SOIL	DILUTION FACTOR
100 µL	1
50 µL	2
25 µL	4
10 µL	10
5 µL	20
2 µL	50
1 µL	100
0.5 µL	200
0.2 µL	500

APPENDIX C
DQO MEETING MINUTES

**DQO Meeting Report
For Development of
UFP-SAP for SWMU 28 – Maintenance Shop Building 1820 Area**

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 28 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 some historical photos of the area. The photos included an oil pan drain device that drained into a UST, an apparent UST on the edge of a hill that overflowed down the hill, and other photos of the area. He provided pdf copies on a CD.
2. Sediment samples will be collected on the SE and NW sides of the site and groundwater samples SE of the site to determine if contamination (if any) from the SWMU is leaving the site.
3. Groundwater samples will be collected during Phase I on the SE side of the site. Tentative sample locations proposed/discussed included: downgradient of the former Building 1818 USTs (2), downgradient of Building 3387/Battery Disposal Area (1), and downgradient of Building 1820 UST and gravel pad (2). One groundwater sample will also be collected on the upgradient side of the site. Groundwater samples can be collected from a geoprobe; temporary wells are not necessary.
4. Some of the fill used to widen the level area of the site includes former pavement material; therefore, PAH contamination such as benzo-a-pyrene may be found downgradient of site.
5. Because hydraulic fluids and other oils may contain PCBs, analysis of areas at, and downgradient of the USTs and the gravel pads should include PCBs.
6. Doug Griffin said that Indiana has some special guidance and requirements for TPH analysis. Tetra Tech will send email directly to Doug to obtain specifics.
7. The locations of samples at pipe discharge points may be determined by defining the coordinates of the discharge points on the drawings and then using GPS to find those locations in the field.
8. 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

Heavy Equipment Submittal

Initial Assessment Study

Kearney Report Excerpts

11010
Ser 09Z3/2002
09 September 02

From: Commander, Crane Division, Naval Surface Warfare Center
To: Commander, Naval Sea Systems Command (SEA 04XI)
Via: Commander, Southern Division, Naval Facilities Engineering
Command

Subj: CPP SPECIAL PROJECT C-302, CONSTRUCT HEAVY EQUIPMENT
MAINTENANCE FACILITY

Ref: (a) OPNAVINST 11010.20F

Encl: (1) DD Form 1391 and Accompanying Data Sheets for Subject
Special Project

1. Enclosure (1) is submitted in accordance with reference (a)
for review and approval.

2. This project is Capital Procurement Program (CPP) funded
project. The estimate is \$950,000, which is above the \$400,000
local approval threshold.

3. This is an FY02 Minor Construction project. Accordingly, we
request expeditious review and approval in order to allow for
award by the end of FY02.

4. Crane's point of contact is Mr. Dan Geldrich, Code 09Z3,
telephone DSN 482-4482 or commercial 812-854-4482.

J. D. HEDGES
By direction

Copy to:
NAVSEA (04XI) (Advance copy)

1. COMPONENT NAVY	FY 2002 MILITARY CONSTRUCTION PROGRAM		2. Date 09/05/2002
3. Installation and Location/UIC: NAVSEA CRANE – SURFACE WARFARE CENTER		4. Project Title Heavy Equipment Maintenance Facility	
5. Program Element	6. Category Code 214-20	7. Project Number C-302	8. Project Cost (\$000) 950

9. COST ESTIMATES				
ITEM	U/M	QUANTITY	UNIT COST	COST (\$000)
CONSTRUCT HEAVY EQUIPMENT MAINTENANCE SHOP				806
GENERAL BUILDING CONSTRUCTION				(383)
SITE CONSTRUCTION				(153)
HVAC				(52)
EXHAUST VENTILATION				(52)
PLUMBING				(5)
FIRE SUPPRESSION				(16)
ELECTRICAL				<u>(145)</u>
SUBTOTAL				806
CONTINGENCY (10%)				80
SIOH (8%)				<u>64</u>
TOTAL REQUEST				950
EQUIPMENT FROM OTHER APPROPRIATIONS			(NON-ADD)	

10. DESCRIPTION OF PROPOSED CONSTRUCTION:

The construction of a 11,000 sf "pre-engineered", steel framed, metal sided, heavy equipment maintenance facility. The size of the building is 155' long by 71' wide, outside of girt to outside of girt. The width of the building has been determined by the requirements of the longest vehicles to be serviced. There will be one 22' wide bay, two 32' bays and two 24' bays.

11. Requirement: _____ M2 **Adequate:** _____ M2 **Substandard:** _____ M2

PROJECT:

The project will consist of construction of the heavy equipment maintenance facility, along with all the associated utilities. The new building will be built adjacent to existing building 2713. Upon completion of this project, the existing heavy equipment maintenance facility will be mothballed for demolition, pending funds availability.

REQUIREMENT:

A new facility is required for maintenance and repair of heavy equipment in the activity's \$9 million heavy

1.COMONENT NAVY	FY MILITARY CONSTRUCTION PROGRAM	2. Date 9/5/2002
3. Installation and Location/UIC: NAVSEA CRANE – SURFACE WARFARE CENTER		
4. Project Title HEAVY EQUIPMENT MAINTENANCE FACILITY	5. Project Number C-302	

equipment inventory. This equipment is essential for the Fleet for many vital weapons subsystems, equipment and components; pyrotechnics; small arms and other critical items. The equipment also heavily supports Crane Army Ammunition Activity, a major tenant and Single Manager for Conventional Ammunition, in its production, storage and transport operations. The inventory includes 250 pieces of construction equipment, including bulldozers, air compressors and truck-mounted cranes. Maintenance and repair of these items are required to insure the reliability, safety, and operability of the equipment as well as prolong the life expectancy of the costly inventory.

CURRENT SITUATION :

Heavy equipment maintenance functions are currently performed in a deteriorated, inadequate wooden structure which was built during World War II from form lumber that had been used in the construction of other buildings at the activity. The wooden structure has exceeded its life expectancy and further repair is no longer feasible. Structural analysis and inspection of the trusses indicate that a 44% overstress exists in the top chord members which are beginning to buckle. Bottom chord members have already failed, requiring temporary supports to be installed. Analysis also indicates structural uplift problems when the facilities are subjected to average wind load. These structural weaknesses could possibly endanger lives and equipment during violent windstorms or snowstorms and will cause serious impairment to automotive maintenance operations should further damage or collapse of the deteriorated structure occur.

IMPACT IF NOT PROVIDED

If the existing heavy equipment maintenance facility is not replaced, the activity will be unable to adequately maintain its large inventory of motorized equipment and accomplish assigned tasks. Further deterioration will result in abandonment of the exiting structure. Reduced reliability and shortages of automotive equipment will severely impact upon the ability of the activity and its tenants to quickly respond to vital military requirements.

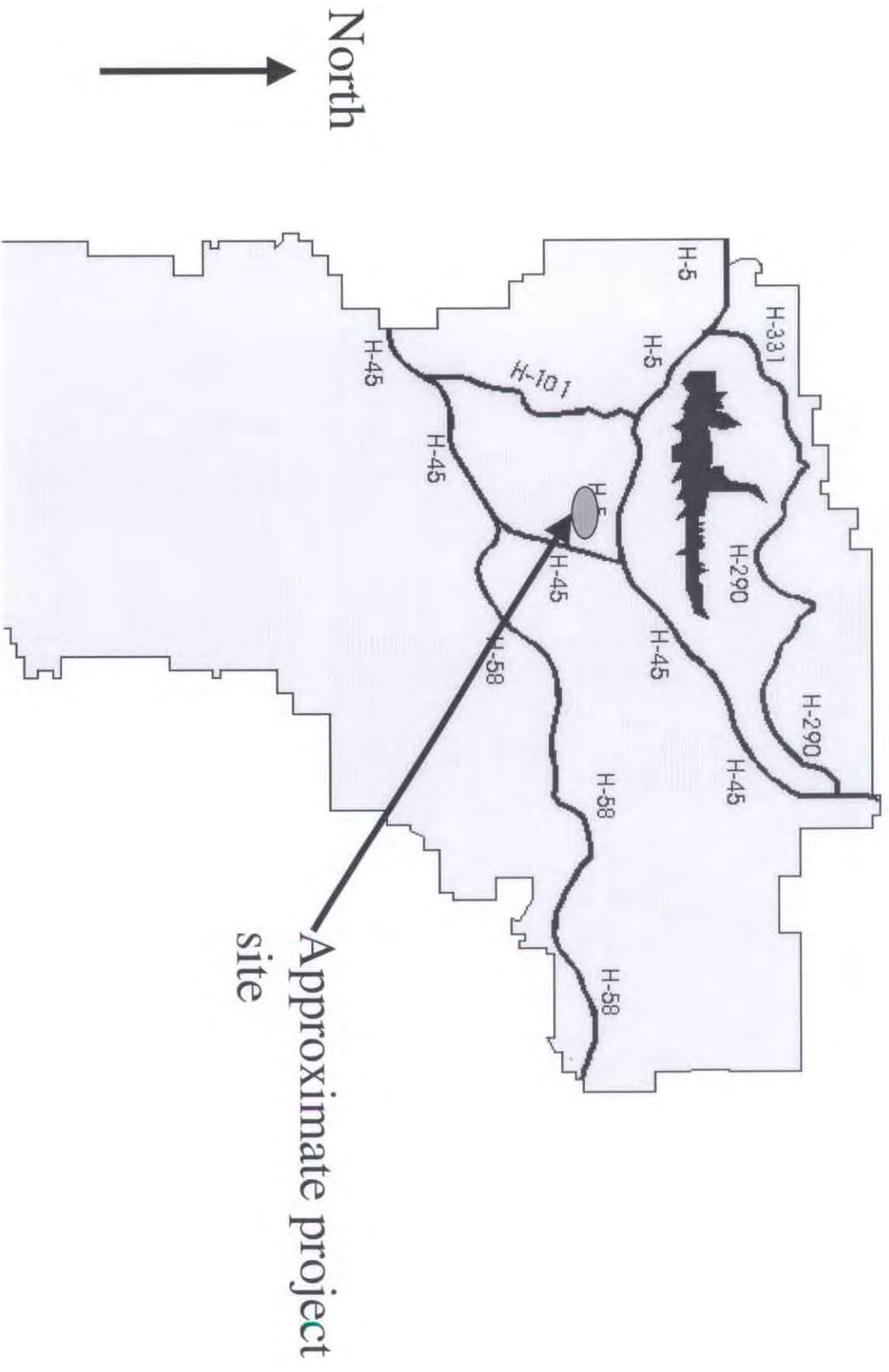
COST ESTIMATE FOR HEAVY EQUIPMENT MAINTENANCE FACILITY

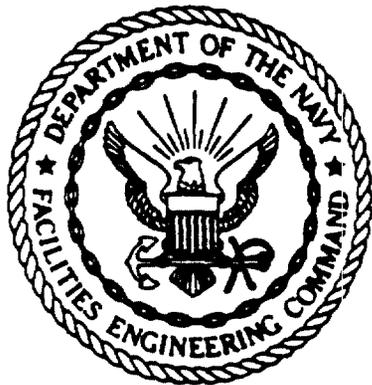
	<u>\$ K</u>		
General Building Construction			382
Foundation Construction	52		
Substructure Construction	92		
Superstructure Construction	124		
Exterior Closure Construction	99		
Interior Wall Construction	15		
Site Construction			153
Site Preparation	67		
Utility Construction	23		
Pavement / Roadway Construction	63		
	<u>SF</u>	<u>\$ / SF</u>	
*HVAC	11,000	4.73	52
*Exhaust Ventilation	9,800	5.30	52
*Plumbing	9,800	0.51	5
*Fire Suppression	11,000	1.45	16
*Electrical	11,000	13.18	145
			<hr/>
Subtotal			805
Contingency		10%	80
SIOH		8%	64
			<hr/>
Total Cost			950

* These costs developed using a \$/SF average

GENERAL BUILDING CONSTRUCTION DETAILS					382,122
Description	Qty	Unit	Unit Cost	Tot Cost	
FOUNDATION CONSTRUCTION					52,010
Rein. Concrete Sp. Footing/Grade Beam	60	CY	170	10,200	
Reinforced Concrete Drilled Piers	20	EA	1700	34,000	
Reinforced Concrete Column Footings	20	CY	170	3,400	
8" x 3" Concrete Found. Wall	30	CY	147	4,410	
SUBSTRUCTURE CONSTRUCTION					92,032
8" Reinforced Concrete Slab on Grade	9800	SF	5	49,000	
5" Reinforced Concrete Slab on Grade	1200	SF	4	4,800	
Vapor Barrier	11000	SF	0.1	1,100	
1" Slab Topping Hardener	9844	EA	3	29,532	
6" Aggregate Base	200	CY	38	7,600	
SUPERSTRUCTURE CONSTRUCTION					124,080
Pre-Engineered, Metal Building	11000	SF	11.28	124,080	
EXTERIOR CLOSURE CONSTRUCTION					99,000
Flashing	700	SF	4.57	3,200	
Metal Louvers	50	SF	18.00	900	
prefinished Metal Gutters	340	LF	4.41	1,500	
prefinished Metal Down Spouts	100	LF	9.00	900	
20' X 16' Overhead Coiling Doors, Mech	3	EA	8,500.00	25,500	
28' X 16' Overhead Coiling Doors, Mech	4	EA	11,075.00	44,300	
14' X 16' Overhead Coiling Doors, Mech	3	EA	6,000.00	18,000	
Double, HM Door 7 Frame W/Hardware	2	EA	900.00	1,800	
Single, HM Door & Frame W/Hardware	4	EA	725.00	2,900	
WALL CONSTRUCTION					15,000
8" cmu Wall Construction	2500	SF	6.00	15,000	
SITE CONSTRUCTION DETAILS					152,900
Description	Qty	Unit	Unit Cost	Tot Cost	
SITE PREPARATION					66,750
Remove Existing Pavement & Base Matl	20000	SF	1	20,000	
Excavate & Remove approx 2" of Soil	1500	CY	10	15,000	
Import/place Controlled Fill	1950	CY	15	29,250	
Saw Cut Existing Asphalt Pavement	500	LF	5	2,500	
UTILITY CONSTRUCTION					23,030
Excavation/Backfill/Compaction	800	CY	5	4,000	
New 6" Sanitary Pipe to main	80	LF	26	2,080	
Tie into Existing Sanitary Pipe	1	EA	500	500	
New Sanitary Manholes	1	EA	2090	2,090	
New 12" Storm Sewer Pipe	370	LF	29	10,730	
New Concrete Catch Basins	4	EA	240	960	
New Reinforced Concrete Headwall	1	EA	500	500	
New 6" Water Main	100	LF	16	1,600	
Tie into Exist Domestic Water Main	1	EA	570	570	
PAVEMENT/ROADWAY CONSTRUCTION					63,120
Excavation/Backfill/Compaction	500	CY	5	2,500	
6" Agg. Base Course	440	CY	35	15,400	
3" Asphalt Concrete Base Course	220	CY	68	14,960	
1 1/2" Asphalt Concrete Surface Course	110	CY	72	7,920	
8" Reinforced Concrete Slab	2760	SF	4.5	12,420	
1" Slab Topping/Hardener	2760	SF	3.25	8,970	
Signage/Striping Allowance	1	ALL	950	950	

SITE MAP FOR NEW HEAVY EQUIPMENT MAINTENANCE FACILITY, NSWCC, CRANE, IN





May 1983

**INITIAL ASSESSMENT STUDY OF
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA**

NEESA 13-003



**NAVAL ENERGY AND ENVIRONMENTAL
SUPPORT ACTIVITY**

Port Hueneme, California 93043

RELEASE OF THIS DOCUMENT REQUIRES PRIOR NOTIFICATION
OF THE CHIEF OFFICIAL OF THE STUDIED ACTIVITY.

NWSC has about 170 miles of railroad tracks. Derailments have occurred throughout the history of the base. However, accidents where chemicals could have been spilled or released to the environment have not occurred at NWSC Crane on the base railroad or on the Milwaukee Railroad running through the base.

6.2.5 Automotive and Heavy Equipment Maintenance Shops and Garage Area

About 50 to 75 gallons per year of carwash cleaner is used at the washrack in the north end of Building 1820. Also, 50 to 75 gallons per year of steam cleaners is used in the adjacent steam washrack. Wastes flow into an oil/water separator adjacent to Building 1820. Prior to about 1972, the oily wastewater flowed into a ditch and eventually into Boggs Creek. Waste oil is collected in a tank at Building 1820. Boiler shop personnel remove approximately 500 gallons of oil per month from this tank and from the oil/water separator. Salvageable oil is burned as fuel in the Building 150 boiler.

About 200 to 300 automotive batteries are disposed of near the garage area. In past years, the battery acid was dumped out of the battery and down the side of the ravine behind buildings 1820 and 1818.

Two solvent dip tanks are located in Building 1820. Each tank, which holds about 20 gallons of solvent, agitene, is emptied about twice a year. The waste solvent was disposed of in the waste oil tank at Building 1820. Currently, waste solvents and oils are segregated and disposed through DPDO.

