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Sampling and Analysis Plan

for

Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment

Site 17, Gould Island
Naval Station Newport
Jamestown, Rhode Island

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62467-04-D-0055
Contract Task Order 458

December 2009

SAP Worksheet #1a -- Title
(*UFP-QAPP Manual Section 2.1*)

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
December 2009

Phase 2 Remedial Investigation
And Baseline Ecological Risk Assessment
Site 17, Gould Island
Naval Station Newport
Jamestown, Rhode Island

Prepared for:
Naval Facilities Engineering Command Mid-Atlantic
9742 Maryland Avenue
Norfolk, Virginia 23511-3095

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard Suite 260
King of Prussia, Pennsylvania 19046-1433
(610) 491-9688

Prepared under:
Contract Number N62467-04-D0055
“CLEAN” Contract Task Order No. 458

QAPP Worksheet #1b (UFP-QAPP Manual Section 2.1) -- Approval Page

Document Title: Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), December 2009, Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment

Lead Organization: Naval Facilities Engineering Command Mid-Atlantic

Preparer's Name and Organizational Affiliation: Tetra Tech NUS, Inc.

Preparer's Address and Telephone Number: 234 Mall Boulevard Suite 260, King of Prussia, Pennsylvania 19046-1433

Preparation Date (Day/Month/Year): 31 December 2009

Investigative Organization's Project Manager: 12/29/09
Signature/Date
Stephen S. Parker, Tetra Tech NUS, Inc.

Investigative Organization's Project QA Manager: T. Johnston 12/29/09
Signature/Date
Tom Johnston, PhD, Tetra Tech NUS, Inc.

Lead Organization's Project Manager:
Signature/Date
Winoma Johnson, PE, U.S. Navy Mid Atlantic

Lead Organization QA Officer:
Signature/Date
Navy Chemist, U.S. Navy Atlantic

Approval Signatures:
Signature/Date
Kymberlee Keckler, U.S. EPA

Signature/Date
Paul Kulpa, RIDEM

QAPP Worksheet #1b (UFP-QAPP Manual Section 2.1) -- Approval Page

Document Title: Draft Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), January 2008, Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment

Lead Organization: Naval Facilities Engineering Command Mid-Atlantic

Preparer's Name and Organizational Affiliation: Tetra Tech NUS, Inc.

Preparer's Address and Telephone Number: 234 Mall Boulevard Suite 260, King of Prussia, Pennsylvania 19046-1433

Preparation Date (Day/Month/Year): March 2008

Investigative Organization's Project Manager: Stephen S. Parker

Digitally signed by Stephen S. Parker
DN: cn=Stephen S. Parker, c=US, ou=Tetra Tech NUS, email=stephen.parker@tetratech.com
Reason: I am approving this document
Date: 2008.03.28 09:52:37 -04'00'

Signature/Date
Stephen S. Parker, Tetra Tech NUS, Inc.

Investigative Organization's Project QA Manager:

Signature/Date
Tom Johnston, PhD, Tetra Tech NUS, Inc.

Lead Organization's Project Manager:

Signature/Date
James Colter, PE, U.S. Navy Mid Atlantic

Lead Organization QA Officer: TUCKERJONATHAN.P.1239524180

Digitally signed by TUCKERJONATHAN.P.1239524180
DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=TUCKERJONATHAN.P.1239524180
Date: 2008.03.27 17:29:16 -04'00'

Signature/Date
Jonathan Tucker, U.S. Navy Atlantic

Approval Signatures:

Signature/Date
Kymberlee Keckler, U.S. EPA

Signature/Date
Paul Kulpa, RIDEM

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) encompasses field sampling and quality assurance requirements for the Phase 2 Remedial Investigation and Baseline Risk Assessment for Site 17, Gould Island, Naval Station Newport, Jamestown, Rhode Island. This document constitutes the planning document, addressing specific protocols for sample collection, sample handling and storage, chain-of-custody, laboratory and field analyses, data validation, and data reporting.

Site 17, located at the northern end of Gould Island, includes the former Building 32 and the immediate surroundings. The site was home to a Navy torpedo overhaul shop in the 1940s until it ceased major operations in the 1950s. During that time, operations included degreasing, parts washing, electroplating, sandblasting, testing, and other operations. The site occupies approximately 6 acres and is bordered by state property to the south and is surrounded by a portion of the East Passage of Narragansett Bay to the east, north, and west. The site buildings have been demolished, and the island is generally unoccupied, but accessed by trespassers occasionally.

The building and facilities were demolished in the period from 1999 through 2002. The concrete roadways were removed at that time and polychlorinated biphenyls (PCBs) were detected in soil near transformer buildings around Building 32. PCB-contaminated concrete and soil were removed under Toxic Substances Control Act (TSCA) removal actions in the same time period.

A Phase 1 remedial investigation (RI) was conducted in 2005. Efforts conducted included extensive sample collection which included groundwater, soil, sediment, and biota samples. Sediment and biota samples were collected from intertidal and subtidal areas around Gould Island.

The Phase 1 RI report recommended that a Phase 2 Remedial Investigation be conducted to provide a baseline ecological risk assessment (BERA) and to close data gaps found.

The Phase 2 RI, which is described in this document, will consist of two portions. The onshore portion will require the installation of soil borings near the former coal pile area and adjacent to the former rigging platform. Borings near the former coal pile area will be used to determine if there is an upgradient source of fuel oil contamination that was found during the Phase 1 RI. Samples will be analyzed for PAHs, Target Analyte List (TAL) metals, gasoline range organics (GRO), and diesel range organics (DRO). Borings will also be completed adjacent to a former PCB removal area at the Former Rigging platform at the north end of the site. These borings are intended to confirm that residual contamination is not present at unacceptable levels. Samples will be analyzed for PCBs, polycyclic aromatic hydrocarbons (PAHs), and select metals.

The offshore portion of the Phase 2 RI will include an extent of contamination sediment study, a BERA, and sediment transport modeling (not detailed in this SAP/QAPP). The extent of contamination effort will consist of sediment samples collected from three distinct areas: the Stillwater basin area, the northeast shoreline, and the northwest shoreline. Phase 1 RI sampling detected PCBs, PAHs, and metals in these areas. Samples to be collected will be analyzed for PCB Aroclors, PAHs, and metals. The data will augment the Phase 1 RI data set.

The BERA effort will include the collection of additional sediment samples and biota samples. Sediment samples will be analyzed for PCB homologues, PAHs, metals, and grain size. Biota samples will be analyzed for PCB homologues, metals, and percent lipids. In addition, sediment will be collected for toxicity testing (28-day *Leptocheirus*).

A Tier 2 sediment transport model study as defined by the *User's Guide for Assessing Sediment Transport at Navy Facilities* will be conducted. Data collection efforts that will be incorporated into the model will include a bathymetry study and grain size analysis. The model will be used to determine the likelihood that sediment contamination in the Stillwater Area and other areas will migrate to other areas. A separate planning document will be prepared to detail the data collection and evaluation efforts for the sediment transport model.

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REFERENCES

LIST OF APPENDICES

A Conceptual Site Model
B Navy Ecological Risk Assessment Tiered Approach
C Selected TtNUS Standard Operating Procedures
D Selected Field Documentation Forms
E Mitkem Laboratories Selected Standard Operating Procedures and Quality Assurance Program Manual
F Columbia Analytical Services Selected Standard Operating Procedures

Acronyms

AA	Atomic Absorption
ANSI/ASQ	American National Standards Institute/American Society for Quality
ASTM	American Society for Standards and Materials
BOD	Biological Oxygen Demand
CA	Corrective Action
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CRDL	Contract-Required Detection Limit
CSM	Conceptual Site Model
CTO	Contract Task Order
CWA	Clean Water Act
DoD	Department of Defense
DCN	Document Control Number
DQI	Data Quality Indicator
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FCR	Field Change Request
FS	Feasibility Study
FSP	Field Sampling Plan
GA	Rhode Island groundwater quality classification – groundwater is suitable for drinking water use without treatment
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPC	Gel Permeation Chromatography
GPS	Global Positioning System
GW	Ground Water
ICP	Inductively Coupled Plasma
IDQTF	Intergovernmental Data Quality Task Force
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols (Manual)
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOU	Memorandum of Understanding
MPC	Measurement Performance Criteria
MQO	Measurement Quality Objectives
MS/MSD	Matrix Spike/Matrix Spike Duplicate
MSR	Management Systems Review
NEIC	National Enforcement Investigations Center
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
PA/SI	Preliminary Assessment/Site Investigation
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCBs	Polychlorinated Biphenyls
PDF	Portable Document Format
PG	Professional Geologist

PM	Project Manager
PQOs	Project Quality Objectives
PRP	Potentially Responsible Party
PRQL	Project-Required Quantitation Limit
PT	Proficiency Testing (previously known as performance evaluation (PE) sample)
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QMP	Quality Management Plan
QS	Quality System
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RTM	Remedial Technical Manager
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SM	Site Manager
SOP	Standard Operating Procedure
SQLs	Sample Quantitation Limits
SRM	Standard Reference Material
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compounds
SW	Surface Water
TCLP	Toxicity Characteristic Leaching Procedure
TBD	To Be Determined
TSA	Technical Systems Audit
UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
VOA	Volatile Organic Analytes
VOC	Volatile Organic Compounds
VSP	Visual Sample Plan

SAP Worksheet #2 -- SAP Identifying Information

(*UFP-QAPP Manual Section 2.2.4*)

Site Name/Number: Site 17, Gould Island, NAVSTA, Newport, Rhode Island
Operable Unit: NA
Contractor Name: Tetra Tech NUS, Inc.
Contract Number: N62467-04-D-0055
Contract Title: Comprehensive Long-Term Environmental Action Navy
Work Assignment Number (optional): CTO 458

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)* .