A washrack is located at the northwest corner of the garage area. The rack is used to clean mud off trucks and to clean out concrete mixer trucks. Only water is used at this washrack. Visual inspection of the washrack and surrounding area revealed that mud and concrete had been washed into the ravine that starts under the rack, but signs of chemical contamination were not present.

6.2.6 Pesticides Shop

2,4D (also known as Tordon) is a broad-leaf killer, 2,4,5-T and 2,4-D are used on lawns at NWSC Crane.

In 1980, 2,4,5-T was applied to 59 miles of fenceline at a rate of 245 gallons per acre. Fencelines were sprayed previously in 1960, 1965, and 1968. Prior to 1960, fencelines were not sprayed.

In 1969, MH30 (maleic, hydrazide, and diethanolamine salt of 6-Hydroxy-3-(2H) Pyridazinone, 58%) was applied on 100 magazines and to both sides of Route 45 from the Bloomington gate to four miles southward, as a trial. The chemical did not work satisfactorily and was not used again.

From about 1950 to 1970, Telvan (80% Monuraon (3-(p-chlorophenyl)-1,1-dimethylurea), made by Dupont, was applied yearly at a rate of 10 to 80 pounds per acre on railroad right of ways.

From about 1950 to 1970, Urealer, by U.S. Borax and Chemical Co., was applied yearly at a rate of 1 to 3 pounds per 100 square feet on magazine gravel drives and electrical substations.

**PRELIMINARY REVIEW/VISUAL SITE INSPECTION REPORT
OF
NAVAL WEAPONS SUPPORT CENTER
CRANE, INDIANA
EPA ID IN5170023498**

Prepared for:

**U.S. Environmental Protection Agency
Region V
230 South Dearborn Street
Chicago, IL 60604**

Prepared by

**A. T. Kearney, Inc.
699 Prince Street
Alexandria, VA 22313**

**EPA Contract No. 68-01-7038
Work Assignment R05-02-45**

March 1987

29. UNIT NAME: Auto Maintenance Shop - Building 1820 (Ref. 3, p.6-46)

Unit Description: Automotive repairs are performed in this building. The unit consists of several sumps which collect waste oil, wastewater, and several open top solvent cleaning tanks. Three drums were observed outside of the shop which were in poor condition (rusted and dented), these drums contained lube oil.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Waste oil and rinse waters are allowed to drain via floor drains into an outside Oil/Water Separator (Unit 32) (Ref. 20). Waste solvent is containerized and taken to the CSF (Unit 47). One of the solvent tanks called the "Parts Boiler" produces an alkaline sludge that is removed to one of the used oil tanks (Ref. 20).

Release Controls: The solvent tanks have no secondary containment. Floor drains in the sumps and floors drain to the Oil/Water Separator (Unit 32).

Release History: Noticeable oil spills and solvent contaminated rags were noted during the VSI.

UNIT 29. (Continued)

Conclusions:

Soil/Groundwater: The potential for release to soil/groundwater is low due to the unit's indoor setting over a concrete floor.

Surface Water: The potential for release to surface water is low due to the unit's indoor setting and the collection of any rinse waters in an Oil/Water Separator.

Air: The potential for release to air is moderate from the solvent wash tanks due to their open-top design.

Subsurface Gas: There is no potential for generation of subsurface gas due to the nature of the wastes and the open nature of the sumps and tanks.

Suggested Further Action:

No further action is suggested for this unit at this time.

30. UNIT NAME: Heavy Equipment Maintenance Shop - Building 1818

Unit Description: Heavy equipment repairs are performed in this building. The unit consists of several sumps that collect waste oil and several open top solvent cleaning tanks.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Waste oil and rinse waters are allowed to drain into an outside Oil/Water Separator (Unit 32) via floor drains (Ref. 20). Waste solvent is containerized and taken to the CSF (Unit 47).

Release Controls: The solvent tanks have no secondary containment. Floor drains route flow into the Oil/Water Separator.

Release History: Noticeable oil spills and solvent contaminated rags were noted during the VSI.

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the unit's indoor setting on a concrete floor.

Surface Water: The potential for release to surface water is low due to the unit's indoor setting and the collection of any rinse waters in an Oil/Water Separator.

Air: The potential for release to air is moderate from the solvent wash tanks due to their open-top design.

Subsurface Gas: There is no potential for generation of subsurface gas due to the nature of the wastes and the open nature of the sumps and tanks.

Suggested Further Action: No further action is suggested for this unit at this time.

31. UNIT NAME: Truck Wash Area at the Heavy Equipment Maintenance Building

Unit Description: This unit consists of two indoor concrete wash racks for trucks and heavy equipment. Rinse waters and any waste oil drain into two drip tracks that drain into the Oil/Water Separator (Unit 32).

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Rinse waters are generated from truck wash-downs.

Release Controls: The unit has sloped concrete floors that drain to two drip tracks.

Release History: Unknown.

UNIT 31. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the unit's release controls.

Surface Water: The potential for release to surface water is low because the unit drains into the Oil/Water Separator.

Air: The potential for release to air is low due to the dilute nature of the wastes handled.

Subsurface Gas: The potential for generation of subsurface gas is low due to the open nature of the unit and dilute nature of the wastes.

Suggested Further Action: No further action is suggested for this unit at this time.

32. UNIT NAME: Oil/Water Separator at the Heavy Equipment
Maintenance Building

Unit Description: This unit is a below-grade sump outside of Building 1820 that receives rinse waters containing oil/degreasers from the Auto and Heavy Equipment Maintenance Buildings. A thin conveyor belt skims oil and deposits it in a separate adjacent concrete sump where it can be pumped out and into a waste oil storage tank.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Oil and degreaser contaminated rinse water is skimmed in the unit. Waste oil is collected in a separate adjacent sump and the remaining water is discharged to the Sanitary Sewer System.

Release Controls: The unit has a removable closed top and overflow valve into the Sanitary Sewer System.

Release History: Unknown.

UNIT 32. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the unit's release controls and the apparent good condition of the unit.

Surface Water: The potential for release to surface water is low due to the apparent good working condition of the oil skimmer and subsequent discharge to the Sanitary Sewer System.

Air: The potential for release to air is low due to the dilute nature of the wastes.

Subsurface Gas: The potential for generation of subsurface gas is low due to the dilute nature of the wastes.

Suggested Further Action: No further action is suggested for this unit at this time.

33. UNIT NAME: Outside Truck Wash Rack adjacent to Building 1818

Unit Description: This unit consists of a truck wash hose and a raised wooden slat platform. Trucks are allowed to wash down on the rack with rinse waters going through the slats and down the hill into an intermittent stream.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Rinse waters from heavy equipment and truck washings are generated and flow through the raised platform and down the hill.

Release Controls: There are no release controls associated with the unit.

Release History: Due to the unit's design, continuous release of rinse waters to the ground beneath the unit occurs during washing.

UNIT 33. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is high due to the unit's construction.

Surface Water: The potential for release to surface water is high because the unit's effluent flows directly into an intermittent stream.

Air: The potential for release to air is low due to the dilute nature of the wastes.

Subsurface Gas: The potential for generation of subsurface gas is low due to the dilute nature of the wastes.

Suggested Further Action:

1. Provisions should be made to collect rinse waters and treat these wastes at the sewage plant.
2. Soils should be sampled underneath the platform and in the drainage ditch that flows down the hillside.

34. UNIT NAME: Roll-Off Boxes Outside Building 1820

Unit Description: These units are typical metal roll-off boxes containing scrap cardboard, wood, and general garbage that is hauled to the Sanitary Landfill (Unit 86). These units are common throughout the site at each building that has any type of ongoing activity.

Date of Start-Up: Unknown.

Date of Closure: Numerous roll-off boxes are in use throughout the site.

Waste Managed: Scrap cardboard, wood, and general garbage is placed in the unit until disposal in the Sanitary Landfill.

Release Controls: The units are located on the paved areas behind each building.

Release History: Unknown.

UNIT 34. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to the nature of the wastes handled.

Surface Water: The potential for release to surface water is low due to the nature of the wastes handled.

Air: The potential for air release is low due to the nature of the wastes handled and the short storage periods.

Subsurface Gas: The potential for generation of subsurface gas is low due to the above-ground design of the units.

Suggested Further Action: No further action is suggested for this unit at this time.

35. UNIT NAME: CONEX Hazardous Waste Transfer Containers
behind Building 820

Unit Description: These units are yellow painted steel transfer vaults in which 4 drums of waste can be placed and transferred to a storage area. There are numerous CONEX containers on site, one of which was behind Building 1820.

Date of Start-Up: Unknown.

Date of Closure: Numerous CONEX containers are in use throughout the site.

Waste Managed: Containerized hazardous waste is temporarily stored in the vaults for transfer between storage areas. The vaults are then moved by forklift and loaders.

Release Controls: The units serve as a release control for containerized wastes during transfer. The units are located on paved areas behind production buildings where containerized waste is generated.

Release History: Unknown.

UNIT 35. (Continued)

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is low due to containerization of wastes within the unit.

Surface Water: The potential for release to surface water is low due to containerization of waste within the unit.

Air: The potential for release to air is low due to containerization of wastes within the unit.

Subsurface Gas: There is no potential for the generation of subsurface gas due to the design characteristics of the unit.

Suggested Further Action: No further action is suggested for this unit at this time.

36 UNIT NAME: Oil Pan Wash Out/Disposal Rack
Adjacent to Building 1820

Unit Description: This unit consists of a metal drip pan that gravity feeds a pipe which drains into an underground waste oil storage tank. The pan is erected on wooden posts and a dissipated wooden overhang.

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: Waste oil from various drip pans and gallon jugs are emptied and washed in the metal drip pan.

Release Controls: There are no release controls employed at the unit.

Release History: The ground beneath the unit is covered with oil stains with remnants of oil encrusted grass at the foot of the posts.

Conclusions: Soil/Groundwater: The potential for release to soil/groundwater is high due to spillage of waste oil onto the ground.

Surface Water: The potential for release to surface water is moderate due to possible run-off from the affected soils into an intermittent stream at the base of the hill.

Air: The potential for release to air is low due to the nature of the waste.

Subsurface Gas: The potential for generation of subsurface gas is low due to the open nature of the unit.

- Suggested Further Action:
1. Soil which shows obvious signs of soil contamination should be removed.
 2. Soil sampling should be performed after removal of visually contaminated soil to verify that there is no further contamination.
 3. The facility should take steps to ensure no future oil spills (i.e., installation of a containment pad and larger drip pan).

37. UNIT NAME: Underground Waste Oil Storage Tank -- Building 1818

Unit Description: This unit is a single shell steel storage tank that has a capacity of 500 gallons. It receives waste oil from the Oil Pan Wash Out (Unit 36) and oil from the Auto Maintenance Shops (Unit 29).

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: The underground storage tank is used for storage of waste oil prior to transfer to the storage tank in Building 2801 or use as boiler feed.

Release Controls: The unit does not have a leak detection system.

Release History: Severe erosion of the hillside near the unit had fully exposed its vent-pipe and part of the tank (Ref. 20).

UNIT 37. (Continued)

Conclusions: Soil/Groundwater: There is a high potential for release to soil/groundwater dependent on the age of the tank and the fact that there is no leak detection system.

Surface Water: The potential for release to surface water is low due to the unit's construction.

Air: The potential for release to air is low due to the design of the unit

Subsurface Gas: The potential for generation of subsurface gas is moderate dependent on the integrity of the tank.

Suggested Further Action: The integrity of the unit should be inspected.

38. UNIT NAME: Underground Waste Oil Storage Tank -- Building 1820

Unit Description: This unit is a single shell steel storage tank that has a capacity of 500 gallons. It receives waste oil from the Heavy Equipment Maintenance Shop (Unit 30) and the Oil/Water Separator (Unit 32).

Date of Start-Up: Unknown.

Date of Closure: The unit is currently active.

Waste Managed: The storage tank is used to store waste oils until transfer to Building 2801 or use as boiler feed.

Release Controls: The unit does not have a leak detection system.

Release History: Unknown.

UNIT 38. (Continued)

Conclusions: Soil/Groundwater: There is a high potential for release to soil/groundwater dependent on the age and integrity of the tank and the fact that there is no leak detection system.

Surface Water: The potential release to surface water is low due to the units construction.

Air: The potential for release to air is low due to the design of the unit.

Subsurface Gas: The potential for generation of subsurface gas is moderate dependent on the integrity of the tanks.

Suggested Further Action: The integrity of the unit should be inspected.

APPENDIX E

Project Screening Level Backup Documentation

NSA Crane SWMU 28 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-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-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
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
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
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	2000 N	140	5.8	5.8
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
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
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
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
Vinyl chloride	75-01-4	0.060 C	0.060 C	0.000056	0.0011	1.5	0.013	0.013	0.0011	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
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(4)	340 N(4)	360 (4)	7200 (4)	1100	18	18	18	IDEM-RDCL	170000 N(4)	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(5)	170 N(5)	120 (5)	2400 (5)	NA	NA	NA	170	R-RSL	17000 N(5)	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(5)	170 N(5)	120 (5)	2400 (5)	470	13	13	13	IDEM-RDCL	17000 N(5)	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(6)	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
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(6)	0.11 N(6)	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														
Cadmium	7440-43-9	70 N	7 N	1.4	28	12	7.5	7.5	7	R-RSL	800 N	990	77	77
Chromium	7440-47-3	0.29 C(7)	0.29 C(7)	0.00083 (7)	0.017 (7)	430(7)	38(7)	38(7)	0.017	RBSSL	5.6 C(7)	650	120	120
Copper	7440-50-8	3100 N	310 N	51	1020	14000	920	920	310	R-RSL	41000 N	62000	2900	2900
Lead	7439-92-1	400 (6)	400 (6)	14 (6)	280 (6)	400	81	81	81	IDEM-RDCL	800 (6)	1300	230	230
Zinc	7440-66-6	23000 N	2300 N	680	13600	100000	14000	10000	2300	R-RSL	310000 N	470000	38000	10000
TPH (GRO, DRO, ERO)														
GRO (C5-C12) Gasoline Range	NA	NA	NA	NA	NA	3100	120	120	120	IDEM-RDCL	NA	4300	1500	1500
DRO (C8-C28) Diesel Range	NA	NA	NA	NA	NA	3100	230	230	230	IDEM-RDCL	NA	5800	2300	2300
ERO (C8-C34) High End Hydrocarbons	NA	NA	NA	NA	NA	3100	230	230	230	IDEM-RDCL	NA	5800	2300	2300
TPH Fractionation														
Aliphatic EC > 5-6	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 6-8	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 8-10	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 10-12	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 12-16	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 16-21	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA
Aliphatic EC > 21-34	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA	NA

NSA Crane SWMU 28 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
						Residential Direct Contact	Migration to Groundwater	Residential Default Closure Level		
Aromatic EC > 8-10	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL
Aromatic EC > 10-12	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL
Aromatic EC > 12-16	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL
Aromatic EC > 16-21	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL
Aromatic EC > 21-34	NA	NA	NA	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL

EPA Regional Screening Level, Industrial Soil ⁽¹⁾ (mg/kg)
NA

2009 IDEM RISC Industrial Closure Levels for Soil (mg/kg) ⁽³⁾		
Industrial Direct Contact	Migration to Groundwater	Industrial Default Closure Level
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 - Value is for acenaphthene.

5 - Value is for pyrene.

6 - One tenth the noncarcinogenic value is less than the carcinogenic value; therefore, the noncarcinogenic value is presented.

7 - Value is for hexavalent chromium.

8 - Office of Solid Waste and Emergency Response soil screening level (EPA, 1994b)

9 - 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 28 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-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-Dichloroethane	107-06-2	0.15 C	0.15 C	2.0 C	2.0 C	5	2	5	0.15	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
Chloroethane	75-00-3	21000 N	2100 N	22000 N	2200 N	NA	62	62	62	IDEM G-DCL
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
Ethylbenzene	100-41-4	1.5 C	1.5 C	3.0 C	3 C	700	1600	700	1.5	T-RSL
Methyl-tert-butyl ether	1634-04-4	12 C	12 C	390 C	390 C	NA	40	40	12	T-RSL
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
Trichloroethene	79-01-6	2.0 C	2.0 C	3.0 C	3.0 C	5	2.8	5	2.0	T-RSL
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
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 ⁽⁵⁾	220 N ⁽⁵⁾	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 ⁽⁶⁾	110 N ⁽⁶⁾	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 ⁽⁶⁾	110 N ⁽⁶⁾	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										
Cadmium	7440-43-9	18 N	1.8 N	NA	NA	5	18	5	1.8	T-RSL
Chromium	7440-47-3	0.043 C ⁽⁷⁾	0.043 C ⁽⁷⁾	NA	NA	100	NA	NA	0.043	T-RSL
Copper	7440-50-8	1500 N	150 N	NA	NA	1300	1500	1300	150	T-RSL

Crane SWMU 28 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
Lead	7439-92-1	15 ⁽⁶⁾	15 ⁽⁸⁾	NA	NA	15	15	15	15	T-RSL
Zinc	7440-66-6	11000 N	1100 N	NA	NA	NA	11000	11000	1100	T-RSL

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 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 - Value is for acenaphthene.