2. Identify regulatory program: CERCLA

3. This SAP is a project specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Navy, EPA, RIDEM, TtNUS, NOAA, Gannett Fleming, Sea Engineering, CH2M Hill, and TechLaw meeting	January 19, 2007
Internal TtNUS meeting	August 23, 2007
Navy, EPA, RIDEM, and TtNUS conference call	October 9, 2007

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

Title	Date
Work Plan for Remedial Investigation, Site 17, Building 32, Gould Island, Naval Station Newport, Rhode Island	July 2004

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

U.S. EPA, Regulatory Oversight	NAVFAC, Responsible Party Representative
RIDEM, Regulatory Oversight	TtNUS, Contractor to NAVFAC
NAVSTA, Property Holder	

7. Lead organization:

U.S. Navy

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

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

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	**NA**
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	**NA**
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	**NA**
Data Management Tasks		
29	Project Documents and Records Table	**NA**
30	Analytical Services Table Analytical and Data Management SOPs	**NA**
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	**NA**
32	Assessment Findings and Corrective Action Responses Table	**NA**
33	QA Management Reports Table	**NA**
D. Data Review		
34	Verification (Step I) Process Table	**NA**
35	Validation (Steps IIa and IIb) Process Table	**NA**
36	Validation (Steps IIa and IIb) Summary Table	**NA**
37	Usability Assessment	**NA**

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Winoma Johnson, PE	Remedial Project Manager	NAVFAC Mid-Atlantic	757-444-3418	winoma.johnson@navy.mil	Not applicable (NA)
Cornelia Mueller	IR Program Manager	NAVSTA Newport Environmental	401-841-7561	cornelia.mueller@navy.mil	NA
Jonathan Tucker	Project Chemist	NAVFAC Atlantic	757-322-8288	jonathan.tucker@navy.mil	NA
Dave Barclift	Ecological Risk Assessor	NAVFAC	215-897-4913	david.barclift@navy.mil	NA
Kymberlee Keckler	Remedial Project Manager	USEPA Region 1 Federal Facilities	617-918-1385	Keckler.Kymberlee@epa.gov	NA
Paul Kulpa	Remedial Project Manager	RIDEM Div Site Remediation	401-222-2797	Paul.Kulpa@dem.ri.gov	NA
Stephen Parker	Project Manager	TtNUS	978-474-8400	stephen.parker@ttnus.com	NA
Kelly Carper	Quality Assurance Manager	TtNUS	412-921-7273	kelly.carper@ttnus.com	NA
Matt Soltis	Health and Safety Manager	TtNUS	412-921-8912	matt.soltis@ttnus.com	NA
Aaron Bernhadt	Ecological Risk Assessor	TtNUS	412-921-8433	aaron.bernhardt@ttnus.com	NA
Thomas Campbell	Site Manager	TtNUS	978-474-8400	thomas.campbell@ttnus.com	NA
Johanna Traut	FOL	TtNUS	978-474-8400	johanna.traut@ttnus.com	NA
Lucy Guzman	Lead Chemist	TtNUS	978-474-8400	lucy.guzman@ttnus.com	NA
Kayleen Jalkut	Lead Geologist	TtNUS	978-474-8400	kayleen.jalkut@ttnus.com	NA
Ed Lawler	Total Quality Manager	Mitkem Laboratories 175 Metro Center Boulevard Warwick, Rhode Island 02886-1755	401-732-3400 ext. 315	elawler@mitkem.com	NA
Mark Wilson	Business Development Manager	Columbia Analytical Services 1 Mustard Street, Suite 250 Rochester, NY 14609	585-288-5380	mwilson@rochester.caslab.com	NA

SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

(*UFP-QAPP Manual Section 2.3.2*)

Project personnel who are responsible for implementing portions of the SAP will be provided copies of the applicable SAP sections. Their signatures or email receipt date will indicate that they have read the applicable SAP sections and will perform the tasks as described. If only a portion of the SAP was reviewed, then personnel should note which sections were reviewed.

Organization: TtNUS

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Stephen Parker	PM, general project management	978-474-8400			
To Be Determined	FOL, field activities management	978-474-8400			
Tom Johnston	QAM, quality assurance management, data quality review oversight	412-921-8615			
Matt Soltis	HSM, health and safety management	412-921-8912			
Lucy Guzman	Project Chemist, laboratory procurement oversight, data quality review, and chemistry support	978-474-8400			
Thomas Campbell	SM, project and field activities management support	978-474-8400			
Aaron Bernhardt	Ecological Risk Assessor, ecological risk assessment	978-921-8433			
To Be Determined: Project Manager will assign	Field Sample Collection Specialist/ Sample collection, packaging, shipment.	978-474-8400			
To Be Determined: Project Manager will assign	GIS specialist, rendering of spatial data	978-474-8400			
To Be Determined: Project Manager will assign	Database specialist, tabular data presentation and analysis	978-474-8400			

Organization: Mitkem Laboratories

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Ed Lawler	Mitkem, Laboratory Operations Manager	401-732-3400, ext. 315			

Organization: Columbia Analytical Services

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Mark Wilson	Columbia Analytical Services, Laboratory Point of Contact	585-288-5380			

Organization: Navy

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Winoma Johnson, PE	Project Manager, prime contractor oversight and management	757-444-0825			
Cornelia Mueller	IR Program Manager, overall lead agency responsibility for project performance	401-841-7561			
David Barclift	Ecological Risk Assessor	215-897-4913			
To Be Determined: Navy Project manager will assign.	Project Chemist	TBD			

Organization: RIDEM

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Paul Kulpa	Project Manager, oversight of Navy performance	401-222-2797			

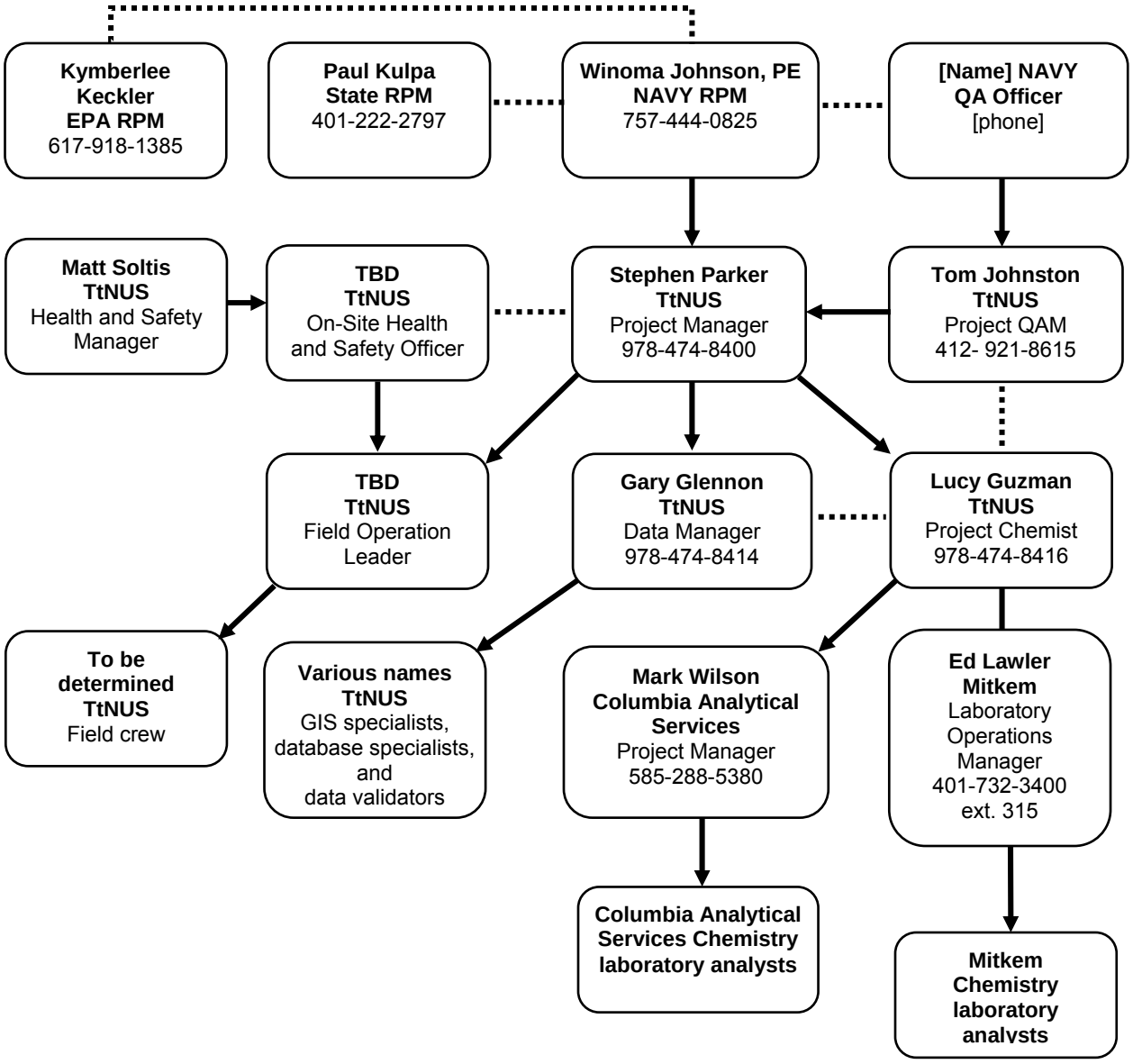
Organization: U.S EPA Region 1

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Kymberlee Keckler	Project Manager, oversight of Navy performance	617-918-1385			
Steve DiMattei	Chemist	617-918-8369			

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— Lines of Communication ·····



SAP Worksheet #6 -- Communication Pathways

(*UFP-QAPP Manual Section 2.4.2*)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Field task modification requests	TtNUS FOL	Johanna Traut	978-474-8400	Prior to implementing requested change, get approval from TtNUS PM the day the modification is necessary. Document via FTMR form within 1 week of implementing the field task modification.
QAPP amendments	Navy Remedial Project Manager (RPM)	Winoma Johnson	457-444-3418	Send scope change to within 1 week to TtNUS Program office prior to implemeting any changes in scope
Changes in schedule	TtNUS PM	Steve Parker	978-474-8400	Inform Navy via schedule impact letter as soon as impact is realized and with enough time to implement the changes in an organized manner. Communication with Navy should occur within 1 or 2 days, or 1 week at the most, of the change in schedule.
Issues in the field that result in changes in scope of field work	TtNUS FOL TtNUS PM	Johanna Traut Steve Parker	978-474-8400 978-474-8400	The FOL will inform the PM on a daily basis of any field issues. The PM will subsequently inform the RPM; and the RPM will issue scope change if warranted; Scope change to be implemented before work is executed
Recommendations to stop work and initiate work upon corrective action	TtNUS FOL TtNUS PM TtNUS QA Officer TtNUS HSM Navy RPM	Johanna Traut Steve Parker Kelly Carper Matt Soltis Winoma Johnson	978-474-8400 978-474-8400 412-921-7273 412-921-8912 757-444-3418	Responsible Party immediately informs subcontractors, the Navy, and Project Team
Analytical data quality issues	Mitkem Laboratory Columbia Analytical Services TtNUS Project Chemist TtNUS QA Mgr	Ed Lawler Mark Wilson Lucy Guzman Tom Johnston	401-732-3400 ext. 315 585-288-5380 412-921-7273 412-921-8615	Immediately notify TtNUS Project Chemist Notify Data Validation Staff and TtNUS PM if necessary