6 - Value is for pyrene.

7 - Value is for hexavalent chromium.

8 - Action level under Safe Drinking Water Act.

Abbreviations:

NA - Not available or not applicable

N - Noncarcinogen

C - carcinogen

NSA Crane SWMU 28 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-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-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
Benzene	71-43-2	1.1 C	1.1 C	8.4	0.034	0.034	IDEM-RDCL	5.4 C	14	0.35
Chloroethane	75-00-3	15000 N	1500 N	80	0.65	0.65	IDEM-RDCL	61000 N	120	10
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	2000 N	140	5.8
Ethylbenzene	100-41-4	5.4 C	5.4 C	4600	13	5.4	R-RSL	27 C	6800	160
Methyl-tert-butyl ether	1634-04-4	43 C	43 C	350	0.18	0.18	IDEM-RDCL	220 C	650	3.2
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
Trichloroethene	79-01-6	2.8 C	2.8 C	4.9	0.057	0.057	IDEM-RDCL	14 C	24	0.35
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	R-RSL	2700 N	890	170
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 ⁽⁴⁾	340 N ⁽⁴⁾	1100	18	18	IDEM-RDCL	170000 N ⁽⁴⁾	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 ⁽⁵⁾	170 N ⁽⁵⁾	NA	NA	170	R-RSL	17000 N ⁽⁵⁾	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	3.6 C	3200	0.7	0.7	IDEM-RDCL	18 C	8000	170
Phenanthrene	85-01-8	1700 N ⁽⁵⁾	170 N ⁽⁵⁾	470	13	13	IDEM-RDCL	17000 N ⁽⁵⁾	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 ⁽⁶⁾	0.11 N ⁽⁶⁾	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

NSA Crane SWMU 28 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
Total PCBs	1336-36-3	NA	NA	NA	NA	NA	None	NA	NA	NA
Metals										
Cadmium	7440-43-9	70 N	7 N	12	7.5	7	R-RSL	800 N	990	77
Chromium	7440-47-3	0.29 C ⁽⁷⁾	0.29 C ⁽⁷⁾	430	38	0.29	R-RSL	5.6 C ⁽⁷⁾	650	120
Copper	7440-50-8	3100 N	310 N	14000	920	310	R-RSL	41000 N	62000	2900
Lead	7439-92-1	400 ⁽⁸⁾	400 ⁽⁸⁾	400	81	81	IDEM-RDCL	800 ⁽⁹⁾	1300	230
Zinc	7440-66-6	23000 N	2300 N	100000	10000	2300	R-RSL	310000 N	470000	10000
TPH (GRO, DRO, ERO)										
GRO	NA	NA	NA	3100	120	120	IDEM-RDCL	NA	4300	1500
DRO	NA	NA	NA	3100	230	230	IDEM-RDCL	NA	5800	2300
ERO	NA	NA	NA	3100	230	230	IDEM-RDCL	NA	5800	2300
TPH Fractionation										
Aliphatic EC > 5-6	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 6-8	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 8-10	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 10-12	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 12-16	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 16-21	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aliphatic EC > 21-34	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aromatic EC > 8-10	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aromatic EC > 10-12	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aromatic EC > 12-16	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aromatic EC > 16-21	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	NA	NA	NA
Aromatic EC > 21-34	NA	NA	NA	NA	TBD	TBD	IDEM-RDCL	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 - Value is for acenaphthene.

5 - Value is for pyrene.

6 - One tenth the noncarcinogenic value is less than the carcinogenic value; therefore, the noncarcinogenic value is presented.

7 - Value is for hexavalent chromium.

8 - Office of Solid Waste and Emergency Response soil screening level (EPA, 1994b)

9 - 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

NSA Crane SWMU 28 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

SSL - Soil Screening Level

Crane SWMU 28 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-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-Dichloroethane	107-06-2	0.15 C	0.15 C	5	2	5	0.15	T-RSL
Benzene	71-43-2	0.41 C	0.41 C	5	5.5	5	0.41	T-RSL
Chloroethane	75-00-3	21000 N	2100 N	NA	62	62	62	IDEM G-DCL
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
Ethylbenzene	100-41-4	1.5 C	1.5 C	700	1600	700	1.5	T-RSL
Methyl-tert-butyl ether	1634-04-4	12 C	12 C	NA	40	40	12	T-RSL
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
Trichloroethene	79-01-6	2.0 C	2.0 C	5	2.8	5	2.0	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
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 ⁽⁵⁾	220 N ⁽⁵⁾	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 ⁽⁶⁾	110 N ⁽⁶⁾	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
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 ⁽⁶⁾	110 N ⁽⁶⁾	NA	23	23	23	IDEM G-DCL
Pyrene	129-00-0	1100 N	110 N	NA	1100	1100	110	T-RSL
Polychlorinated Biphenyls								
Aroclor-1016	12674-11-2	0.96 C	0.96 C	0.5	0.5	0.43	0.43	IDEM GW

Crane SWMU 28 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
Aroclor-1221	11104-28-2	0.0068 C	0.0068 C	0.5	0.5	0.43	0.0068	T-RSL
Aroclor-1232	11141-16-5	0.0068 C	0.0068 C	0.5	0.5	0.43	0.0068	T-RSL
Aroclor-1242	53469-21-9	0.034 C	0.034 C	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1248	12672-29-6	0.034 C	0.034 C	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1254	11097-69-1	0.034 C	0.034 C	0.5	0.5	0.43	0.034	T-RSL
Aroclor-1260	11096-82-5	0.034 C	0.034 C	0.5	0.5	0.43	0.034	T-RSL
Total PCBs	1336-36-3	NA	NA	0.5	0.5	0.43	0.43	IDEM GW
Metals								
Cadmium	7440-43-9	18 N	1.8 N	5	18	5	1.8	T-RSL
Chromium	7440-47-3	0.043 C ⁽⁷⁾	0.043 C ⁽⁷⁾	100	NA	NA	0.043	T-RSL
Copper	7440-50-8	1500 N	150 N	1300	1500	1300	150	T-RSL
Lead	7439-92-1	15 ⁽⁸⁾	15 ⁽⁸⁾	15	15	15	15	T-RSL
Zinc	7440-66-6	11000 N	1100 N	NA	11000	11000	1100	T-RSL

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 - Value is for acenaphthene.

6 - Value is for pyrene.

7 - Value is for hexavalent chromium.

8 - Action level under Safe Drinking Water Act.

Abbreviations:

NA - Not available or not applicable

N - Noncarcinogen

C - carcinogen

NSA Crane SWMU 28 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-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-Dichloroethane	107-06-2	21.2	R5 ECOS	NA	21.2	21.2
Benzene	71-43-2	0.255	R5 ECOS	NA	0.255	0.255
Chloroethane	75-00-3	--	--	NA	NA	NA
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
Ethylbenzene	100-41-4	5.16	R5 ECOS	NA	5.16	5.16
Methyl-tert-butyl ether	1634-04-4	--	--	NA	NA	NA
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
Trichloroethene	79-01-6	12.4	R5 ECOS	NA	12.4	12.4
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
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						
Cadmium	7440-43-9	0.36	Eco SSL	0.36	0.00222	0.00222
Chromium	7440-47-3	26	Eco SSL	26	0.4	0.4
Copper	7440-50-8	28	Eco SSL	28	5.4	5.4
Lead	7439-92-1	11	Eco SSL	11	0.0537	0.0537
Zinc	7440-66-6	46	Eco SSL	46	6.62	6.62

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.

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 28 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	29.8
1,1,2,2-Tetrachloroethane	79-34-5	0.85	R5 ECOSED	0.85	1.36	0.127
1,1,2-Trichloroethane	79-00-5	0.518	R5 ECOSED	0.518	1.24	28.6
1,1-Dichloroethane	75-34-3	0.000575	R5 ECOSED	0.000575	NA	20.1
1,1-Dichloroethene	75-35-4	0.0194	R5 ECOSED	0.0194	0.031	8.28
1,2-Dichloroethane	107-06-2	0.26	R5 ECOSED	0.26	NA	21.2
Benzene	71-43-2	0.142	R5 ECOSED	0.142	NA	0.255
Chloroethane	75-00-3	--	None	NA	NA	NA
Chloromethane	74-87-3	--	None	NA	NA	10.4
cis-1,2-Dichloroethene	156-59-2	0.20894	R5 ECOSED	0.20894	NA	0.784
Ethylbenzene	100-41-4	0.175	R5 ECOSED	0.175	1.1	5.16
Methyl-tert-butyl ether	1634-04-4	--	None	NA	NA	NA
Tetrachloroethene	127-18-4	0.99	R5 ECOSED	0.99	0.468	9.92
Toluene	108-88-3	1.22	R5 ECOSED	1.22	NA	5.45
trans-1,2-Dichloroethene	156-60-5	0.654	R5 ECOSED	0.654	1.05	0.784
Trichloroethene	79-01-6	0.112	R5 ECOSED	0.112	0.0969	12.4
Vinyl chloride	75-01-4	0.202	R5 ECOSED	0.202	NA	0.646
Xylenes (total)	1330-20-7	27	R5 ECOSED	27	65	10
Polycyclic Aromatic Hydrocarbons						
2-Methylnaphthalene	91-57-6	0.0202	R5 ECOSED	0.0202	0.0202	3.24
Acenaphthene	83-32-9	0.00671	R5 ECOSED	0.00671	0.0067	682
Acenaphthylene	208-96-8	0.00587	R5 ECOSED	0.00587	0.0059	682
Anthracene	120-12-7	0.0572	R5 ECOSED	0.0572	0.0572	1480
Benzo(a)anthracene	56-55-3	0.108	R5 ECOSED	0.108	0.108	5.21
Benzo(a)pyrene	50-32-8	0.15	R5 ECOSED	0.15	0.15	1.52
Benzo(b)fluoranthene	205-99-2	10.4	R5 ECOSED	10.4	NA	59.8
Benzo(g,h,i)perylene	191-24-2	0.17	R5 ECOSED	0.17	0.17	119
Benzo(k)fluoranthene	207-08-9	0.24	R5 ECOSED	0.24	0.24	148
Chrysene	218-01-9	0.166	R5 ECOSED	0.166	0.166	4.73
Dibenzo(a,h)anthracene	53-70-3	0.033	R5 ECOSED	0.033	0.033	18.4
Fluoranthene	206-44-0	0.423	R5 ECOSED	0.423	0.423	122
Fluorene	86-73-7	0.0774	R5 ECOSED	0.0774	0.0774	122
Indeno(1,2,3-c,d)pyrene	193-39-5	0.2	R5 ECOSED	0.2	0.017	109
Naphthalene	91-20-3	0.176	R5 ECOSED	0.176	0.176	0.0994
Phenanthrene	85-01-8	0.204	R5 ECOSED	0.204	0.204	45.7
Pyrene	129-00-0	0.195	R5 ECOSED	0.195	0.195	78.5
Polychlorinated Biphenyls						
Aroclor-1016	12674-11-2	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1221	11104-28-2	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1232	11141-16-5	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1242	53469-21-9	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1248	12672-29-6	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1254	11097-69-1	0.0598	R5 ECOSED	0.0598	NA	0.000332
Aroclor-1260	11096-82-5	0.0598	R5 ECOSED	0.0598	NA	0.000332
Total PCBs	1336-36-3	0.0598	R5 ECOSED	0.0598	0.0598	0.000332
Metals						
Cadmium	7440-43-9	0.99	R5 ECOSED	0.99	0.99	0.00222
Chromium	7440-47-3	43.4	R5 ECOSED	43.4	43.4	0.4
Copper	7440-50-8	31.6	R5 ECOSED	31.6	31.6	5.4
Lead	7439-92-1	35.8	R5 ECOSED	35.8	35.8	0.0537
Zinc	7440-66-6	121	R5 ECOSED	121	121	6.62

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 28 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-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-Dichloroethane	107-06-2	910	R5 ECO SW	910	100	0.15	NA	
Benzene	71-43-2	114	R5 ECO SW	114	370	0.41	NA	
Chloroethane	75-00-3	2100	T-RSL	NA	NA	2100	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	
Ethylbenzene	100-41-4	14	R5 ECO SW	14	90	1.5	NA	
Methyl-tert-butyl ether	1634-04-4	11070	R3 BTAG SW	NA	11070	12	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	
Trichloroethene	79-01-6	47	R5 ECO SW	47	21	2	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	
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	
Aroclor-1221	11104-28-2	0.00012	R5 ECO SW	0.00012	0.000074	0.0068	0.014	
Aroclor-1232	11141-16-5	0.00012	R5 ECO SW	0.00012	0.000074	0.0068	0.014	
Aroclor-1242	53469-21-9	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	
Aroclor-1248	12672-29-6	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	
Aroclor-1254	11097-69-1	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	
Aroclor-1260	11096-82-5	0.00012	R5 ECO SW	0.00012	0.000074	0.034	0.014	
Total PCBs	1336-36-3	0.00012	R5 ECO SW	0.00012	0.000074	NA	0.014	
Metals								
Cadmium	7440-43-9	0.15	R5 ECO SW	0.15	0.25	1.8	2	
Chromium	7440-47-3	42	R5 ECO SW	42	85	0.043	16	
Copper	7440-50-8	1.58	R5 ECO SW	1.58	9	150	13	
Lead	7439-92-1	1.17	R5 ECO SW	1.17	2.5	15	65	
Zinc	7440-66-6	65.7	R5 ECO SW	65.7	120	1100	120	

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 0.1x 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.

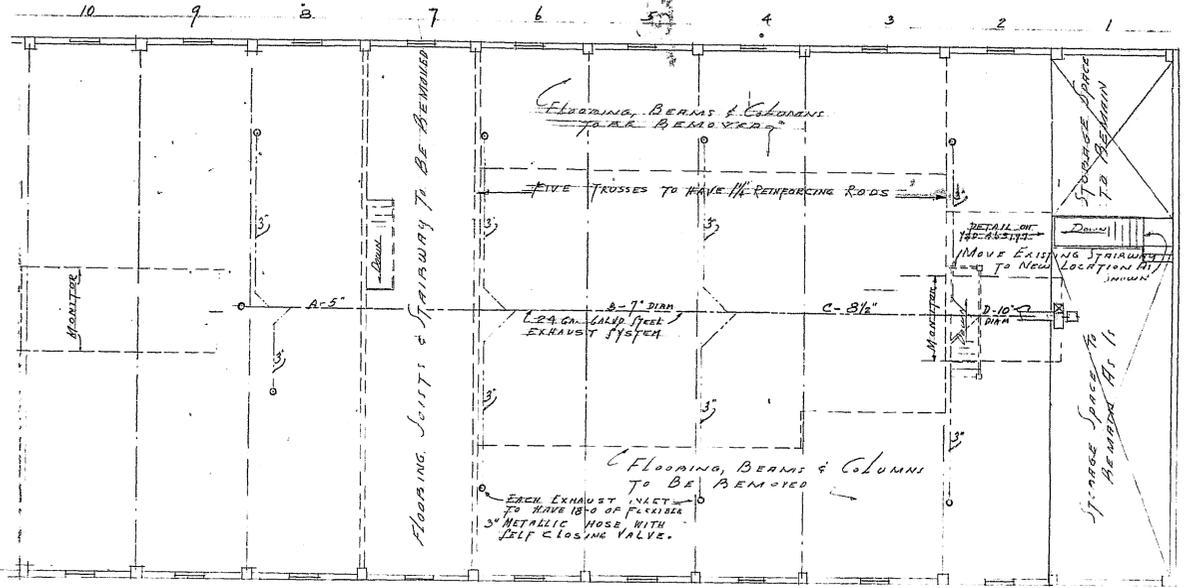
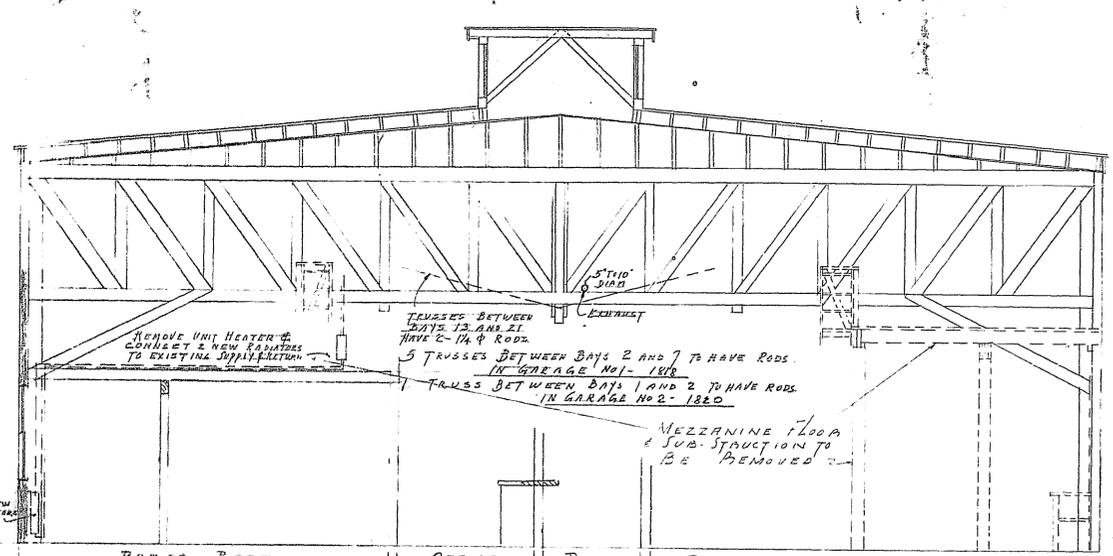
APPENDIX F