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(*UFP-QAPP Manual Section 2.4.3*)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Steve Parker	PM	TtNUS	Oversees project, financial, schedule, and technical day to day management of the project.	B.A. Biology, M.S. Environmental Studies, 20 years environmental investigation experience.
TBD	FOL	TtNUS	Supervise, coordinate, and perform field sampling activities prepare all geological interpretation and text.	Can be provided upon request
Tom Johnston	QAM	TtNUS	Ensure Quality aspects of the CLEAN program are implemented.	B.S. Chemistry, PhD Analytical Chemistry, 29 years environmental experience
Matt Soltis	HSM	TtNUS	Oversee TtNUS CLEAN Program safety	B.S. Industrial Safety Sciences, 24 years of environmental experience
Lucy Guzman	Project Chemist	TtNUS	Prepare SAP, prepare lab scope, coordinate with lab, and data quality review.	M.S. Chemistry, over 25 years experience in environmental analysis and quality control.
Thomas Campbell	SM and Project Health and Safety Officer	TtNUS	Prepare SAP, HASP, and assist wit h technical management of the project.	M.S. Zoology, 14 years of environmental experience
Ed Lawler	Total Quality Manager	Mitkem	Coordinate analyses with lab chemists, ensure the scope is followed, QA data packages, communicate with TtNUS staff.	Can be provided upon request
Mark Wilson	Business Development Manager	Columbia Analytical Services	Coordinate analysis with lab chemists, ensure the scope is followed, QA data packages, communicate with TtNUS staff.	Can be provided upon request
Aaron Bernhardt	Ecological Risk Assessor	TtNUS	Perform baseline ecological risk assessment calculations, provide oversight of ecological sampling design.	B.S. Biology, M.S. Chemical Hazard Assessment, 17 years ecological risk assessment experience
TBD: TtNUS project manager will assign.	Field Crew Members, database specialists, and GIS specialists	TtNUS	Collect, package, and ship samples in accordance with work plan.	Sufficient training to be able to implement TtNUS work plans and SOPs to meet project objectives.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(*UFP-QAPP Manual Section 2.4.4*)

The following table is used to identify and describe any specialized/non-routine project specific training requirements or certifications needed by personnel in order to successfully complete the project or task. OPNAV 5090.1 instructions are not considered specialized training; the OPNAV training requirements represent routine, minimum requirements that are mandatory for all DON projects.

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
See below						

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, 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(b)(4). Safety requirements are addressed in greater detail in the accompanying site-specific TtNUS Health and Safety Plan (HASP), prepared under separate cover.

The selected analytical laboratories (Mitkem Laboratories and Columbia Analytical Services) have successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program and described in “Department of Defense Quality Systems Manual (DOD QSM)” January 2006.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(*UFP-QAPP Manual Section 2.5.1*)

Project Name:	**Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment SAP**	Site Name:	**Site 17, Gould Island Naval Station Newport**
Projected Date(s) of Sampling:	**September 2009 – July 2010**	Site Location:	**Jamestown, Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: January 18, 2007

Scoping Session Purpose: Discuss Phase 2 RI and Baseline Ecological Risk Assessment requirements

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
S. Parker	Project Manager	TtNUS, Inc.	978.474.8434	stephen.parker@ttnus.com	Project manager
A. Bernhardt	Risk Assessor	TtNUS, Inc.	412-921-8433	aaron.bernhardt@ttnus.com	Ecological risk assessor
J. Colter	Senior Project Manager	NAVFAC MIDLANT	757-444-4217	james.colter@navy.mil	Senior Project Manager
C. Mueller	Base Manager	Navy	401-841-7561	cornelia.mueller@navy.mil	Base Manager
B. Chadwick		Navy		bart.chadwick@navy.mil	
K. Keckler	Remedial Project Manager	EPA	617-918-1385	keckler.kymberlee@epa.gov	Remedial Project Manager
E. Hayter		EPA	706-355-8303	hayter.earl@epa.gov	Sediment transport modeler
B. Hoskins		EPA	617-918-8375	Hoskins.bart@epa.gov	Ecological risk assessor
P. Kulpa	Project Manager	RIDEM	401-222-2797	Paul.Kulpa@dem.ri.gov	State Remedial Project Manager
K. Finkelstein	Biologist	NOAA	617-918-1499	ken.finkelstein@noaa.gov	
C. Jones	Sediment transport scientist	SEA Engineering		cjones@seaengineering.com	Navy consultant
P. White	Sediment transport scientist	CH2M HILL		patricia.white@chsm.com	Navy consultant
S. Pauwels	Risk Assessor	Techlaw		pauwels.stan@epa.gov	EPA consultant
T. Finlayson	Project Manager	Gannett Fleming	781-380-7750	rtfinlayson@gfnet.com	EPA consultant

Comments/Decisions:
- Reviewed findings of Phase 1 RI.
- Agreed that the site investigation would examine the extent of contamination in areas identified in the Phase I.
- Discussed aspects of the Baseline Ecological Risk Assessment and recommended that two toxicity endpoints be used and that biota sampling should include a third species in addition to clams and mussels.
- The sediment transport model scope of work was reviewed and recommendations were made to include a bathymetric survey and the collection of samples for grain size analysis.
- Sediments, and soils that may be discharged to sediments, shall be analyzed for PCBs, PAHs, and TAL Metals as needed for for resolving extent of contamination and calculation of risk.

Action Items:
- Navy will prepare a bulleted list of proposed data that would be collected under the Phase 2 RI.
- The Tier 1 Sediment Transport Model report would be revised based on EPA comments.

Consensus Decisions:
- A Tier 2 Sediment Transport Model would provide information necessary for developing remedial alternatives in the Feasibility Study.

Project Name:	**Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment SAP**	Site Name:	**Site 17, Gould Island Naval Station Newport**
Projected Date(s) of Sampling:	**September 2009 – July 2010**	Site Location:	**Jamestown, Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: August 23, 2007

Scoping Session Purpose: Discuss overall Phase 2 RI requirements

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
S. Parker	Project Manager	TtNUS, Inc.	978.474.8434	stephen.parker@ttnus.com	Project manager
L. Guzman	Project Chemist	TtNUS, Inc.	978.474.8416	lucy.guzman@ttnus.com	Project chemist, SAP developer
A. Franke	Chemist	TtNUS, Inc.	978.474.8413	ann.franke@ttnus.com	SAP Author
T. Campbell	Site Manager	TtNUS, Inc.	978.474.8404	thomas.campbell@ttnus.com	SAP Author

Comments/Decisions:

- Reviewed scope of the site investigation WP/QAPP development.
- Identified four primary documents as sources of information for the WP/QAPP: Final Remedial Investigation For Site 17: Building 32, Gould Island, NAVSTA Newport, Rhode Island (Tetra Tech NUS, 2006), Final Work Plan For Remedial Investigation Site 17 Building 32, Gould Island, NAVSTA, Newport, Rhode Island (Tetra Tech NUS, 2004), Final Derecktor Shipyard Marine Ecological Risk Assessment Report, NAVSTA, Newport, Rhode Island (SAIC, 1997), Navy's Statement of Work for the project, and Navy's Confirmation of Negotiations.
- Reviewed current conceptual site model; objectives of site investigation; soil, sediment, and biota sampling to be performed; and analyses to be performed.
- T. Campbell will draft background text based on the conclusions of the Phase I RI and the current conceptual site model. A. Franke will begin drafting the data quality objectives.

Action Items:

- Develop draft WP/QAPP for S. Parker to review by October 25, 2007.
- Prepare technical specification for analytical laboratory procurement.

Consensus Decisions:

- Use the Work Plan/Quality Assurance Project Plan for Pre-Design Investigation, Former Sewage Treatment Plant, Naval Air Station South Weymouth, Weymouth, Massachusetts, Internal Draft (July, 2007), in additional to the Uniform Federal Policy for Quality Assurance Project Plans Manual (IDQTF, 2005), as the models to develop the SI work plan/QAPP.

Project Name:	**Phase 2 Remedial Investigation and Baseline Ecological Risk Assessment SAP**	Site Name:	**Site 17, Gould Island Naval Station Newport**
Projected Date(s) of Sampling:	**September 2009 – July 2010**	Site Location:	**Jamestown, Rhode Island**
Project Manager:	**Stephen Parker**		

Date of Session: October 9, 2007

Scoping Session Purpose: Discuss Preliminary Worksheets 10 and 11

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
S. Parker	Project Manager	TtNUS, Inc.	978.474.8434	stephen.parker@ttnus.com	Project manager
J. Colter	Senior Project Manager	NAVFAC MIDLANT	757-444-4217	james.colter@navy.mil	Senior Project Manager
C. Mueller	Base Manager	Navy	401-841-7561	cornelia.mueller@navy.mil	Base management
D. Barclift	Risk Assessor	Navy	215-897-4913	david.barclift@navy.mil	Risk assessor
K. Keckler	Remedial Project Manager	EPA	617-918-1385	keckler.kymberlee@epa.gov	Remedial Project Manager
B. Hoskins	Sediment transport scientist	EPA	617-918-8375	Hoskins.bart@epa.gov	Sediment transport modeler
P. Kulpa	Project Manager	RIDEM	401-222-2797	Paul.Kulpa@dem.ri.gov	Remedial Project Manager
A. Bernhardt	Risk Assessor	TtNUS, Inc.	412-921-8433	aaron.bernhardt@ttnus.com	Ecological risk assessor
K. Finkelstein	Biologist	NOAA	617-918-1499	ken.finkelstein@noaa.gov	Ecologist
T. Finlayson	Project Manager	Gannet Fleming	781 380-7750	rtfinlayson@gfnet.com	EPA consultant
C. Deacutis	Risk Assessor	RIDEM Support	401-222-4700	deacutis@gso.uri.edu	Narragansett Bay Estuary Program
L. Guzman	Project Chemist	TtNUS, Inc.	978.474.8416	lucy.guzman@ttnus.com	Project chemist, SAP developer
A. Franke	Chemist	TtNUS, Inc.	978.474.8413	ann.franke@ttnus.com	SAP Author
T. Campbell	Site Manager	TtNUS, Inc.	978.474.8404	thomas.campbell@ttnus.com	SAP Author

Comments/Decisions:

- Reviewed content of submitted preliminary worksheets 10 and 11.
- Discussed aspects of ecological sampling – analytical range of compounds, dose response relationships to be determined, collocate sediment samples with toxicity samples, add Phase 1 location IDs to sample figures,
- Discussed rationale for the collection of Asian shore crabs and green crabs. Asian shore crabs are less mobile but more aggressive than green crabs.
- Decided that laboratory analysis for PCB homologues would be conducted for the ecological samples. A total PCB number would be used for the ecological risk assessment.
- Decision made that the January 2007 meeting was a DQO meeting.
- Comment was made on how the data will be used and how the results would be handled. A weight of evidence approach was accepted.
- Comment was made by RIDEM that they would like a diversity analysis conducted. Navy did not concur. RIDEM requested another assessment end point. Tissue samples were suggested as another assessment endpoint. RIDEM would look into this option and the approach to average tissue samples.