SWMU 28 HISTORICAL DRAWINGS AND PHOTOS

- Historical Drawings of Buildings 1818 and 1820
- Historical Photos of Building 1818

Historical Drawings of Buildings 1818 and 1820

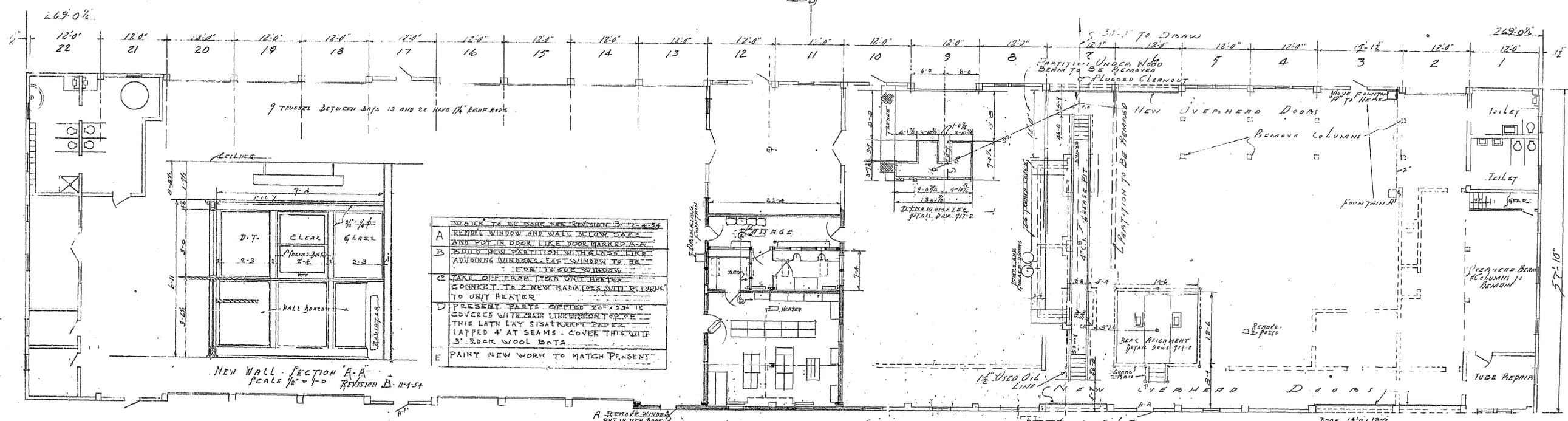
- PWO-917-1 - Modification of Garage (Building 1818)
- ORD-474-Sheet 4 - Plans for Garage (Building 1820)
- PWO-622 - Plot Plan
- ORD-474-Sheet 9 - Plumbing Arrangement
- PW-3820 - Industrial Waste Treatment Facilities
- PW-6530 - Eliminate Explicit Discharges
- PW-2723 - New Drains at Washrack
- PW-2777 - New Stone and Asphalt
- PWO-4048 - Plot Plans
- PWO-5873 - Underground Tanks



DOOR A 14'-0" x 7'-0" SIMILAR TO DOOR A-A SCALE 1/4" = 1'-0" REVISION B-11-4-54

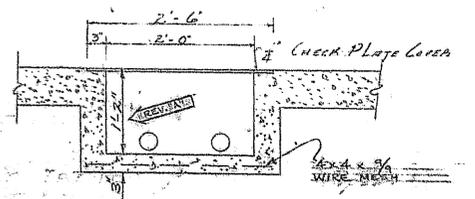
SECTION "B-B" SCALE 1/4" = 1'-0" PART 13 ROOF OFFICE PASSAGE RADIATOR ROOM REVISED SECTION FOR REVISION B-11-4-54

SECOND FLOOR PLAN EXISTING MEZZANINE FLOORS TO BE REMOVED EXCEPT AS SHOWN

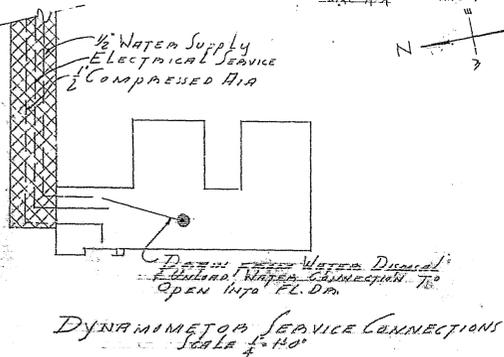


- A REMOVE WINDOW AND WALL BELOW SAME AND PUT IN DOOR LIKE DOOR MARKED A-A
- B BUILD NEW PARTITION WITH LATH LIKE EXISTING WINDOW. PUT WINDOW IN BRICK FOR 12'-0" x 7'-0"
- C TAKE OFF FROM THEM UNIT HEATER CONNECT TO 2 NEW RADIATOR WITH RETURNS TO UNIT HEATER
- D PRESENT PARTS OFFICE 24' x 22' IS COVERED WITH CHAIN LINK FENCE. THIS LATH LAY SINKRUM PAPER LAPPED 4" AT SEAMS - COVER THIS WITH 3" ROCK WOOL BATS
- E PAINT NEW WORK TO MATCH PRESENT

NEW WALL SECTION A-A SCALE 1/4" = 1'-0" REVISION B-11-4-54



TRENCH FOR GREASE PIPES SCALE 1" = 1'-0"

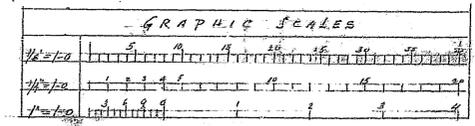


DYNAMOMETER SERVICE CONNECTIONS SCALE 1/2" = 1'-0"

FLOOR PLAN SCALE 1/8" = 1'-0"

LEGEND
 - STRUCTURE TO REMAIN AS IS
 - STRUCTURE TO BE REMOVED
 - STRUCTURE TO BE ADDED (NEW WORK)
 LEGEND DOES NOT APPLY TO PIPE WORK

OVERHEAD DOORS TO BE SIMILAR TO EXISTING OVERHEAD DOORS



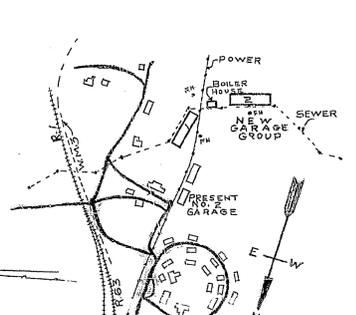
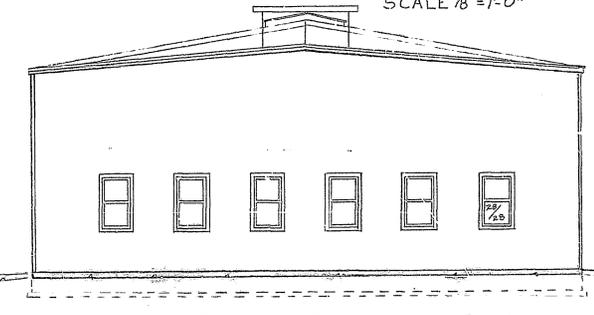
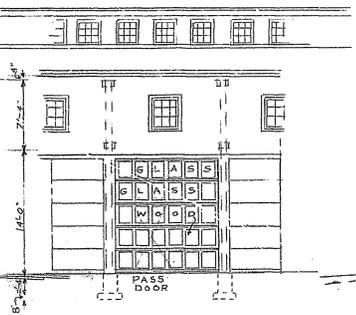
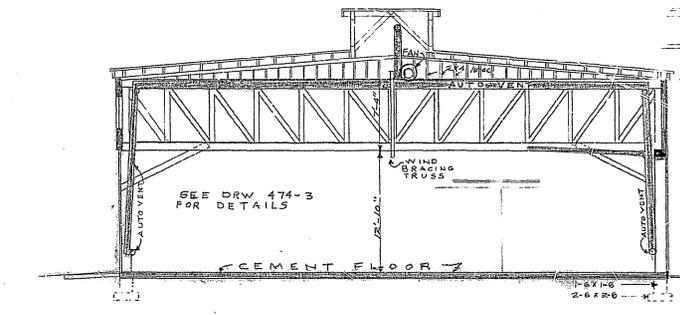
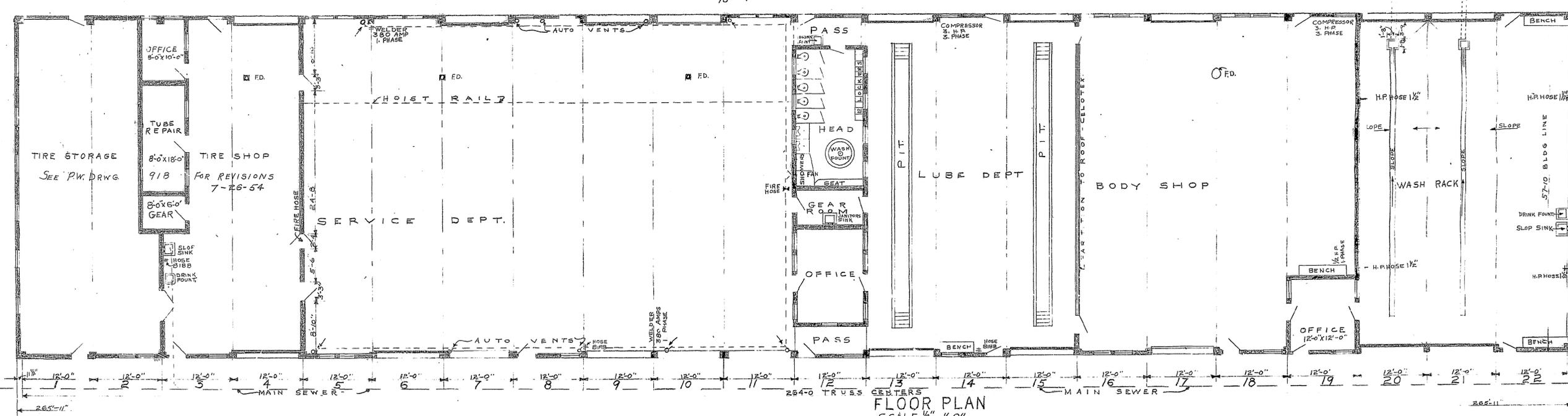
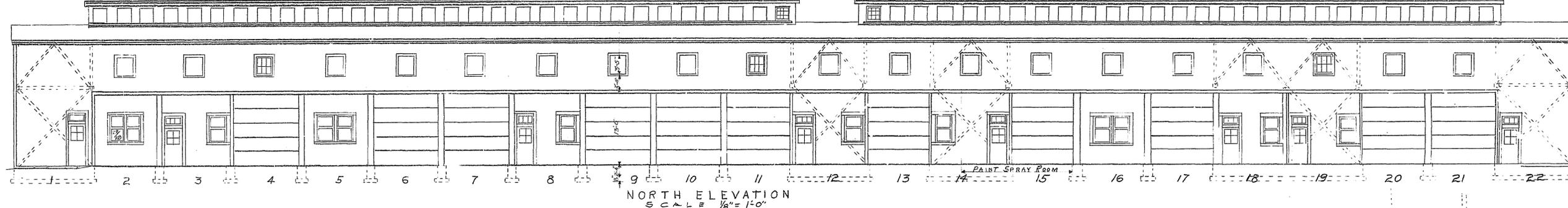
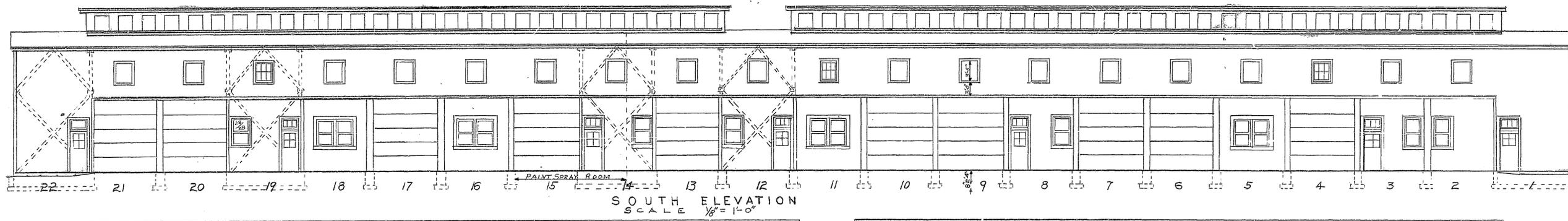
NOTE: USE ABOVE TRENCH DETAILS FOR DYNAMOMETER SERVICE CONNECTIONS

SATISFACTORY TO [Signature] DATE 11-9-54

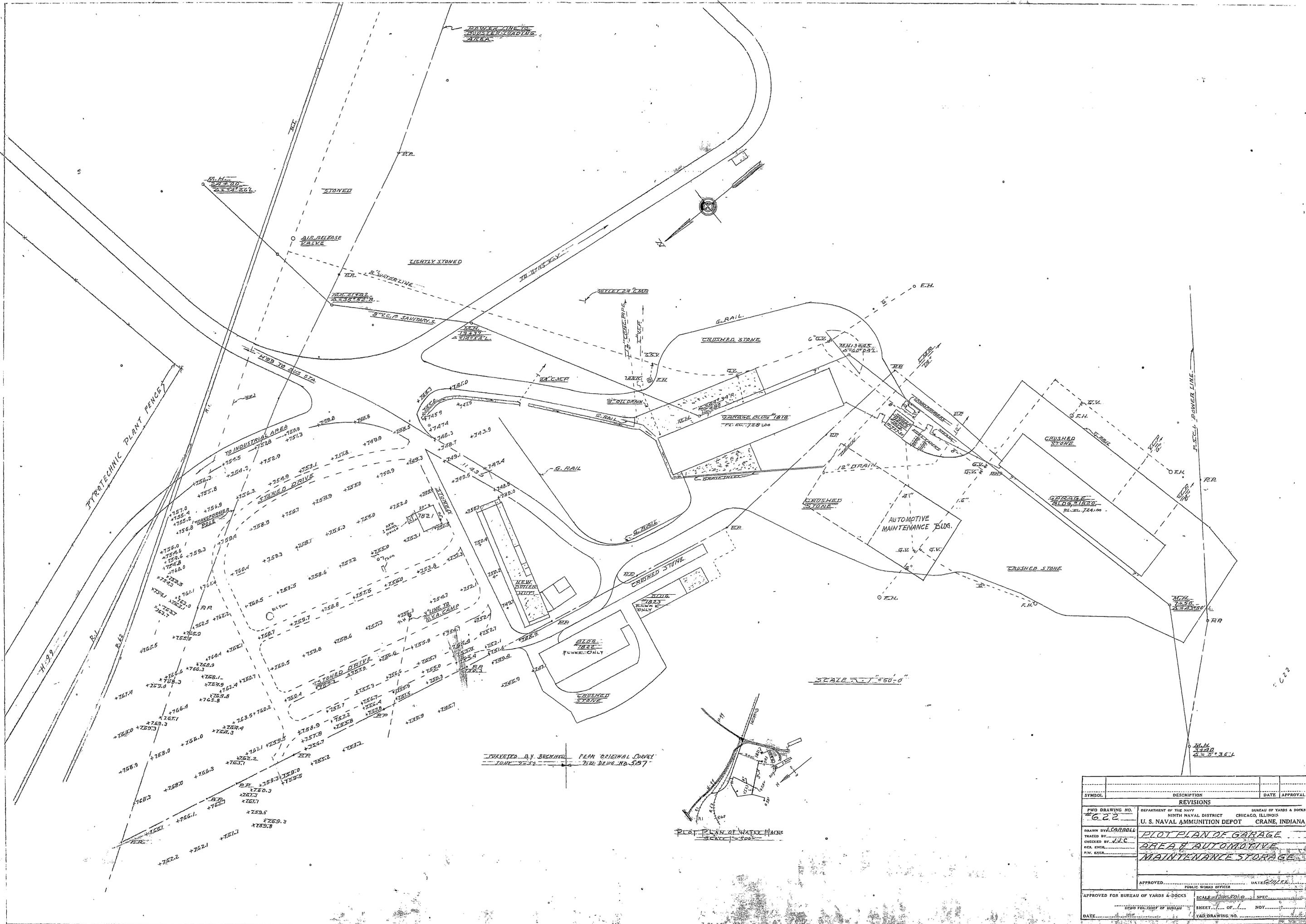
SATISFACTORY TO [Signature] DATE 8-11-54