Action Items:

- Develop a draft decision tree/flow chart by October 26, 2007.
- Add Phase 1 RI sample loactions to proposed sediment sample location map.

Consensus Decisions:

- Complete Gould Island Phase 2 RI SAP.
- The content of the preliminary worksheets 10 and 11 were acceptable to all parties.

SAP Worksheet #10 -- Problem Definition

10.1 Site Location and Physical Characteristics

Gould Island is located in the East Passage of Narragansett Bay, approximately 1.5 miles west of Newport, Rhode Island. Gould Island is located between Aquidneck and Conanicut Islands, and occupies approximately 52 acres. Site 17, which is located on the northern portion of Gould Island, is made up of land associated with the former torpedo overhaul shop (Building 32) and occupies approximately 6 acres of land. The Site is bordered by state property to the south and is surrounded by a portion of the East Passage of Narragansett Bay to the east, north, and west (Figure 10-1).

Figure 10-2 presents the Site features. The Site is centered on the former Building 32, which served as a torpedo overhaul shop between the 1940s and the 1950s. A series of man-made structures were built at the Site on a combination of filled land and a natural island formation. The Site buildings have been demolished, and the island is generally unoccupied, but is accessed by trespassers occasionally.

The north end of Gould Island, where the Site lies, is subject to prevailing (southwest in summer and northwest in winter) wind exposure and currents almost year round. Sedimentation is not evident in the intertidal areas, but some has occurred in the portion of the Stillwater Basin adjacent to the firing pier. The intertidal shoreline is subject to wave action and consists of a mixture of deteriorated steel sheetpile wall and a stony beachface (Figure 10-2).

Sediments are subject to the transport mechanisms governing the surface water. The water currents in the area are tidal, but also wind-driven. SAIC (1997) reported that the orientation of Narragansett Bay makes bay waters and the coastline particularly exposed to summer southerly winds, as well as winter north winds. Current patterns in the bay have been studied, and it has been reported that the winds and tide both equally direct water currents and mixing energy, and suggest that wind events can permeate the entire water column and at times provide more force than tidal flow in the bay (Weisburg, 1976).

It has been reported that a prevailing north to south current is present in the area, although this was not clearly evident to the divers performing the work during the Phase 1 RI along the shoreline (TtNUS 2005). Given that the bay is actually a large estuary, fed by freshwater provided via landmass on the east, west, and predominantly north sides, it would stand to reason that there must be a net north to south flow. If there is a prevailing current in the area small particles will be transported downstream based on their size and density.

Coastal geologists visited the site in January 2006 and conducted an evaluation of aerial photography available for the site. At this time, they noted sediment trapped by physical features, and orientation of sediments in these traps further suggested a north to south flow in the area of the site.

Fill was placed in the northern portion of the Site in the 1940s (placement of fill is described in Section 10.2 below). A till layer is present under the fill. In the southern portion of the site, till underlies the soils altered during site development. The bedrock at the Site is characterized as metamorphosed sedimentary rock, predominantly phyllite.

The groundwater at the Site flows generally from south to north across the Site. However, overburden groundwater appears to flow to the northeast, whereas the bedrock groundwater appears to flow to the northwest. Tidal influence was noted in both the overburden and bedrock aquifers at the Site during the Phase 1 RI. Phase 1 RI packer testing results indicated that the bedrock aquifer did not yield appreciable amounts of water (TtNUS, 2005).

10.2 Site History

Gould Island was purchased by the U.S. Government in 1918 (Presidential Proclamation, 1918). The southern end of the island was immediately developed as a torpedo storage and seaplane station (Snyder and Enright, 2003). The northern end of the island, where the Site resides, was developed in the 1940s as a weapons support center for naval vessels.

The Site was radically altered by the construction of the torpedo overhaul shop (Building 32) in the 1940s. The former hillside at the north end of the island was cut down approximately 20-30 feet, to the current elevation. That material was used as fill to construct the firing pier and other structures at the north end of the site. This resulted in the placement of fill where former Buildings 41, 50, and 44 were located (Figure 10-2).

Building 32 housed electroplating, machining, parts washing, buffing, grinding, and other machinery operations during the 1940s until 1951 (EEI, 1983). Other structures on the property included transformer buildings, a power plant, an acetylene generator building, an administration building, and various structures used for loading and unloading personnel, torpedoes, and other materials from submarines and small vessels. A discussion of the types of contaminants can be found in the proceeding section 10.3.

Ownership of the southern three-fourths of the island was transferred to the State of Rhode Island in the late 1970s. NAVSTA retains ownership of the northern section. A fence separates the two properties (Figure 10-2).

The buildings and facilities and the concrete roadways were demolished in the period from 1999 through 2002. Polychlorinated biphenyls (PCBs) were found in soil and concrete near transformer Buildings 53, 54, 56, 59, 60, and 61 and the rigging house (Building 52) when they were demolished (Figure 10-2). The rigging platform was located at Building 52. It is presumed to have been used to load heavy equipment on and off boats and barges, given the presence of a three-leg derrick also shown on historic maps for this area. PCB-contaminated concrete and soil were removed under Toxic Substances Control Act (TSCA) removal actions in the same time period. The source of the PCBs at the rigging house is unknown. The Phase 1 RI report indicates that, based on documentation from prior targeted environmental investigations and removal actions performed at the Site (described in Appendix A of the Phase 1 RI report), soils containing PCBs at concentrations below 10 mg/kg (the 2002 PCB sampling interim clean-up goal) are likely to be present at the former locations of these buildings.

A number of targeted environmental investigations and removal actions were performed at the Site prior to the Phase 1 RI. These investigations and actions are detailed in Appendix A of the Phase 1 RI report (TtNUS, 2006). The following discussion summarizes the description in the Phase 1 RI report of the contaminants found at the Site as of the completion of the Phase 1 RI.

10.3 Contaminants Found

The Phase 1 RI efforts included the collection of the following samples:

- 87 soil samples from borings and test pits,
- 14 groundwater samples from monitoring wells,
- 9 groundwater “discrete zone samples” from bedrock fracture zones,
- 66 sediment samples from intertidal and subtidal areas around Gould Island,
- 21 biota samples (clams and mussels (as available),
- 9 aqueous samples from standing water in test pits and in underground utilities, and
- 2 samples of soil and sludge from these utilities, and two samples of concrete.

In addition, data gathered for the RI included groundwater flow information, information on ecological receptors present and likely to be present, geotechnical information on soils and bedrock pertinent to groundwater and contaminant movement, information on sediment characterization, and over 300 soil samples for headspace screening analysis and soil characterization. Sediment data collected previously

by the United States Environmental Protection Agency (EPA) in 2003 were used to the extent possible in the risk assessments for the Site.

Activities associated with former Site operations have resulted in the presence of volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides, PCBs, petroleum hydrocarbons, metals, and cyanide in Site soils and adjacent sediments. Additionally, VOCs, SVOCs, petroleum hydrocarbons, and metals were detected in the Site groundwater at low concentrations. Tetrachloroethene and pentachlorophenol were the only contaminants that exceeded the Groundwater Objectives for GA aquifers established in the Rhode Island Department of Environmental Management (RIDEM) Remediation Regulations, and to federal Maximum Contaminant Levels (MCLs).

Soil results show the presence of specific, definable locations where contaminants [fuel oil, polycyclic aromatic hydrocarbons (PAHs), metals, and PCBs] are present in soil at concentrations exceeding state criteria. Most of these locations are sumps, isolated from the surrounding soils by the Building 32 foundation. However, one location is outside the foundation, at the former position of the dust collection building and acid storage building at the southwest corner of Building 32. Apparent “hot spots” of metals and petroleum are present in this soil. Although the contaminated area appears to be limited in extent, the contamination was not fully delineated and the source of petroleum is unknown. It is suspected that petroleum contamination is related to the presence of the former coal bed located adjacent to the southwest corner of Building 32: a past common practice to increase the ignitability of coal was to prime it with fuel oil, but use of this practice at this site is unconfirmed. Contaminants tend to leach from soils into groundwater over time with the chemicals the most soluble and least sorptive chemicals leaching the most readily. The Phase 1 RI report indicated that additional focused investigations may be necessary to find the source of the petroleum and confirm extent of metals contamination.

RIDEM personnel have suggested the following as additional sources for the contaminated area in the southwestern corner of Building 32: Petroleum contamination that was found beneath a vault, which contained a series of USTs, located west of Building 33; a number of drains known to exist in Building 33 which may have discharged to leach fields and/or points on the northern or western side of the island; and a groundwater interceptor trench which was located south and west of Building 32, which apparently discharged to the eastern side of the island.

In groundwater, low concentrations of VOCs and SVOCs were detected in several monitoring wells across the site, with no discernable spatial concentration pattern. Because of the lack of spatial pattern, a source of these organic contaminants is not immediately evident. Highest concentrations included pentachlorophenol and tetrachloroethene, both of which only minimally exceeded the respective GA Groundwater Objective and MCL (Rhode Island classifies site groundwater as “GA” which indicates that

groundwater is suitable for consumption without treatment). It was noted that the upgradient groundwater wells contained low concentrations of PAHs. Metals did not exceed drinking water standards. A low concentration of petroleum was detected in the well at the former location of Building 44, a former fuel storage facility. This is likely to be a remnant from the historic fuel release that occurred at Building 44, where there is an ongoing long-term monitoring program being conducted under the RIDEM Underground Storage Tank (UST) regulations.

RIDEM personnel state that PCBs were found in groundwater in the former location of Building 54, which is a transformer building, demolished and remediated under TSCA and RIDEM regulations. While PCBs were not detected in groundwater monitoring wells downgradient of Building 54 (TtNUS, 2001), trace levels of PCBs (<1 mg/L) were detected in standing water excavations completed for removal of PCB-contaminated soil at Building 54 (Foster-Wheeler, 2004).

Accumulated water in two test pits, TP-06 and TP-08 (not shown on Figure 10-2), exhibited detections of fuel components (benzene, toluene, and xylenes), petroleum hydrocarbons, and PAHs, likely from the fuel releases in these areas (area near southwest corner of Building 32). The source of the detections of BTEX and TPH in accumulated water in two test pits is currently unknown, but is one of the targets of this investigation. Water from test pits also showed detectable metal concentrations (chromium, lead, zinc, and copper), possibly a result of sandblasting, dust storage, or electroplating chemical storage in the area of the southwest corner of Building 32. Contaminants in groundwater will migrate with the groundwater, the movement of each contaminant being retarded by factors that reflect the chemical and physical nature of the chemical. The more mobile chemicals such as VOCs and soluble metals will migrate faster than the less mobile contaminants such as high molecular weight organic compounds such as PCBs and SVOCs and insoluble metals.