NOTE: REVISION TO SPRINKLER SYSTEM AND NEW STAIRWAY ARE SHOWN ON 920-142197

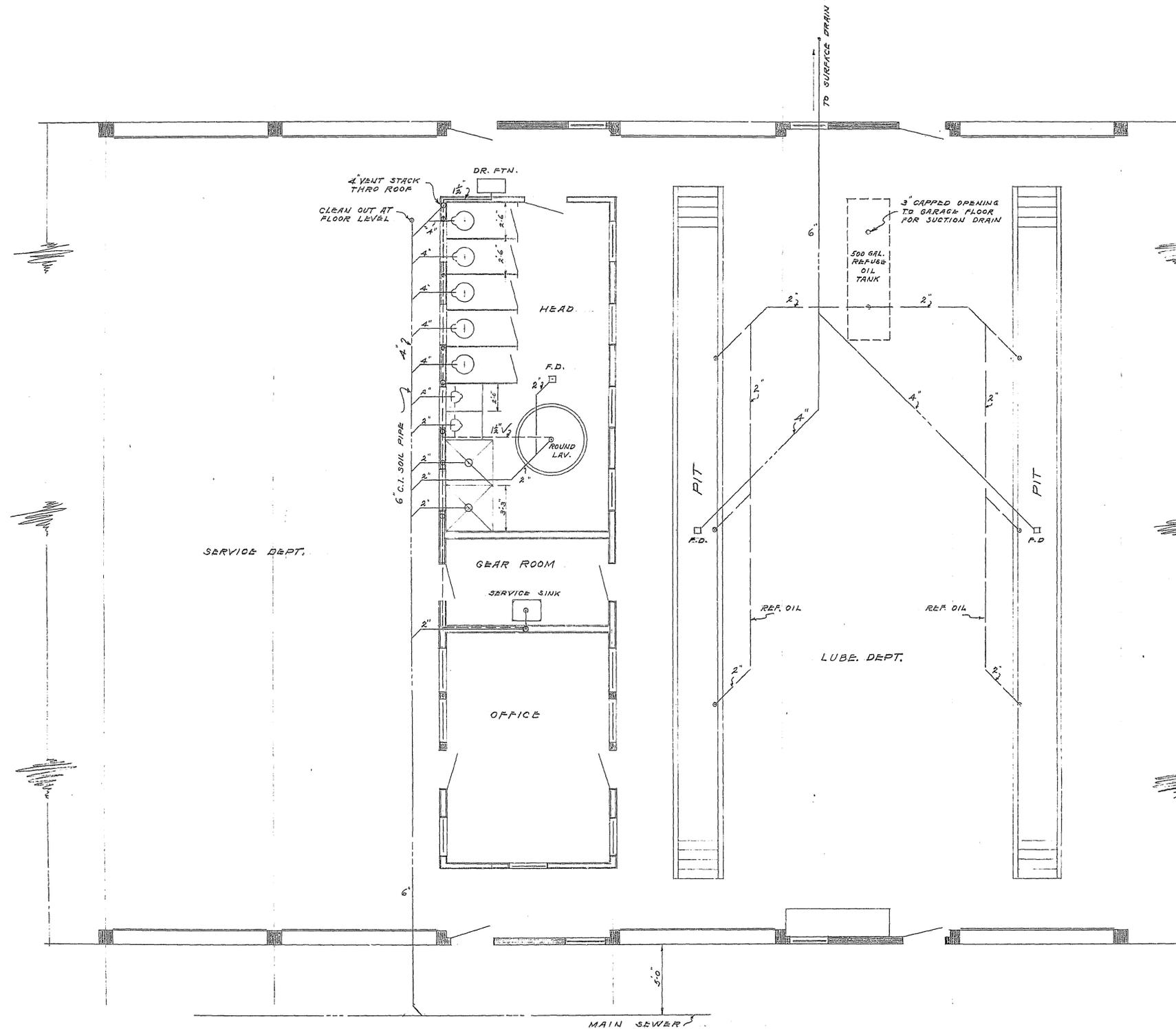
REVISIONS		BUREAU OF YARDS & DOCKS	
REV B	REMOVE WINDOW, MAKE DOOR, EDUCATION SECTION	JNC	11-4-54
REV A	REMOVE WINDOW, MAKE DOOR, EDUCATION SECTION	JNC	11-4-54
REV C	TRENCH DEPTH 8" TO 12" (SEE DETAIL LOWER LEFT)	S.P.C.	
DESCRIPTION		BY	DATE
PWO DRAWING NO. 917-1		DEPARTMENT OF THE NAVY NINTH NAVAL DISTRICT GREAT LAKES, ILLINOIS U.S. NAVAL AMMUNITION DEPOT CRANE, INDIANA	
DESIGN		MODIFICATION OF GARAGE No. 1 BLDG. NO. 1818	
DRAWN		PLAN, SECTIONS & DETAILS	
CHECKED		APPROVED	
SUPERVISED		DATE 8-18-54	
JOB NO.		DIRECTOR	
BRANCH MGR.		APPROVED FOR BUREAU OF YARDS & DOCKS	
P. P. ENGR.		DPO FOR CHIEF OF BUREAU	
DATE		SHEET 1 OF 2	
DATE		SCALE AS SHOWN	
DATE		NO.	
DATE		SCALE	



SYMBOL	DESCRIPTION	BY	DATE	APPROVAL
REVISIONS				
TWO DRAWING NO. ORD. 474 6-21-45		DEPARTMENT OF THE NAVY NINTH NAVAL DISTRICT U. S. NAVAL AMMUNITION DEPOT CRANE, INDIANA		BUREAU OF YARDS & DOCKS GREAT LAKES, ILLINOIS CRANE, INDIANA
DESIGN	PLANS FOR GARAGE BLDG. NO 2 S.W. QUARTER SECTION E. 7			
DRAWN				
CHECKED				
BY				
DFWO DESIGN DIV. JOB NO. CHECKED BRANCH MGR. F. P. ENGR.	REV. 7-26-45 BODY SHOP ENLARGED, LANTERN ADDED REV. 8-4-45 DOORS CHANGED, OFFICE MOVED, EXHAUST #2 ELEC. ADDED REV. 8-5-45 BOILER ROOM ADDED, WASH RACK DRAIN CHANGED REV. 7-22-54 CORRECTED TO DATE COPY OF ORIGINAL DRAWING 8-26-59 PEK	APPROVED _____ DATE _____ PUBLIC WORKS OFFICER		
APPROVED FOR BUREAU OF YARDS & DOCKS DFWO FOR CHIEF OF BUREAU		SCALE $1/8" = 1'-0"$ SHEET 4 OF _____ V & D DRAWING NO. _____	SPEC. _____ NBY _____	



SYMBOL	DESCRIPTION	DATE	APPROVAL
REVISIONS			
P.W.O. DRAWING NO. 622	DEPARTMENT OF THE NAVY NINTH NAVAL DISTRICT U. S. NAVAL AMMUNITION DEPOT	BUREAU OF YARDS & DOCKS CHICAGO, ILLINOIS CRANE, INDIANA	
DRAWN BY: CORROLL	PLAT PLAN OF GARAGE		
TRACED BY: J.H.C.	AREA & AUTOMOTIVE		
DES. ENGR.:	MAINTENANCE STORAGE		
P.W. ENGR.:			
APPROVED: _____		PUBLIC WORKS OFFICER	DATE: _____
APPROVED FOR BUREAU OF YARDS & DOCKS		SCALE: 1" = 50'-0"	SPEC: _____
DATE: _____		SHEET: _____ OF _____	NOY: _____
		Y&D DRAWING NO.	



PLAN
SCALE 1/4" = 1 FT.

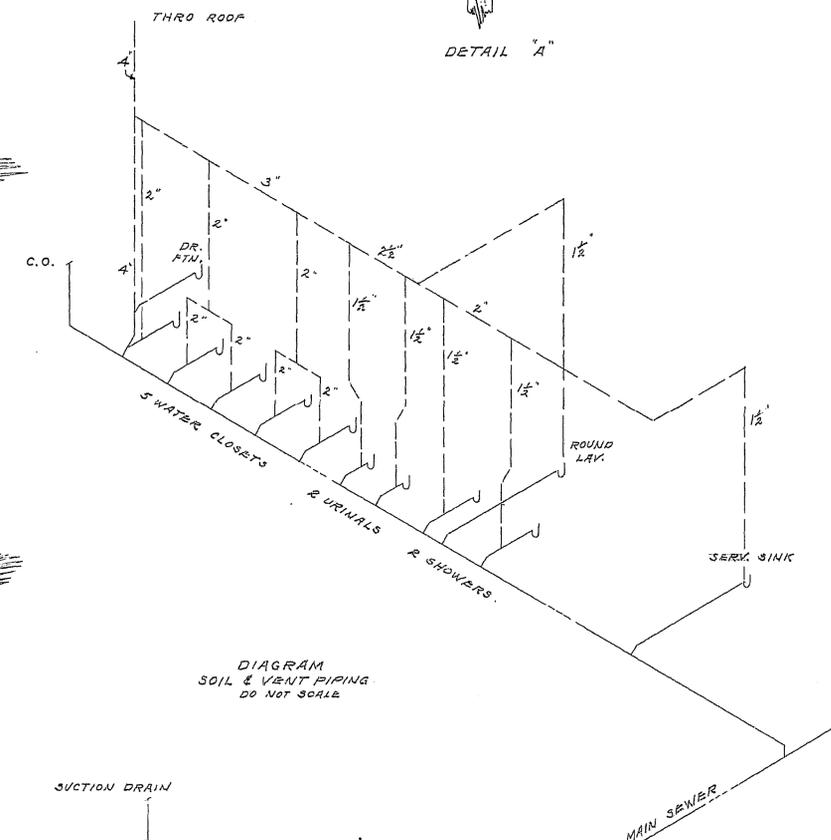
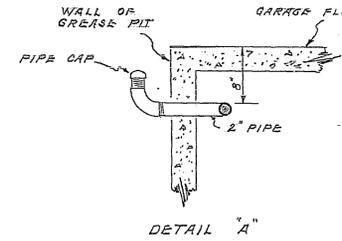


DIAGRAM
SOIL & VENT PIPING
DO NOT SCALE

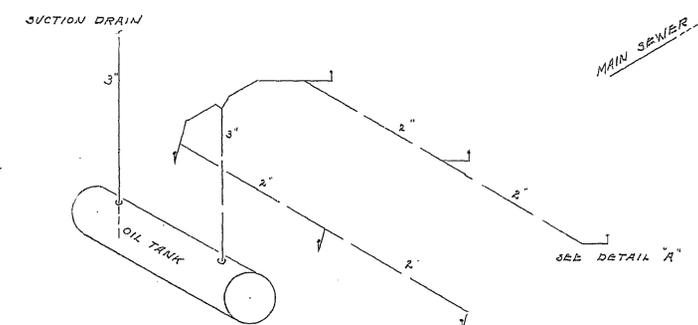
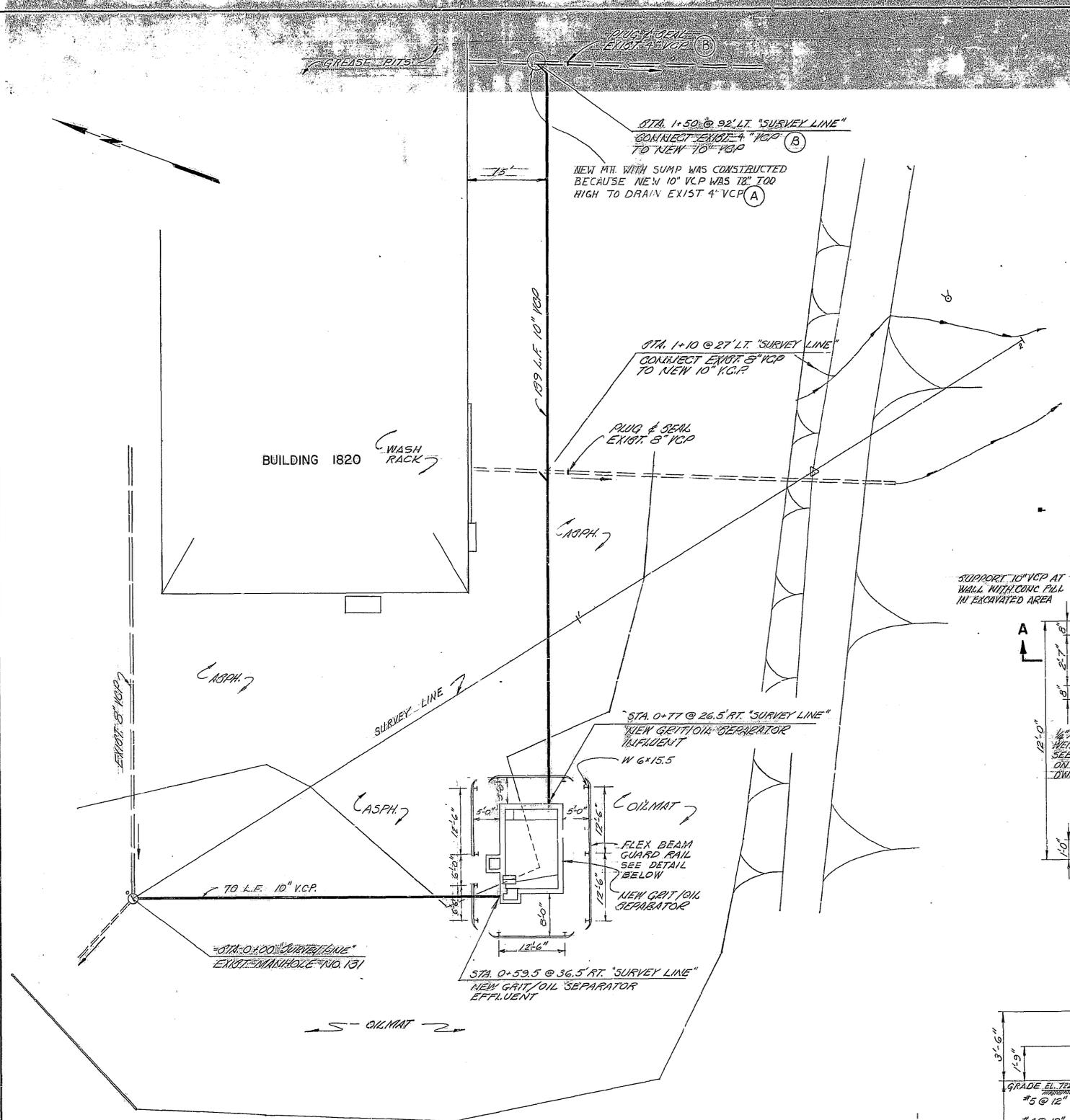


DIAGRAM
REFUSE OIL PIPING
DO NOT SCALE

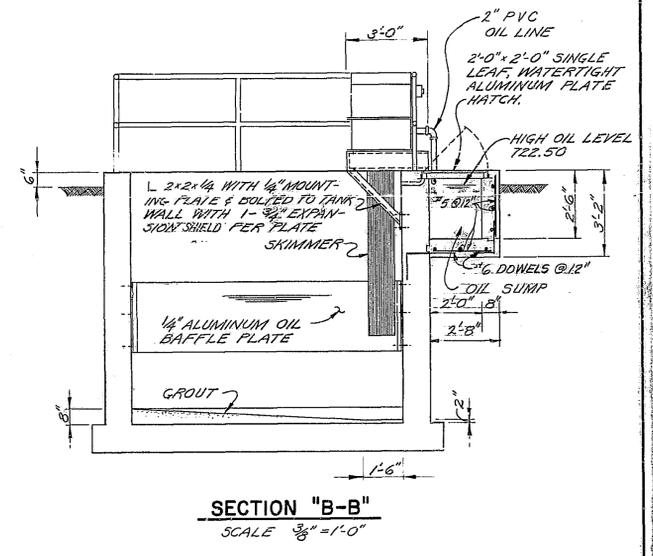
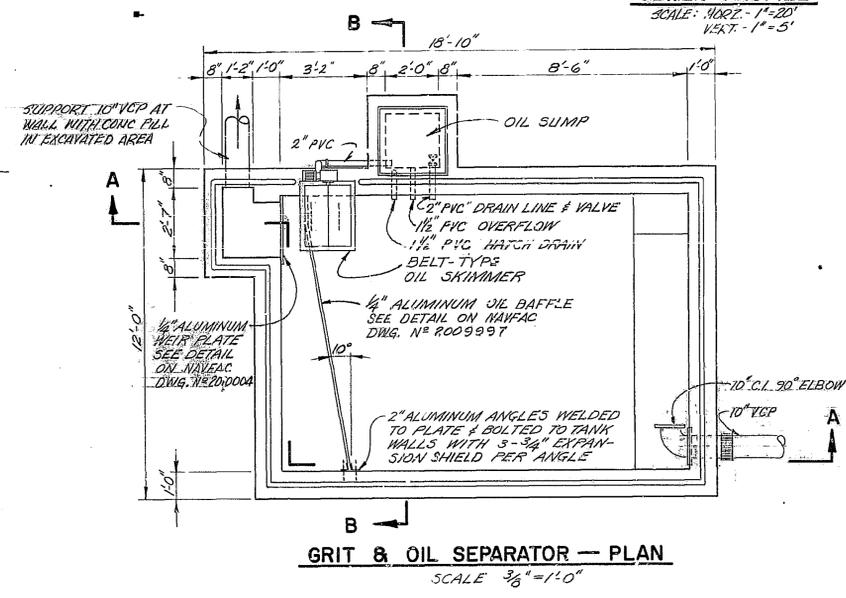
PLUMBING ARRANGEMENT GARAGE BUILDING NO. 2 N.A.D. CRANE, IND.		
PROJECT NO.	U. S. NAVAL AMMUNITION DEPOT CRANE, IND.	APPROVED BY
DATE		U.S. NAVAL OFFICER
DESIGNED BY		FIRST LT. <i>Ba</i>
CHECKED BY		CALC. OFFICER
PLANNED BY		WORK. OFFICER
DATE	ENGINEERING DEPT. OFFICE OF FIRST LIEUTENANT	DRAWING NO. <i>00274</i>
DATE	SCALE AS SHOWN	SHEET 3 OF 9A

REVISIONS				
LTR	DESCRIPTION	PREP'D BY	DATE	APPROVED
1	REVISED AS BUILT - NEW MANHOLE (CONSTRUCTED @ STA 1+50)	W. J. STEEG	11/1/82	RES
2	EXISTING 4" V.C.P. SHOWN FROM GREASE PIT	W. J. STEEG	11/1/82	RES
3	CHANGED TO EXISTING 4" V.C.P.	W. J. STEEG	11/1/82	RES

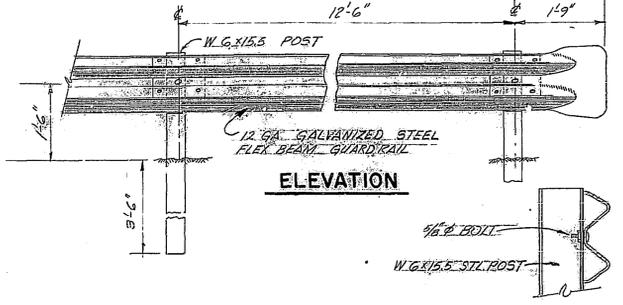


Station	Existing	New	Notes
725	70 L.F. 10" V.C.P. @ 0.28%	159 L.F. 10" V.C.P. @ 0.28%	
720	EXISTING GRADE		
715	718.00 EXIST. 5" V.C.P. INV 717.91	718.25	
710	STA. 0+10 SURVEY LINE CONNECT EXIST. 4" V.C.P. TO NEW 10" V.C.P.		
705	STA. 0+10 SURVEY LINE CONNECT EXIST. 4" V.C.P. TO NEW 10" V.C.P.		

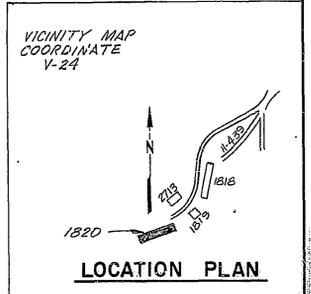
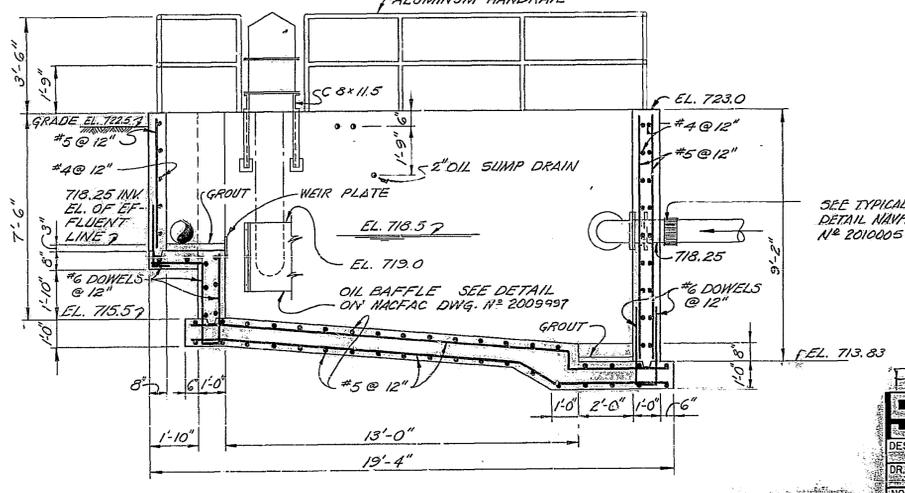
SEWER PROFILE
SCALE: HORIZ. 1"=20'
VERT. 1"=5'



SITE PLAN
SCALE 1"=10'



SECTION THRU GUARD RAIL
NO SCALE



P.W. 3820

HENRY B. STEEG & ASSOCIATES, INC. ENGINEERS, INDIANAPOLIS, INDIANA

DEPARTMENT OF THE NAVY NAVAL FACILITIES ENGINEERING COMMAND NORTHERN DIVISION PHILADELPHIA, PA.

NAVAL AMMUNITION DEPOT CRANE, INDIANA

INDUSTRIAL WASTE TREATMENT FACILITIES

BUILDING 1820 GRIT & OIL SEPARATOR

18" SEWER CONNECTION

APPROVED: [Signature] DATE: 11/1/82

OFFICER IN CHARGE

SIZE: CODE IDENT NO. 80091 NAVFAC DRAWING NO. 2009998

SATISFACTORY TO: DATE: 11/1/82

APPROVED: [Signature] DATE: 11/1/82

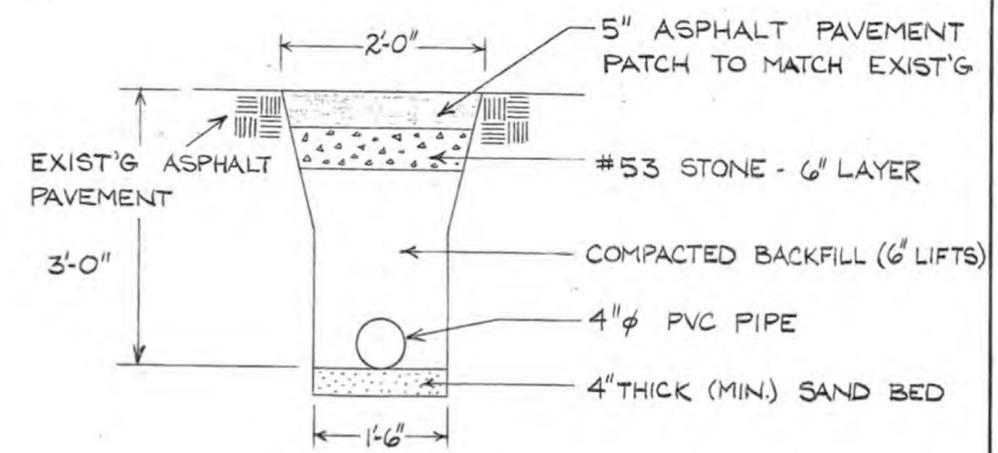
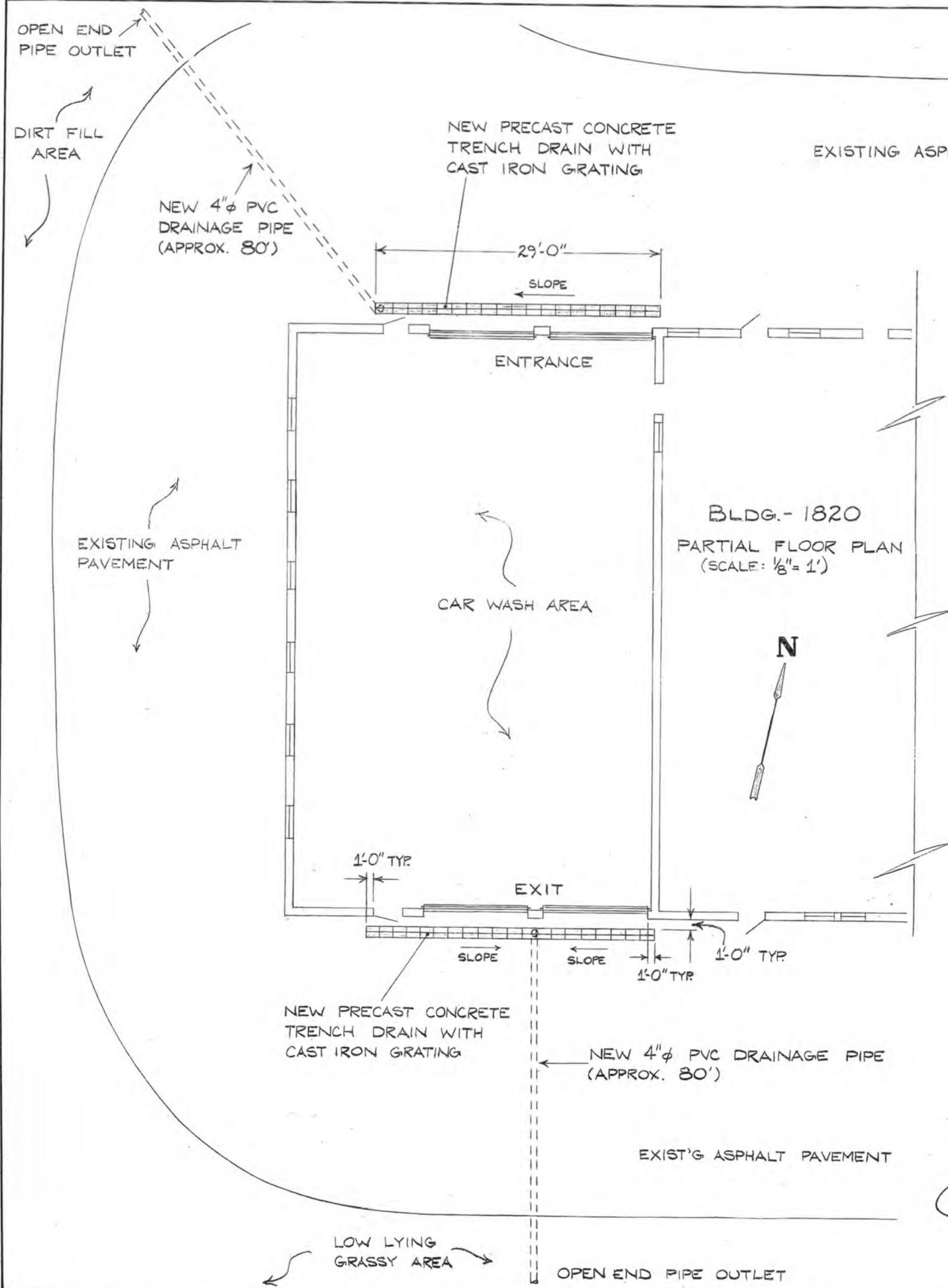
INORDV FOR COMMANDER/NAIFAC

CONSTR. CONTR. NO. N62472573-C-0093

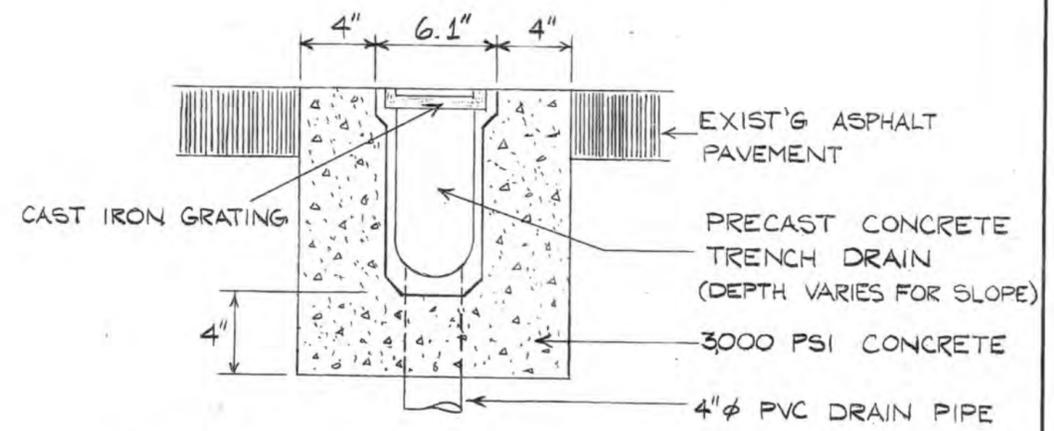
SCALE: AS SHOWN SPEC: 04-73-0093 SHEET 16 OF 29

TOP FLOOR OF GARAGE BLDG. 1820. ELEV. 724.00

ACO DRAIN INC.
 EXECUTIVE COMMONS EAST
 SUITE-214
 29525 CHAGRIN BLVD.
 CLEVELAND, OHIO 44122
 216/464-5603



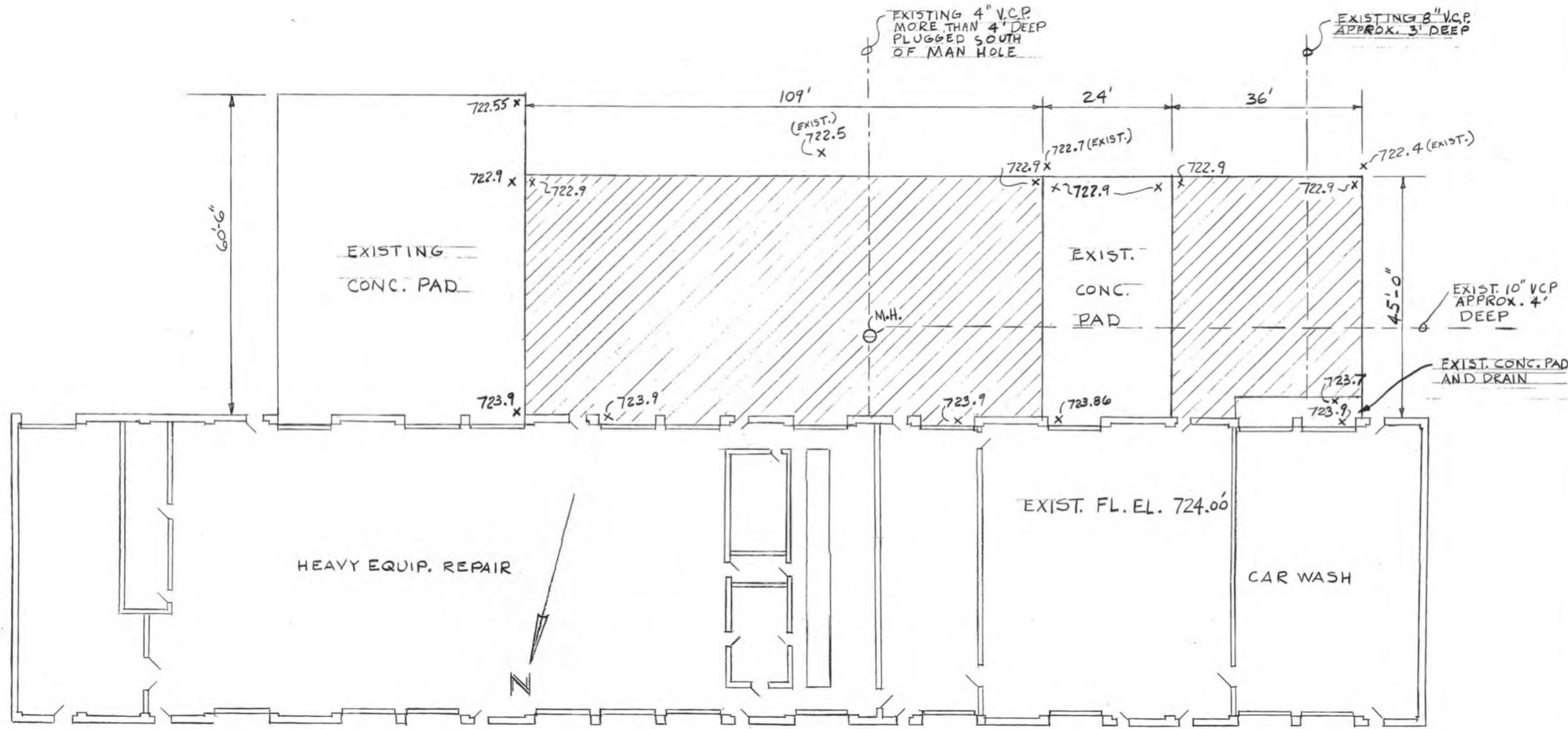
DRAIN PIPE SECTION THRU PAVEMENT (TYP.)
 NO SCALE



DRAINAGE TRENCH SECTION (TYP.)
 NO SCALE

NOTE:
 NEW PRECAST CONCRETE TRENCH DRAIN SHALL BE EQUAL TO OR GREATER IN QUALITY AND PERFORMANCE TO "ACO CHANNEL SLOPE" DRAIN, SERIES NW 100. THE SLOPE OF TRENCH DRAIN SHALL NOT BE LESS THAN 0.6 PERCENT.