In addition to the observed petroleum-related contamination in groundwater, an overland release of PCBs to sediments appears to have occurred north of Building 32, near the former rigging platform. This is the location of a PCB-contaminated soil removal action conducted in 1999-2002. Prior to the removal actions, PCBs were likely transported overland with runoff from the area of the soil removal action, and deposited in sediments in this area.

Sediment concentrations of metals, PAHs, and PCBs in specific areas along the shoreline and in the subtidal zone exceeded sediment benchmarks. These areas are identified as the Stillwater Area to the north of the Site, the Northeast Shoreline Area, which received waste discharges from Building 32 operations, and, to a lesser degree, the Northwest Shoreline Area, which also received waste discharges from Building 32 operations (Figure 10-2). Compounds and elements detected at concentrations exceeding Long and Morgan sediment screening guidelines [Effects Range-Low (ER-L)] in Stillwater Area

samples included 11 PAHs at concentrations up to 28,000 ug/kg, total aroclor (up to 41,000 ug/kg), copper, lead, and mercury at concentrations up to 92 mg/kg, 90 mg/kg and 0.2 mg/kg respectively. In samples collected from the Northeast Shoreline Area, nine PAHs (up to 2400 ug/kg), aroclor-1260 (3,300 ug/kg), cadmium (3.2 mg/kg), chromium (3900 mg/kg), copper (78 mg/kg), lead (690 mg/kg), and mercury (0.7 mg/kg) were detected at concentrations above ER-L criteria. Samples collected from the Northwest Shoreline Area contained 13 SVOCs, aroclor-1260, cadmium, copper, and lead at concentrations above ER-L criteria, though at lower concentrations than other stations. Over time, contaminants can leach from sediments into surface water the same way they leach from soil into groundwater.

While chromium speciation was not conducted with the chemical analysis, it was deteremined that chrome VI, a more toxic form of chromium would not be present in the site because of the reducing environment observed in the Gould island sediment, as well as the water chemistry and other metals present in the sediment. Such a condition would not allow chromium released in a CrVI state to remain so after many years present in the sediment. It was therefore concluded that chromium in the sediment was likely to be present in a more stable CrIII state (TtNUS 11/19/08).

PCB concentrations in sediments were noted to be highest in the subtidal sediment at the Stillwater Area, north of the site. PCBs detected in the sediment at the Site included primarily Aroclor 1260. Aroclor 1260 was the PCB mixture that had been found in soil and concrete near the transformer buildings when they were demolished. The distribution of PCBs in sediment samples indicates a gradient, with highest concentrations in the north areas and lower concentrations to the south, along the entire eastern shoreline of Gould Island, continuing well south of the Site at the northern end of the island. PCBs were also found in clams and mussels at the discharge locations and in the Stillwater Area. A PCB removal action was conducted west of the former Building 54, and this removal action continued to the intertidal area.

Because PCBs are almost insoluble in water, the high concentrations of PCBs in sediments of the Stillwater Area are likely to remain in this area until the physical properties of the shoreline change. The breakwater protecting this shoreline has deteriorated, and hydraulic forces that can mobilize these sediments will increase as this barrier degrades. In addition, the former rigging platform at the north shoreline has partially collapsed, allowing soils behind the former sheet piling to erode. Continued erosion from the shoreline may cover the contaminated sediments and debris from the former rigging platform unless it is repaired. However, over time, the effects of hydraulic forces may re-expose these sediments, subjecting them to potential mobilization to other areas.

PAHs detected in sediment may be related to former degreasing operations in the overhaul shop, or they may be a remnant of fuel piping and storage at Building 44. PAHs were found in sumps and vaults within

the Building 32 foundation, and some of these sumps were previously noted to have floor drains that connected to the drains exiting to the northeast shoreline. Some PAHs at outfall locations near SD-304 and SD-305 are likely a result of releases at the southwest corner of Building 32, carried to the shoreline through pipelines and sewer systems (See Phase 1 RI Report).

Metals, and, to a lesser degree, cyanide were found at sediment sample locations where outfalls once discharged to the subtidal shoreline. Elevated concentrations of heavy metals present in the sediment near existing and historic outfalls can be traced back through pipeline transport from the electroplating rooms and dust collector, where high concentrations of heavy metals were found in soil samples. It is noted that cyanide was also found in the subtidal sediment sample near the former landfill at Gould Island (SD311, a reference sample southwest of the Site and presumed to be less affected by depositional sediment originating from the Site). This indicates a possible second, off-site source of cyanide in the area.

Overall, contaminants appear to have been released to the sumps and equipment trenches in Building 32, as well as through drains and waste discharge systems at the site. For the most part, the building systems appear to have collected and routed the waste materials from the source locations to the shoreline during operations at the Site, with one exception, in the area of the former dust collection building located at the southwest corner of Building 32.

Five primary pathways of contaminant transport were identified: The first is the storm drainage system and “trench drain” that gathered groundwater and storm water from the roadway and storage areas (acid storage shed, dust collection building) outside the southwest corner of Building 32. This drain discharged that water to the shoreline at the southeast corner of Building 32. The second pathway is the floor drain system from the electroplating room and other portions of the building that appears to have gravity-drained wastes from the building to the shoreline to the east. The third pathway is the sanitary waste system upgrade installed in approximately 1956 and 1957: sewer system upgrades were installed to redirect some sanitary waste to the northwest shoreline. Some mixing of electroplating materials with the sewer waste appears to have occurred. At this location a junction box and dry well were found, constructed with a gravel bottom positioned to allow liquids to filter to the bay. The fourth pathway is releases of PCBs from transformers via groundwater migration and overland flow to adjacent sediments. The fifth pathway is releases of petroleum and VOCs from USTs, pipes or degreasing operations to groundwater. Refer to Section 10.1 for a description of contaminant pathways once they enter the marine environment.

10.4 Risk Assessment

Currently the site remains vacant. The Navy patrols the area, which is also reportedly posted to keep off government property. However, there is evidence that the property is occasionally visited by trespassing fishermen. The site is only accessible by boat, the nearest land mass being Jamestown (Conanicut Island), 1600 yards (approximately ¾ mile) to the west. Newport and Middletown lie approximately 1 mile to the east. The Navy reports that they will continue to hold this property for the foreseeable future for possible industrial or military use. Thus, likely future use is limited to trespassers, industrial workers, and construction workers. Other potential future uses of the site include use by recreational visitors. There is no foreseeable future residential use at Gould Island due to its location.

The remainder of the island, which is technically not part of the site is owned by the state of Rhode Island. This property is wooded and unoccupied. The State of Rhode Island identifies this area as “wildlife refuge”, similar to other remote islands in Narragansett Bay.

The Baseline Human Health Risk Assessment performed during the Phase 1 RI evaluated human exposure to surface soil, subsurface soil, groundwater, sediment, and shellfish.

Non-cancer risks estimated for adult recreational visitors, trespassers, and current industrial workers at the study area were below the non-cancer target risk level of 1.0.

The non-cancer risks to the current and future child recreational visitors, future industrial workers, future construction workers, and fishermen (both recreational and subsistence fishermen ingesting shellfish) were estimated to be above the target risk level of 1.0. These risks are driven by specific contaminants including chromium (intertidal sediments); cadmium (subsurface soil); PCBs, arsenic, and thallium (shellfish ingestion); and PAHs and PCBs within excavation trenches (groundwater and soil).

The cancer risks estimated for the future recreational visitors and trespassers exposed to surface soils and intertidal sediments, current industrial workers exposed to surface soils, as well as recreational fishermen consuming clams and mussels from the site, were within or less than the EPA cancer risk target range of 1E-4 to 1E-6. The cancer risk for the current industrial workers, future industrial workers, and the current and future recreational fishermen exceeded the RIDEM acceptable risk level of 1E-5.
The cancer risks to the future construction workers and current and future subsistence fishermen were estimated to be greater than the EPA cancer risk target range of 1E-4 to 1E-6. These unacceptable risks are driven by specific contaminants, including: PCBs, pesticides, PAHs, and pentachlorophenol (shallow groundwater) and PCBs, PAHs, beta-BHC and arsenic (shellfish).

It should be noted that the subsistence fishing scenario is not known to currently exist and is unlikely in the future because of the unlikely assumption that 180 meals per year would be obtained from waters adjacent to the Site. To do so would require either year-round habitation at Gould Island, or repeated travel to this location by boat, ignoring many other more suitable fishing areas in favor of this single location. In addition, some of the contaminants which predict risk to fishermen (PAHs, arsenic) are found at similar concentrations in reference shellfish samples collected in Rhode Island waters, indicating that these contaminants are indicating that the shellfish do not appear to be accumulating these contaminants only from the site.

Based on the non-cancer and cancer evaluations, the following contaminants with non-cancer hazard quotients greater than 1.0 or cancer risks greater than 10^{-6} in a scenario with cumulative cancer risks greater than the RIDEM cancer risk level of 10^{-5} were identified as COCs: chromium in sediment; cadmium in subsurface soils; PAHs, gamma-BHC, heptachlor epoxide, 2-methylnaphthalene, naphthalene, and PCBs in trench air; PCBs, beta-BHC, thallium, and arsenic in clams; PCBs, PAHs, and arsenic in mussels; and PAHs and pentachlorophenol in shallow groundwater.

Exposure to lead, measured through blood lead models for worker exposures, was found to be below EPA's level of concern. The Baseline Human Health Risk Assessment calculated the probability that blood lead levels would exceed 10 µg/dl was less than the acceptable level of 5% established by EPA.

A screening ecological risk assessment was performed to evaluate the possibility of ecological risks to the terrestrial and marine receptors exposed to contaminants associated with the site. This screening assessment was conducted to determine whether any contaminants of potential ecological concern exist and to determine whether a more detailed, baseline ecological risk assessment (BERA), should be conducted.

No contaminants of potential concern (COPCs) were retained as contaminants of concern (COCs) for plants or soil invertebrate animals. A food chain model was used to estimate contaminant uptake into higher trophic level organisms, such as herring gulls, raccoons, rodents, and other birds. No contaminants were retained as COC for wildlife for further evaluation in the BERA.

For the marine exposures, SVOCs (primarily PAHs), pesticides, PCBs, and metals detected in the sediment were determined to be COPCs. The presence of these COPCs may result in adverse effects to marine organisms at the Site, therefore, they warrant additional evaluation. The Phase 1 RI report (TtNUS, 2006) concluded that a Baseline Ecological Risk Assessment (BERA) is necessary to quantify actual risks to ecological receptors from these contaminants in the sediment near the Site.

The Phase 1 RI report recommended that a Phase 2 RI be conducted to provide a BERA and to address data gaps defined in Section 10.5. This would include detailed evaluation of risk to ecological receptors from contaminants present, as well as additional evaluations of sediments of the Stillwater Area to the north of the Site, resolution of extent of fuel contamination at the southwest corner of Building 32, and clarification of the source of this contamination.