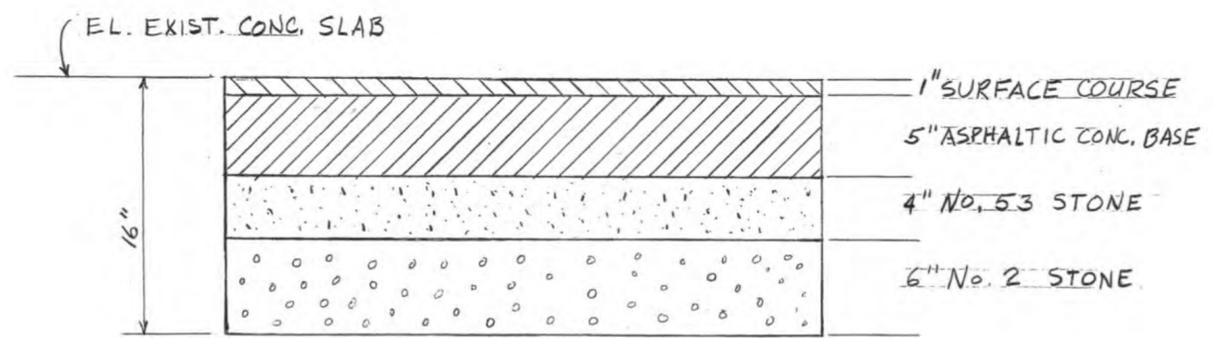
SATISFACTORY TO <i>09B</i> <i>James A. Hunt</i> DATE <i>2-8-85</i>	N. W. S. C., CRANE, INDIANA NEW DRAINS AT WASHRACK BLDG.-1820 P. W. SKETCH NO. 2723 APPROVED <i>[Signature]</i> DATE <i>2/11/85</i> PUBLIC WORKS OFFICER
SATISFACTORY TO <i>096</i> <i>James P. Conley</i> DATE <i>2/8/85</i>	
SATISFACTORY TO DATE _____	



FLOOR PLAN OF BLDG. 1820
SCALE 1/16" = 1'-0"

NOTES

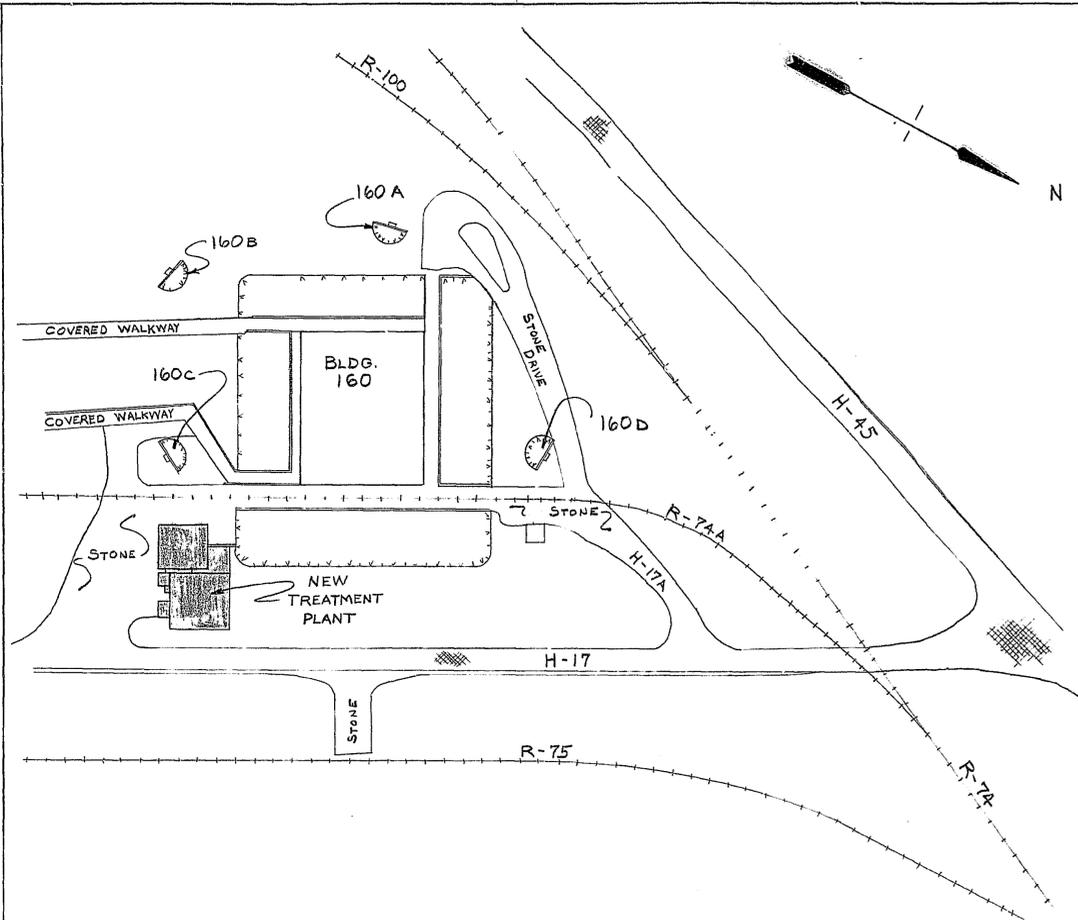
1. GOV'T SHALL EXCAVATE FOR STONE AND ASPHALT CONCRETE.
2. CONTRACTOR SHALL PLACE AND COMPACT STONE AND ASPHALT CONCRETE.



TYPICAL CROSS-SECTION
NO SCALE

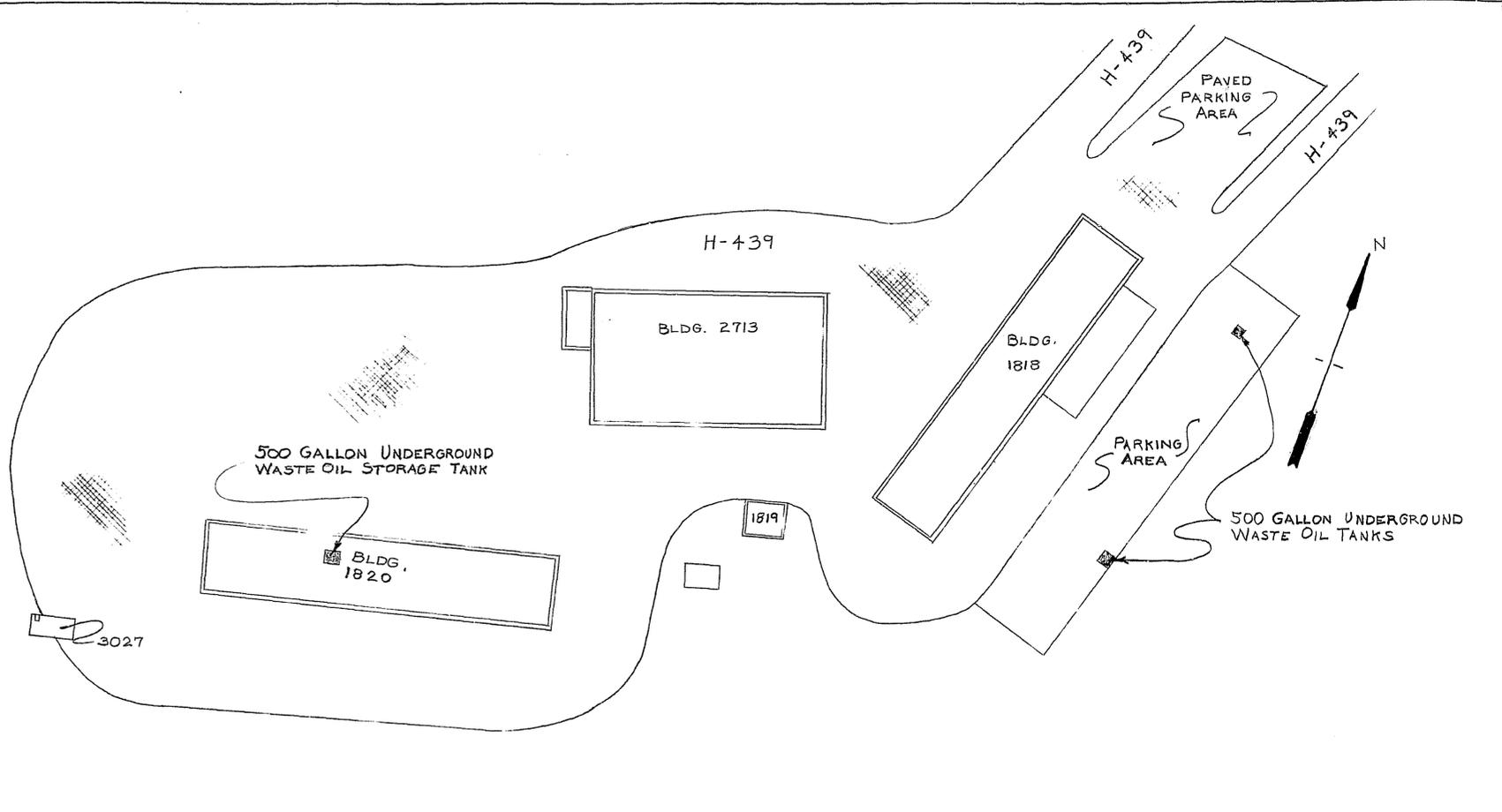
SATISFACTORY TO 096 James S. Conley DATE 8-1-86		N. W. S. C., CRANE, INDIANA	
SATISFACTORY TO 104 R. L. Voth DATE 7-30-86		DRAWN L.H.W.	NEW STONE AND ASPHALT BLDG. 1820 P. W. SKETCH NO. 2777
SATISFACTORY TO DATE _____		DESIGN ENG L.H.W.	
		P. W. ENG JMM	
		APPROVED	DATE 7/8/86
		PUBLIC WORKS OFFICER	

WR: 096-022-4



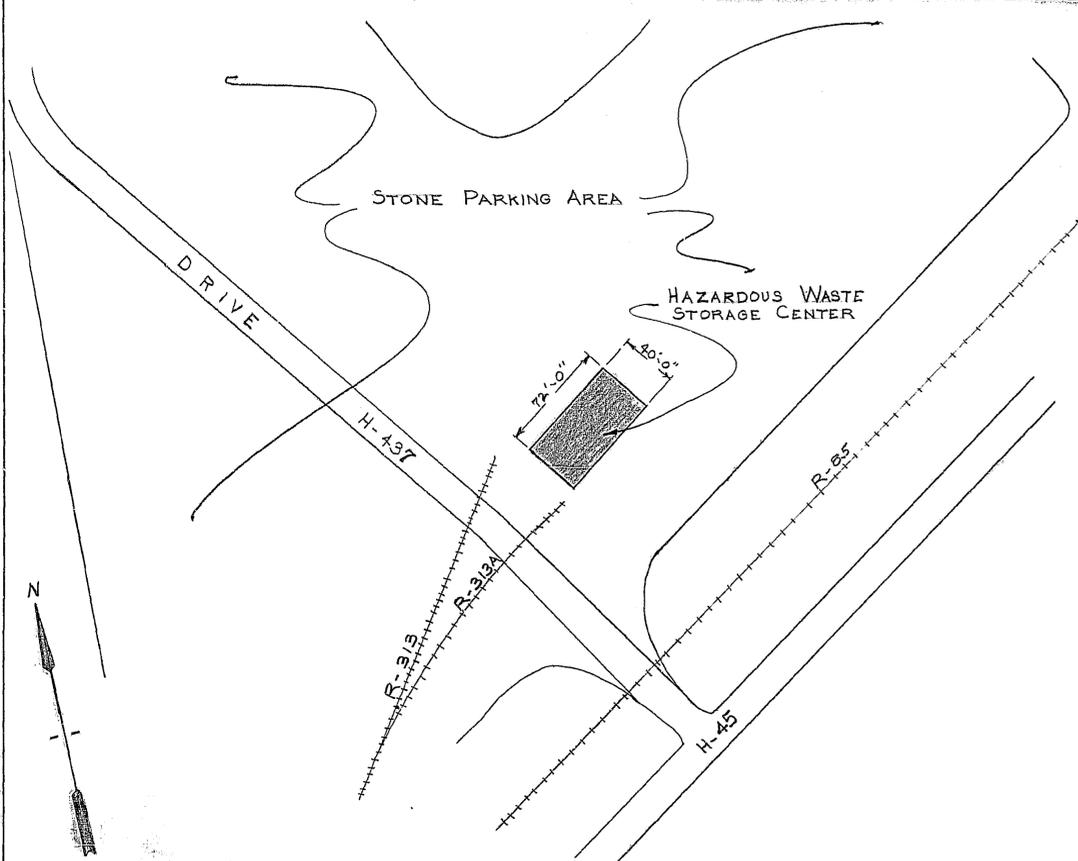
PLOT PLAN - BLDG. 160
SCALE 1" = 50'-0"

7



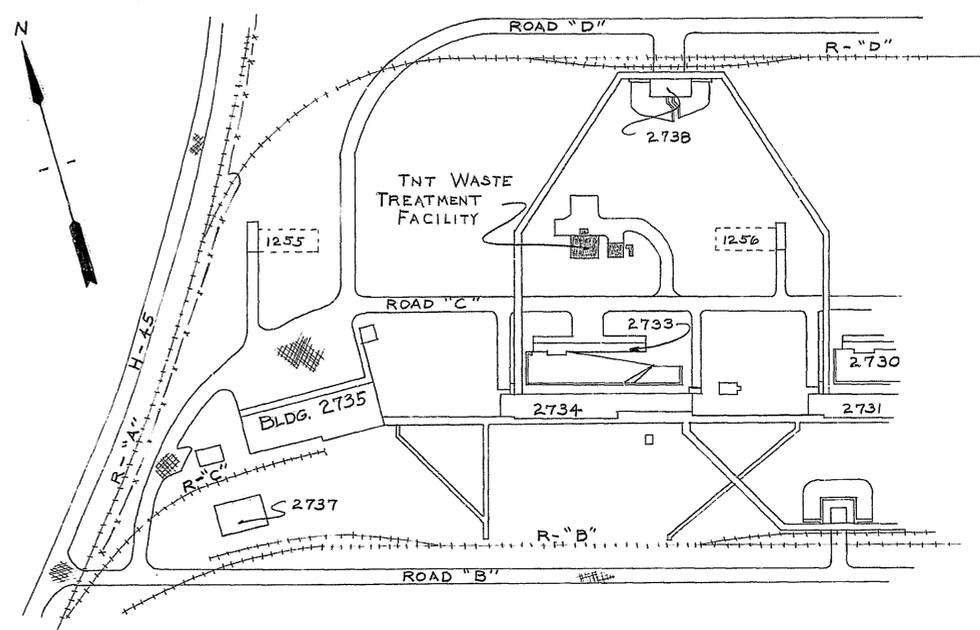
PLOT PLAN - BLDGS. 1818, 1820
SCALE 1" = 50'-0"

8



PLOT PLAN - BLDG. 2993
SCALE 1" = 50'-0"

9

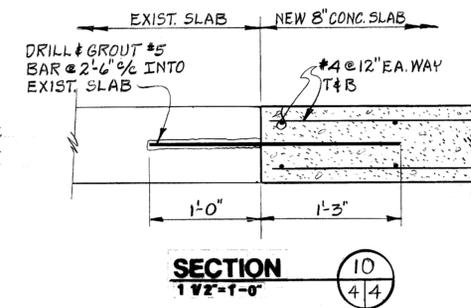
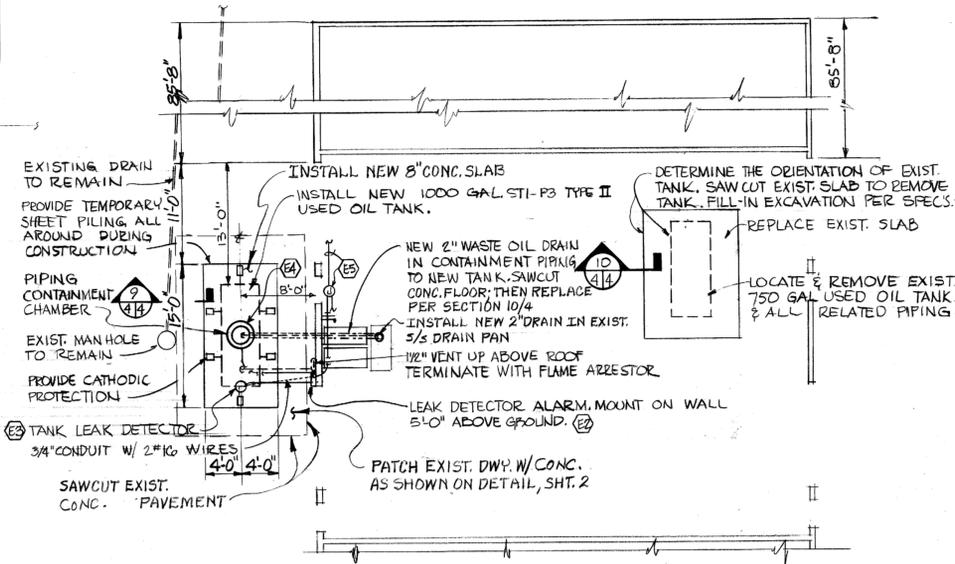
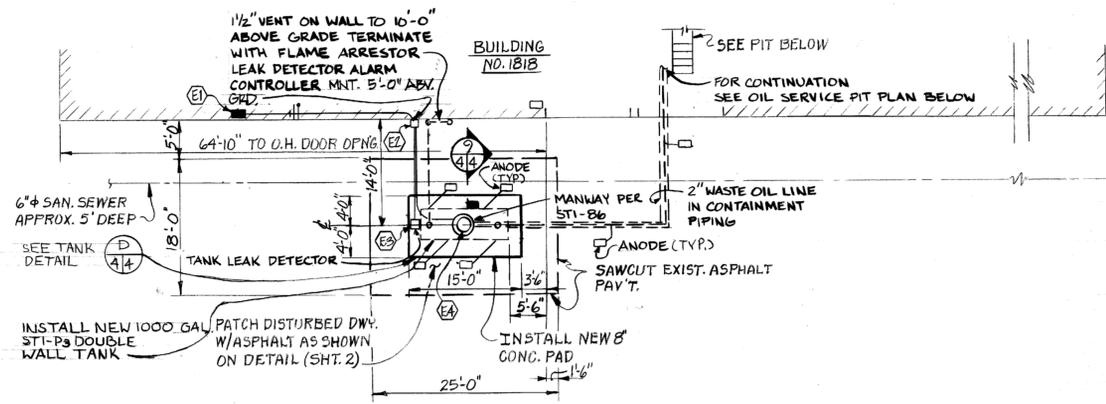