10.5 Problem Definition

At the completion of the Phase 1 RI, several data gaps were identified. To complete the RI it is necessary to close the data gaps sufficiently to complete the delineation of contamination and to complete the evaluation of risks. The Phase 1 RI also recommended that a Baseline ERA be conducted to quantify the actual risks to marine ecological receptors from the contaminants identified in the screening risk assessment in the marine sediment at the Site.

Specifically, data are needed in order to:

- Confirm extent of metals contamination in soil near the former dust collector at the southwest corner of building 32.

- Identify the source of petroleum contamination in soils near the southwest corner of former Building 32. Data obtained in this effort will be used to determine if remediation is necessary and to estimate potential remediation costs.

- Determine whether remnant PCBs, PAHs and metals, are present in the soil in the vicinity of the former PCB release area near the former rigging platform. If remnant contaminants are present, they may be poses adverse risk to human and terrestrial receptors.

- Determine if remnant PCBs, PAHs, and metals in the vicinity of the former PCB release area near the rigging platform continue to be a source of contamination in the Stillwater Area sediments. A BERA will be conducted in the Stillwater Area to determine if remnant contaminants in the PCB release area may be contributing to adverse risk to ecological marine receptors.

- Determine the extent of sediment contamination in three offshore areas that include the Stillwater Area north of the Site, the northeast shoreline where outfalls discharged, and the northwest shoreline where a sanitary sewer discharged. The extent of contamination will be used to determine if adverse ecological risk exists in these areas.

- Determine if unacceptable risk to marine ecological receptors exists by conducting a BERA consisting of steps 3 through 8 of the Ecological Risk Assessment Guidance for Superfund in accordance with Navy guidance (Navy Policy For Conducting Ecological Risk Assessments, April 1999). The BERA will use data collected during the Phase 1 RI and data collected as part of this effort (Phase 2 RI).

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

The following text describes the development of project quality objectives (PQOs) using EPA's data quality objective (DQO) process. Steps 2 through 7 of the DQO process are presented. The primary data users of this investigation will be the United States Navy (Navy).

Step 2 - Identification of the Decision Statement

Based on the Phase 2 RI data to be generated, as well as the existing Phase 1 RI data, the following decisions or determinations will be made by Navy, with input from EPA and RIDEM:

1. Confirm western extent of metals contamination already identified near the former dust collector at the southwest corner of Building 32.

2. Determine if petroleum contamination of soils near the southwest corner of former Building 32 is from an upgradient source. If it is so determined, further work may be needed under state regulations, otherwise the source will be presumed to be the former activities at Building 32.

3. In the area of the former PCB release near the former rigging platform, determine whether remnant PCBs, PAHs, and metals are in soil are likely to pose an unacceptable level of human health or ecological risk. If they are, the data will be evaluated as to whether these contaminants may have been or may now be a source of contamination in the Stillwater Area marine sediment. If the concentrations are present at levels above state direct exposure criteria and ecological sediment criteria, consider the soils in this area for remedial action during the FS to reduce their concentrations. Further investigation may be needed to determine the extent of contamination.

4. Determine if the extent of PAH, PCB, and metals contamination in the marine sediment at the Site has been adequately delineated. If so, then cease delineation efforts. If not, then evaluate the need with EPA and RIDEM to continue to delineate marine sediment contamination.

5. Determine through the completion of a BERA if unacceptable risk to ecological receptors exists in marine offshore habitats at the Site. If the BERA predicts unacceptable risk then a decision will be made in conjunction with EPA and RIDEM if a remedial action should be conducted. Remedial action alternatives will be evaluated during the FS. If the BERA does not predict unacceptable risk, then no remedial action will be conducted.

6. Determine if offshore contaminated marine sediments are likely to erode and transport through hydrodynamic processes by conducting a Tier 2 sediment transport evaluation. If sediments in contaminated areas are found to be likely to erode and transport a decision will be made during the FS stage of this investigation whether to remediate these areas. If sediments are not found to be likely to erode and transport, a decision will be made not to conduct a remedial action.

All of the information obtained during the Phase 2 RI will also be used to the refine the conceptual site model (CSM) as it was described in the Phase 1 RI. The Phase 1 CSM is included in this SAP in Appendix A.

Step 3 - Inputs to the Decision

Inputs to the decisions include laboratory chemical and physical data (i.e., grain size), laboratory toxicity data, and sediment transport modeling data. The decisions will be made according to the decision rules in Step 5.

Data generated from the laboratory analysis of soil and sediment samples collected during the Phase 2 RI will be used in combination with the previous data collected during the Phase 1 RI to resolve the extent of Site contamination. The sediment data will also be inputs to the BERA. The analytical parameter groups were chosen based on contaminants detected during the Phase 1 RI.

The decision statements are identified in the headers for the following decision inputs.

<u>Soil Investigations (Decision Statements 1 and 2)</u>

Soil borings will be advanced in the southwest corner of the Site, near the former coal storage area, and will be analyzed for PAHs, Target Analyte List (TAL) metals, and total petroleum hydrocarbons (TPH) measured as gasoline range organic compounds (GRO) and diesel range organic compounds (DRO). (Figure 11-1).

Soil borings will be advanced in the area near the former rigging platform and analyzed for PCBs, PAHs, and TAL metals (Figure 11-1).

Marine Sediment Extent of Contamination Investigations (Decision Statement 3)

Sediment samples from the Stillwater Area, the northeast shoreline, and the northwest shoreline will be collected and analyzed for PCBs, PAHs, and TAL metals to better define the extent of contamination for these chemicals (Figure 11-2).

Concentrations of contaminants exceeding Long and Morgan sediment screening guidelines [Effects Range-Low (ER-L) and Effects Range-Medium (ER-M)] and reference concentrations will be identified and plotted on figures.

Ecological Investigation (Decision Statement 4)

After the analytical results are obtained from the sediment contamination extent investigation, locations will be selected for collecting biota tissue samples and additional sediment samples (Figure 11-3). This sampling will be performed to obtain data for the BERA. The sediment samples will undergo 28-day *Leptocheirus plumulosus* full-life-cycle chronic toxicity tests (ASTM Method E 1367-03, Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Invertebrates) and will be analyzed for PCB homologs, metals, PAHs, TOC, and grain size. The biota tissue samples will be analyzed for PCB homologs, metals, and percent lipids. The data will be used to assess risk to benthic invertebrates, shellfish, mammals, and birds. If site-related risk is determined to exist, then COCs will be identified and preliminary remediation goals (PRGs) will be developed that will be protective of marine organisms during the FS.

Tier 2 Sediment Transport Model (Decision Statement 5)

A Tier 2 sediment transport evaluation will be conducted. The evaluation will use data from previously collected sediment samples and may also require the collection of additional sediment samples. The model will be used to determine the likelihood that sediment contamination in the Stillwater Area will migrate. This information will later be used to in the evaluation of remedial alternatives for sediments at the site. However a separate work plan document will be prepared that will contain all specific information on the methodology of the Tier 2 sediment transport model.

Project Action Limits

The Phase 2 RI and Phase 1 RI analytical data will be compared with the project action limits (Worksheet 15) as described in Step 5. The project action limits are set at the appropriate matrix-specific, risk-based or regulatory screening criteria, as follows:

- Soil – Coal bed area– The RIDEM Method 1Industrial/Commercial TPH Direct Exposure Criterion (RIDEM, 2004) (Worksheet 15a).

- Soil – Former rigging platform area – The RIDEM Industrial/Commercial Direct Exposure Criteria (RIDEM, 2004) and ecological sediment criteria (Worksheet 15b). A comparison to ecological sediment criteria (ER-L and ER-M sediment guidelines) will be conducted under the assumption that soil from the rigging platform has, is, or will erode into the Stillwater Area.

- Sediment – Extent investigation – The ecological risk-based criteria for marine sediment are listed in Worksheet 15b. These action limits are applicable to screening the data in order to identify COPCs for the BERA. As part of characterizing the extent of contamination, and for informational purposes, the sediment data will also be plotted on a site map and labeled as to whether they exceed ER-L and ER-M sediment benchmarks and reference concentrations (Long and Morgan, 1991 and Long et al., 1995). The ER-L and ER-M guidelines are presented in Table 11-1 below.

- Sediment – Ecological investigation – The ecological risk-based criteria for marine sediment listed in Worksheet 15c. Sediment samples will also be screened against reference values as shown in Figure 11-3. Reference samples will be collected during the Phase 2 RI from one of the following locations: Potter Cove or Cranston Cove, Jamestown, RI – The final location will be determined as described in Step 7, Worksheet 11.

- Biota – The calculated chemical concentration in the tissue samples that would result in an EEQ that is equal to one in the conservative food chain model. The calculations were based on exposure inputs and toxicity reference values, as detailed in Worksheet 15d.

Table 11-1

Comparison Criteria for Sediment Contamination Extent Mapping(1)

Analyte	CAS Number	Sediment Benchmarks(2)	
		ER-L (mg/Kg)	ER-M (mg/Kg)
PAHs			
2-Methylnaphthalene	91-57-6	0.07	0.670
Acenaphthene	83-32-9	0.016	0.500
Acenaphthylene	208-96-8	0.044	0.640
Anthracene	120-12-7	0.0853	1.100
Benzo(a)anthracene	56-55-3	0.261	1.600
Benzo(a)pyrene	50-32-8	0.43	1.600
Benzo(b)fluoranthene	205-99-2		

Analyte	CAS Number	Sediment Benchmarks[2]	
		ER-L (mg/Kg)	ER-M (mg/Kg)
Benzo(g,h,i)perylene	191-24-2		
Benzo(k)fluoranthene	207-08-9		
Chrysene	218-01-9	0.384	2.800
Dibenzo(a,h)anthracene	53-70-3	0.0634	0.260
Fluoranthene	206-44-0	0.6	5.100
Fluorene	86-73-7	0.019	0.540
Indeno(1,2,3-c,d)pyrene	193-39-5		
Naphthalene	91-20-3	0.16	2.100
Phenanthrene	85-01-8	0.24	1.500
Pyrene	129-00-0	0.665	2.600
TAL Metals			
Aluminum	7429-90-5		
Antimony	7440-36-0	2	25
Arsenic	7440-38-2	8.2	70
Barium	7440-39-3		
Beryllium	7440-41-7	-	
Cadmium	7440-43-9	1.2	9.6
Calcium	7440-70-2	-	
Chromium	7440-47-3	81	370
Cobalt	7440-48-4		
Copper	7440-50-8	34	270
Iron	7439-89-6		
Lead	7439-92-1	46.7	218
Magnesium	7439-95-4	-	
Manganese	7439-96-5		
Mercury	7439-97-6	0.15	0.71
Nickel	7440-02-0	20.9	51.6
Potassium	7440-09-7	-	
Selenium	7782-49-2		
Silver	7440-22-4	1.0*	3.7
Sodium	7440-23-5	-	
Thallium	7440-28-0	-	
Vanadium	7440-62-2		
Zinc	7440-66-6	150	410
PCBs			
Aroclor-1016	12674-11-2	0.0227	0.180
Aroclor-1221	11104-28-2	0.0227	0.180
Aroclor-1232	11141-16-5	0.0227	0.180
Aroclor-1242	53469-21-9	0.0227	0.180
Aroclor-1248	12672-29-6	0.0227	0.180
Aroclor-1254	11097-69-1	0.0227	0.180
Aroclor-1260	11096-82-5	0.0227	0.180
Total PCBs		0.0227	0.180

Analyte	CAS Number	Sediment Benchmarks[2]	
		ER-L (mg/Kg)	ER-M (mg/Kg)

1. These criteria will be used as described in Worksheet #11 to identify on a site map the concentrations of samples from the sediment extent investigation that exceed the criteria.
2. Long & Morgan (1991) and Long et. al (1995). Effects Range-Low (ER-L) and Effects Range-Median (ER-M) benchmarks for marine and estuarine sediments. The ER-L values are below the ER-M values, and the ER-L values are equal to the Project Action Limits in Worksheet #15b except for the ER-L of silver, which is higher than the Action Limit in Worksheet #15b. Therefore, the Project Quantitation Limit goals listed for sediments in Worksheet #15b are sufficiently low for mapping purposes.

Project action limits also help in the selection of sampling and analytical methods and in determining the project quantitation limits (PQLs) that laboratories must achieve. The PQLs are also presented in Worksheet 15.

Step 4 - Definition of the Boundaries of the Study

Sampling activities for the Phase 2 RI will be conducted late 2009 and 2010. The seasonal timing of activities will coincide with the Phase 1 RI to achieve temporal consistency.

Soil Investigations

Soils will be sampled near the former coal bed, an access road, and the reported former location of USTs adjacent to the former power plant of surface and subsurface soil that may have been impacted by overland and underground releases of petroleum and associated PAHs. Petroleum was detected downgradient of these areas during the Phase 1 RI, but the source of the contamination was not identified and the upgradient extent was not determined. Overland and underground releases of petroleum may have impacted surface and subsurface soil through percolation and infiltration.

The foundation of the power plant will not be investigated due to its inaccessibility resulting from the presence of remnant basement/foundation infrastructure. If information is found to suggest contaminants may have migrated under the foundation, this area may be added to a later investigation.

A second soil investigation will be focused on the former rigging platform PCB investigation area defined by the western portion of the former rigging platform. This area may have been impacted by overland releases from former PCB sources. A soil removal action was performed in 2002 to address a PCB release in the former rigging platform area.

Marine Sediment Extent of Contamination Investigations

The boundaries of the Stillwater Area are defined by the former rigging platform, the firing pier, and the breakwater ruins. Vertically, the boundaries of this investigation will extend from the surface up to maximum of four feet below sediment surface. Surficial contamination was detected in two sediment samples collected during the Phase 1 RI. This investigation will delineate the extent of vertical and horizontal contamination in the Stillwater Area that resulted from overland release, waste discharge and underground releases that occurred historically at the Site.

The boundaries of the northeast and northwest shoreline study area were determined by the locations of Phase 1 samples and the length of shoreline defining the northeast and northwest portions of the site. The Phase 1 samples were placed at the terminus of former outfall and waste drainage pipes and dock structures. This investigation will only target the surfical subtidal substrate to measure potential contamination from overland releases, waste discharges, and underground releases that occurred at the Site.

Ecological Investigation

After the analytical results are obtained from the sediment contamination extent investigation, locations will be selected for the ecological investigation. This investigation will be performed to obtain data for the BERA. The data will be used to assess risk to benthic invertebrates, shellfish, mammals, and birds.

Based on the concentrations detected from the extent investigation, locations will be selected over a concentration gradient (i.e., high, moderate, and low concentrations) to determine the relationship between sediment concentrations and toxicity or tissue concentrations with the goal of developing dose-response curves.

Ecological samples will be spatially located around the Site. The investigation will focus on three areas: Stillwater Area, Northeast Shoreline, Northwest Shoreline, and South end of island. A reference area will include the Potter Cove and/or Cranston Cove, Jamestown as described in Step 7.

Tier 2 Sediment Transport Model

The geographical area to be evaluated by the Tier 2 sediment transport model will include those areas included as part of the extent of contamination sediment investigation and the ecological risk investigation.

Step 5 - Decision Rules

The decisions and determinations listed in Step 2 will be made according to the following rules:

Soil Decisions

If PAH, TAL metal, and/or TPH (measured as GRO and DRO) concentrations in all soil samples near the southwest corner of the Site during the Phase 1 and Phase 2 RIs are less than the action limits identified in Worksheet 15, the associated locations will be identified as not exceeding the action limits, and no further action is needed; otherwise, the associated locations will be identified as exceeding the action limits and the areas with concentrations that exceed the action limits will be included for consideration of remedial action during the FS. A decision on potential remedial actions to be potentially taken will be made in conjunction with EPA and RIDEM. The decision making process will consider factors such as contaminant concentration levels, remedial action costs, and volume of contamination.

If contaminants are found in soil borings upgradient of the SW corner of the Site or if it is determined that contaminants may be present beyond the sampled area at concentrations exceeding the criteria in Worksheet 15, assume the contamination originated from an upgradient source, and further investigation may be needed under RIDEM UST/Petroleum regulations. Otherwise, declare the source to be identified as the former Building 32 and the extent of contamination to be established and cease investigation for this decision rule.

If measured concentrations of PCB, PAH, and metals in all soil samples collected from the former PCB release near the former rigging platform are less than action limits identified in Worksheet 15, the associated locations will be plotted on a site map as not exceeding the action limits and no further action is needed; otherwise, the associated locations will be labeled on the map as exceeding the action limits and the areas with concentrations that exceed the action limits will be included for consideration of remedial action during the FS. The FS will evaluate these locations as a possible source of the PCB, PAH, and metals contamination in the Stillwater Area sediment. This evaluation will be based on factors such as the relative levels of concentrations detected in the soil and sediment, and the types of PCB Aroclors detected in the soil and sediment.

Sediment Samples – Extent of Contamination

If measured concentrations of PCB, PAH, and metals detected in all sediment and biota samples in the Stillwater, Northeast Shoreline, and Northwest Shoreline Areas are less than action limits identified in Worksheet 15 and reference values (reference values will be obtained from reference locations sampled in Potter Cove or Cranston Cove, Jamestown) or if they are above the action limit but below the reference

values the associated locations will be plotted on a site map as not exceeding the action limits or reference values and no further action is needed.

If the measured concentration of a chemical is above the action limit and reference values in any sample, that chemical will be identified as a COPC and will be used to support the preparation of the BERA as described below.

Ecological Samples – Baseline Ecological Risk Assessment

The Phase 2 RI data (sediment and biota analytical data and toxicity test results) will be used in conjunction with the Phase 1 analytical data (sediment and biota) to conduct a BERA according to the decision tree in Figure 11-4. Following is a more detailed description of the decision tree elements. The decision rules listed below were developed to determine whether the chemical concentrations in the sediment and/or biota at the site are causing an unacceptable risk to the following ecological endpoints: benthic invertebrates, target biota species, and mammals and birds that consume the biota (these will be termed piscivorous wildlife in the BERA even though it will be assumed that they are consuming only shellfish in the food chain model). The Navy ecological risk assessment tiered approach is illustrated in a flow chart presented in Appendix B.

Benthic Invertebrate Endpoint

Potential risks to this endpoint will be determined by evaluating two measures of effects: sediment chemistry and sediment toxicity in a lines of evidence approach. The following presents the decisions that will be made based on each of these measures of effects.

Sediment chemistry: At each sample location where sediment chemistry data is obtained, the analytical data will initially be compared to the sediment screening level to determine whether the chemical will be initially selected as a COPC. If the measured chemical concentration in the sediment sample is greater than its respective screening level, the chemical will be selected as a COPC. If the chemical concentration in the sediment sample is less than its respective screening level, the chemical will not be selected as a COPC.

Sediment Toxicity: At each sample location where the 28-day *Leptocheirus plumulosus* toxicity test is conducted, the results of the toxicity test will be evaluated to determine whether the sediment is considered toxic to invertebrates at that location. Three test endpoints will be measured to evaluate risk to benthic invertebrates; survival, growth rate, and reproduction of the test organisms. In accordance with ASTM Method E 1367-03, site sample toxicity tests will consist of 5 replicate test chambers per treatment

to measure each test endpoint (survival, growth rate, and reproduction). These replicate tests will be used to calculate mean test end points, as described in the following paragraphs. The toxicity tests will be reviewed for adherence to ASTM Method E 1367-03 protocols.

The survival rates of the amphipods in the Site samples will be statistically compared to the survival rates in each reference sample (analysis of variance ANOVA, $p < 0.05$) to determine if the survival rate of the organisms in the Site samples is lower than the survival of organisms in the reference samples. The site sample results will be compared to the results in each of the reference sample individually (i.e., each site sample data set will be compared to each of the three reference sample data sets). Based on the results of the statistical comparison described above, the following decisions will be made for sediment invertebrates:

- If mean survival in any Site sample is not statistically different than the mean survival in at least two of the three reference samples, then that location will be classified as "No unacceptable site-related risk to sediment invertebrates" (i.e., risk not different than regional conditions).

- If mean survival in any Site sample is statistically different from the mean survival in at least two of the three reference samples, then that location will be classified as "Potentially unacceptable site-related risk to sediment invertebrates".

The growth rates of the amphipods in the Site samples will be statistically compared to the growth rates in the reference samples (analysis of variance ANOVA, $p < 0.05$) to determine if the growth rate of the organisms in the Site samples is lower than the growth of organisms in the reference samples. Based on the results of the statistical comparison described above, the following decisions will be made for sediment invertebrates:

- If mean growth in any Site sample is not statistically different than the mean growth in at least two of the three reference sample, then that location will be classified as "No unacceptable site-related risk" (i.e., risk not different than regional conditions).

- If mean growth in any Site sample is statistically different from the mean growth in at least two of the three reference samples, then that location will be classified as "Potentially unacceptable site-related risk".

The reproduction rates of the amphipods in the Site samples will be statistically compared to the reproduction rates in the reference samples (analysis of variance ANOVA, $p < 0.05$) to determine if the reproduction rate of the organisms in the Site samples is lower than the reproduction of organisms in the

reference samples. Based on the results of the statistical comparison described above, the following decisions will be made for sediment invertebrates:

- If mean reproduction in any Site sample is not statistically different than the mean reproduction in at least two of the three reference sample, then that location will be classified as “No unacceptable site-related risk” (i.e., risk not different than regional conditions).