PLOT PLAN - BLDG. 3044
SCALE 1" = 100'-0"

10

SATISFACTORY TO	PWO DWG REF	DEPARTMENT OF THE NAVY
DATE	PWO DRAWING NO.	NAVAL WEAPONS SUPPORT CENTER
SATISFACTORY TO	4048	CRANE, INDIANA
DATE	DESIGN	PLOT PLANS
SATISFACTORY TO	DRAWN	BLDGs:
DATE	CHECKED	160, 1818, 1820, 2993, 3044
SATISFACTORY TO	APPROVED	SCALE AS NOTED
DATE		SHEET 3 OF 5

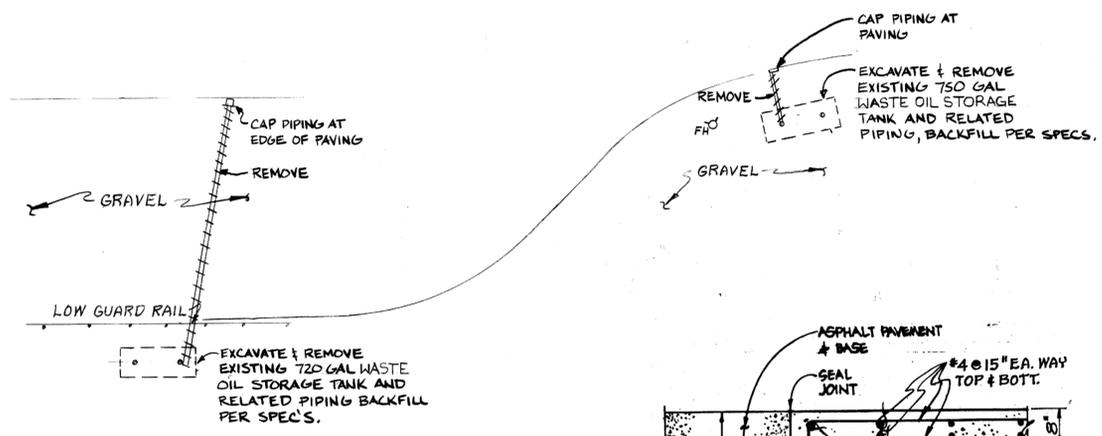
REVISIONS			
LTR	DESCRIPTION	PREP'D BY	DATE
A	AS BUILT	D.O.	11-93



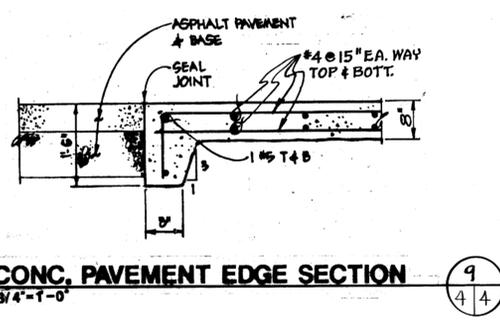
BLDG. 1820
SITE PLAN
1-1-0

ELECTRICAL DRAWING NOTES:

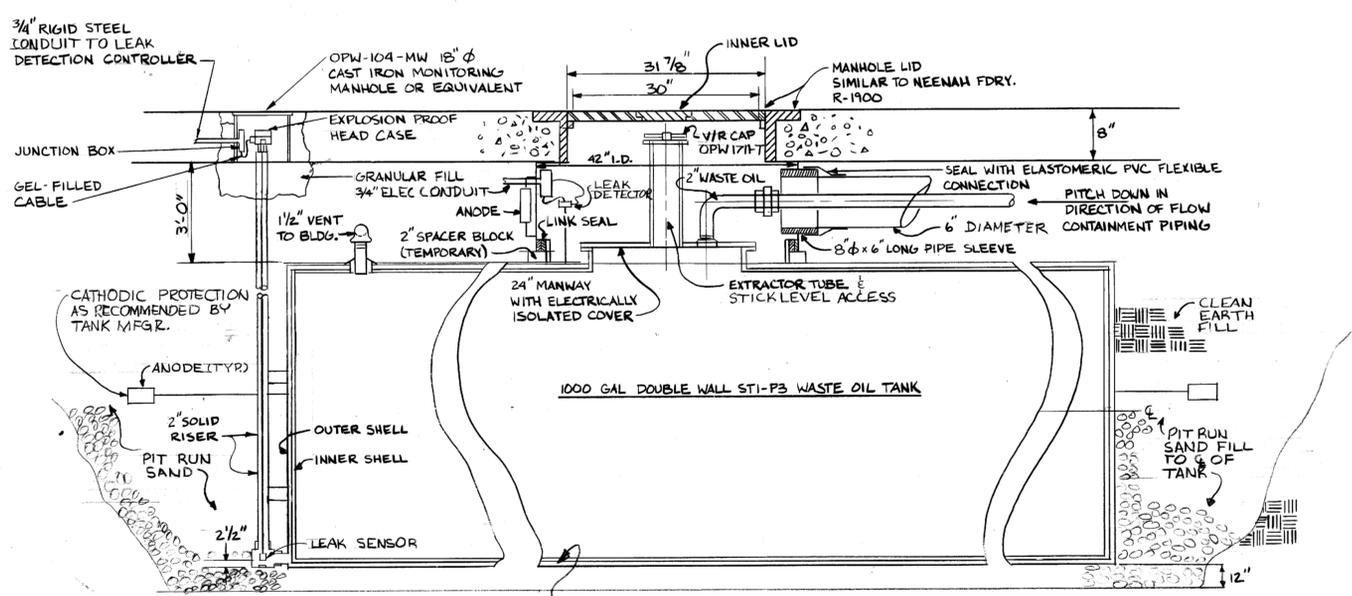
- E1 EXISTING MACHINE SHOP EQUIPMENT ELECTRICAL PANEL. CIRCUIT NUMBER 8 IS A 20-AMPERE 1-POLE SPARE BREAKER. PROVIDE A CIRCUIT BREAKER LOCK-ON DEVICE. THIS CIRCUIT SHALL FEED THE LEAK DETECTOR PANEL. PROVIDE A 3/4 INCH RIGID CONDUIT WITH 2-#12 CONDUCTORS AND 1-#12 GROUND WIRE FROM THE CIRCUIT BREAKER PANEL TO THE LEAK DETECTOR PANEL. PROVIDE AN EXPLOSION-PROOF CONDUIT SEAL ON THE CONDUIT.
- E2 NEW LEAK DETECTOR CONTROL PANEL. PROVIDE A 3/4 INCH RIGID CONDUIT WITH 2-#16 CONDUCTORS AND 1-#12 GROUND WIRE FROM THE DETECTOR CONTROL PANEL ROUTED UNDERGROUND TO EACH LEAK DETECTOR. PROVIDE AN EXPLOSION-PROOF CONDUIT SEAL ON EACH CONDUIT.
- E3 TANK LEAK DETECTOR SENSOR ASSEMBLY. SEE DETAIL D ON THIS SHEET FOR INFORMATION.
- E4 TANK OVERFLOW LEAK DETECTOR SENSOR. SEE DETAIL D ON THIS SHEET FOR INFORMATION.
- E5 EXISTING EXIT LIGHTING CONDUIT, JUNCTION BOX, AND CIRCUIT WIRING. EXTEND A 3/4 INCH RIGID CONDUIT WITH 2-#12 CONDUCTORS AND 1-#12 GROUND WIRE FROM THE EXISTING EXIT LIGHT CIRCUIT TO THE LEAK DETECTOR PANEL. PROVIDE AN EXPLOSION-PROOF CONDUIT SEAL ON THE CONDUIT.



BLDG. 1818
SITE PLAN
1-1-0

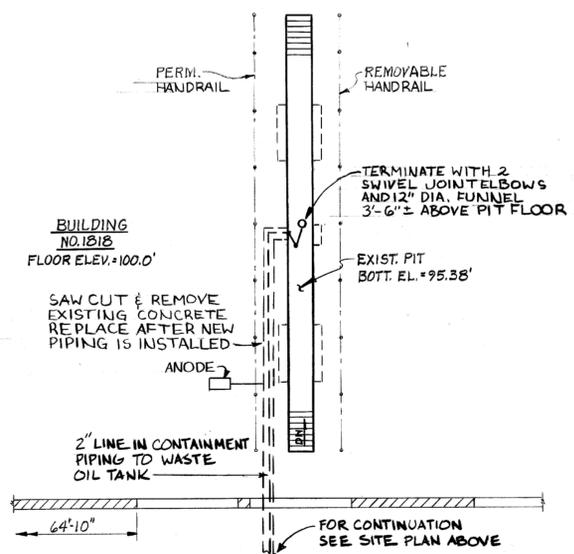


CONC. PAVEMENT EDGE SECTION
3/4-1-0

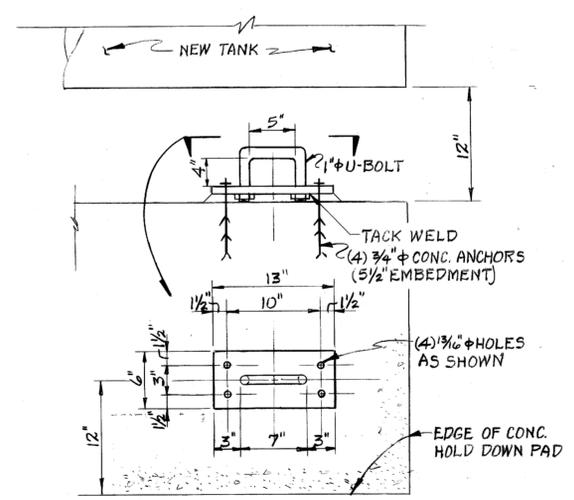


GENERAL NOTE:
MANUFACTURERS OF COMMERCIAL PRODUCTS ARE SHOWN AS A GUIDE TO THE QUALITY AND STYLE REQUIRED FOR THIS PROJECT. OTHER COMMERCIAL PRODUCTS EQUIVALENT TO THE ITEMS SPECIFIED WILL BE ACCEPTABLE, SUBJECT TO THE APPROVAL OF THE OFFICER-IN-CHARGE.
A PROVIDE A MINIMUM 12" OF SAND BEDDING BETWEEN CONC. PAD AND TANK.

DETAIL
D



PLAN OIL SERVICE PIT
1/8-1-0



TANK HOLD DOWN ANCHOR DETAIL
1/2-1-0

PEAS <small>REGISTERED PROFESSIONAL ENGINEER ARCHITECTS</small> <small>CINCINNATI, OHIO</small> PROJECT NO. 0492-12	SATISFACTORY TO <i>10/21/91</i> DATE <i>09/20/91</i>	PWO DWG REF PWO DRAWING NO. 5873	DEPARTMENT OF THE NAVY NAVAL FACILITIES ENGINEERING COMMAND NAVAL WEAPONS SUPPORT CENTER CRANE, INDIANA
	SATISFACTORY TO <i>04/21/91</i> DATE <i>10/21/91</i>	DESIGN <i>M. LIPPERT/T. HUMBERT</i> DRAWN <i>W. H. HARRIS</i> CHECKED <i>JAC</i> SUPV. <i>W. H. HARRIS</i>	UNDERGROUND TANKS SITE PLAN & DETAILS BUILDINGS 1818 & 1820
SATISFACTORY TO <i>09/21/91</i> DATE <i>10/21/91</i>	APPROVED PUBLIC WORKS OFFICER	SIZE F 80091	SCALE NOTED SPEC. NO. 04-90-7040 SHEET 4 OF 5

Historical Photos of Buildings 1818

Looking west at UST east of Building 1818



Vent and fill line at UST east of Building 1818



PHOTOGRAPH BY ALPHONSE GRYL

B-1218

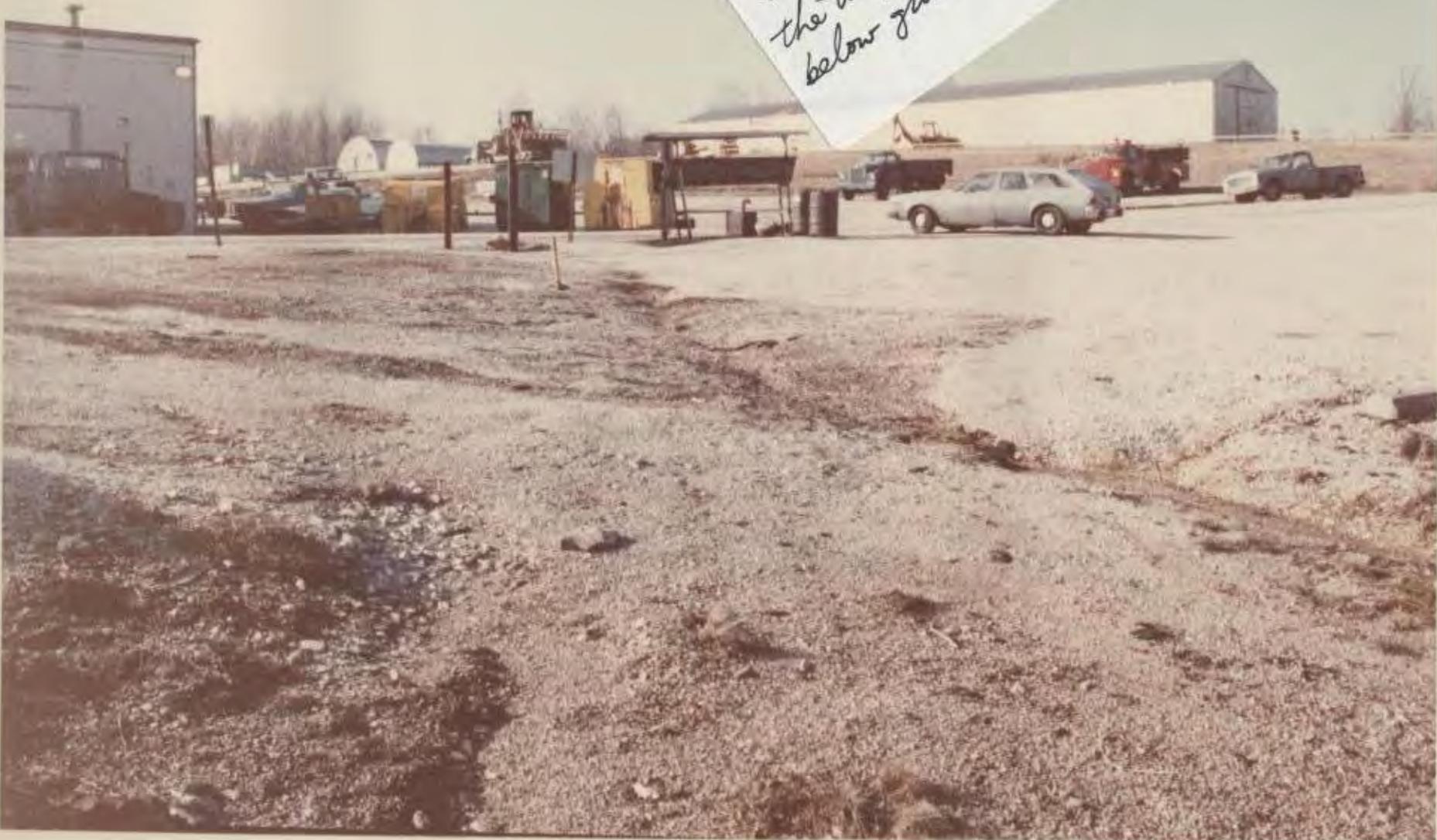
Waste oil tank buried here



Looking east and downhill from UST east of Building 1818



Waste motor oil is dumped into
the trough which drains to a
below ground tank.



Looking west at UST east of Building 1818



Oil Pan Wash Out/Disposal Rack east of Building 1818



Oil Pan Wash Out/Disposal Rack



Looking south along east side of Building 1818