- If mean reproduction in any Site sample is statistically different from the mean reproduction in at least two of the three reference samples, then that location will be classified as “Potentially unacceptable site-related risk”.

If any Site sample is classified as "potentially unacceptable site-related risk", concentration plots will be generated for the chemicals detected in the sediment samples. For some chemicals such as PAHs and PCBs, the plots may be generated for total values as opposed to the individual constituents. Concentration plots curves also will be generated with the mean ER-M quotient (ERM-Q), which will be calculated for each location using the following procedure:

1. Calculate the ERM-Q for the following parameters:
    - High Molecular Weight (HMW) PAHs
    - Low Molecular Weight (LMW) PAHs
    - Total PCBs
    - Individual metals (arsenic, cadmium, chromium, copper, lead, nickel, and zinc)

2. Average the ten ERM-Qs listed above for an overall mean ERM-Q

The concentration plots will consist of the constituent concentration along the x-axis and the percent survival, growth, or reproduction along the y-axis. The plots will be visually inspected to determine if there is a relationship between contaminant concentrations and toxicity (i.e., dose response curve), which may then be used to determine site-specific ecological benchmarks. If a dose-response relationship is observed for a chemical, no observed effects concentrations (NOECs) and lowest observed effects concentrations (LOECs) will be determined. If there is not a clear dose-response relationship for a chemical, the chemical data will be further evaluated to determine whether observed toxicity can be related to chemical concentrations. The NOECS will be the highest concentration of that chemical in a sample that was not classified as "potentially unacceptable site-related risk” and the LOEC will be the lowest concentration of that chemical in a sample that was classified as "potentially unacceptable site-related risk”. Professional judgment may be needed depending various factors such as the strength of the dose-response curve, difference in concentrations between the effects, etc.

Biota Tissue Concentration Endpoint

Biota chemistry: At each sample location where biota chemistry data is obtained, the analytical data will initially be compared to the maximum chemical concentration in the reference samples to determine whether the chemical concentration is elevated above reference concentrations. If the chemical concentration in the biota sample is less than maximum chemical concentration in the reference samples, it will be determined that the chemical is not site related in the organism. If the chemical concentration in the biota sample is greater than maximum chemical concentration in the reference samples, it will be determined that the chemical may be bioaccumulating in the organism. The concentrations of the chemicals that may be bioaccumulating in the tissue will be compared to tissue residue values (TRVs) (if available). TRVs, also known as critical body residues (CBRs), are tissue concentrations identified in scientific literature that are expected to result in impacts to the aquatic organism. They are different from background concentrations in that tissue concentrations greater than background concentrations do not necessarily indicate that impacts to the aquatic organism will occur. If the measured chemical concentration in the tissue sample is greater than the TRV, the organisms may be impacted by the chemical concentrations in their tissue. If the measured chemical concentration in the tissue sample is less than the TRV, the organisms are not likely to be impacted by the chemical concentrations in their tissue. If a TRV is not available for a chemical, but the concentration of that chemical is greater than background, then potential risks to aquatic organisms from that chemical will be discussed in the uncertainty analysis section.

For the mussel and clam PCB data, plots of chemical concentrations in tissue samples versus the chemical concentrations in the co-located sediment samples will be generated for chemicals that are detected in at least half of the tissue samples and have concentrations that are greater than reference concentrations in at least one sample. If the tissue/sediment concentration curve cannot be generated for a specific COPC, that COPC may be evaluated using other endpoints as shown on Figure 11-4. If there is not a positive correlation greater than $r^2 = 0.65$ (where r^2 is the correlation coefficient) between the chemical concentrations in tissue samples and the co-located sediment samples, it will be determined that the chemicals are not bioaccumulating in the tissue at the site.

Authors note: This is a difficult endpoint to evaluate for the following reason: 1. The crabs that are collected are assumed to have obtained their contamination over a larger area than one sample location. Therefore, the data cannot be used to evaluate risks from a particular sample location. Rather, the risks will be evaluated for the area where the crabs were collected (i.e., stillwater basin, northeast shoreline, etc.) 2. There is limited toxicological data based on chemical concentrations in tissue there are no standard methods for developing tissue residue value.

For the crab PCB data, plots of chemical concentrations in tissue samples versus the chemical concentrations in the sediment samples in the general area (i.e., Stillwater basin, Northeastern Shoreline, Northwestern Shoreline, South of Island) will be generated for chemicals that are detected in at least half of the tissue samples and have concentrations that are greater than reference concentrations in at least one sample. If there is not a positive correlation greater than $r^2 = 0.65$ (where r^2 is the correlation coefficient) between the chemical concentrations in tissue samples and the sediment samples in the general areas as described above, it will be determined that the chemicals are not bioaccumulating in the tissue at the site.

Overall Risk Evaluation: A lines of evidence approach will be used to determine whether the three measures (sediment chemistry, sediment toxicity, and biota chemistry) are resulting in an adverse impact to invertebrates at a particular location.

Piscivorous Wildlife Endpoint

Food chain modeling will be conducted for chemicals that are determined to be bioaccumulating in the tissue (as described above) to determine potential risks to piscivorous wildlife. The exposure parameters for the food chain model are included in Appendix B. Chemicals not detected in the biota samples will be included in the food chain model, but only the incidental ingestion pathway will be evaluated for those chemicals. Because the chemical concentrations may vary significantly in the different areas around the site, and because piscivorous wildlife will be feeding over a larger area than one sample location, separate food chain models will be conducted in each of the general areas (i.e., Stillwater basin, Northeastern Shoreline, Northwestern Shoreline, South of Island). The following presents the approach that will be used and the decisions that will be made to evaluate potential risks to piscivorous wildlife.

As a first step, the average tissue concentrations in each of the general areas (i.e., Stillwater basin, Northeastern Shoreline, Northwestern Shoreline, South of Island) will be compared to the project action limits found in Worksheet 15d. If a biota chemical concentration is below the action limit, food chain modeling will not be conducted for that chemical in that area. If the average concentration is greater than the action limit, that chemical will be identified as a COPC and included in the food chain model for that area.

- If the hazard quotient (HQ) is less than one for the chemical, based on average chemical concentrations, average exposure assumptions, and the No Observed Adverse Effects Level (NOAEL), it will be determined that risks to piscivorous wildlife are acceptable. Chemicals with HQs that approach 1.0 will be discussed in the uncertainties section as to whether they may support a cumulative risk to receptors.

- If the HQ is greater than one for the chemical, based on average chemical concentrations, average exposure assumptions, and the Lowest Observed Adverse Effects Level (LOAEL), it will be determined that risks to piscivorous wildlife are unacceptable.

- If the HQ is greater than one for the chemical, based on average chemical concentrations, average exposure assumptions, and the NOAEL, but is less than one based on the LOAEL, professional judgment will be used to determine whether risks to piscivorous wildlife are unacceptable. Professional judgement will include an evaluation of the following items at a minimum: detection frequency, magnitude of the HQ, home range of the receptor versus the size of the contaminated area, etc.

Tier 2 Sediment Transport Model

Specifics on the decision rules for the Tier 2 Sediment will be developed and presented in a separate work plan document. The results of the bathymetry study and the grain size analysis will be inputs to the Tier 2 sediment transport model. The model will be used to determine the likelihood that sediment contamination in the Stillwater Area and other areas will migrate to other areas. This information will be used in the evaluation of possible remedial alternatives for sediments at the Site.

Step 6 - Limits on Decision Errors

The Phase 2 RI program consists of the collection of samples for laboratory analysis and collection of field measurements. Sufficient numbers of samples, appropriate analytical and field methods, and appropriate quality assurance/quality control protocols will be applied to minimize errors that may affect future use of the data and subsequent decision making.

The number of sample locations to be collected for the fuel oil extent of contamination and the former rigging platform area investigations were based on professional judgment which took into account several factors. These decision factors included the size of the area to be investigated, findings of the Phase 1 RI investigation, the end uses for analytical data, and the cost of conducting the investigation. The fuel oil investigation, which is focused on a larger area, required more samples than the former rigging platform

investigation. The area over which the fuel oil investigation is focused is in the vicinity of the former coal pile. This land use history would suggest that potential hydrocarbon contamination could extend over a large area. The placement of samples extends westward from the Phase 1 RI test pit locations near the location of the former coal pile. The area where the former rigging platform investigation will occur is focused on a smaller area where a PCB removal took place. The number of samples collected in each area was considered by the planning team to yield a good spatial representation of the areas to be sampled for the available funding, with an emphasis on filling data gaps in those areas that were sparsely sampled in previous investigations. The Navy is proposing a biased sample effort in areas where contaminants have previously been identified. In addition, these locations are at and beyond areas that have been identified as source or release points. Targeting sampling to these areas minimizes a false negative decision (minimizes the possibility that contamination will be missed where it is actually present). There is no approach to minimize a false positive decision (minimize the possibility that contamination will be found where it does not exist) and the Navy accepts bias to false positive results.

Analytical results of Phase 1 RI test pit locations adjacent to the southwest corner of former Building 32 detected petroleum products although the source was not confirmed. A sample set of nine boring locations was selected to determine presence of fuel oil contamination upgradient of the former test pits in this area. The boring locations were selected upgradient of the former test pit area where elevated PAHs, Petroleum, and metals were found. If PAHs similar to those found in the former test pits are found in these borings, or if oil contamination is found at these locations, it may be presumed that there is an upgradient source of contamination to this area. If PAHs dissimilar to those in the former test pits are found in these borings, it will be presumed that the PAHs found at Building 32 originated from a former operation within or adjacent to building 32. Concentrations of metals will be compared with those found in the soils nearby, and used to assist determination of the western extent of the metals contamination in this area.

A sample set of four locations was selected to investigate the area adjacent to the former rigging platform PCB release area. Samples are spaced 20 feet apart across the former PCB release area at onshore locations adjacent to the Stillwater Area.

Several factors were taken into consideration to determine, thorough professional judgment, the number of marine sediment investigation samples. The number and placement of samples in the Stillwater Area reflected the size of the area being investigated. A greater number of samples were allocated to this area since only two Phase 1 RI samples were collected. Samples proposed along the northeast, northwest, and southern shorelines of the island are more strongly influenced by the locations of Phase 1 RI samples. Phase 2 sample locations were placed in proximity to locations where contaminants were detected and not detected in Phase 1 samples. In addition, the number and placement of Phase 2

sediment samples also considered and the nature of the marine environment. The dynamic physical characteristics of the ocean environment make distribution of contaminants more uniform. Currents, tides, storm events, and boat traffic have all contributed to this uniformity. Previous investigations have suggested that the area of contamination in marine sediments is restricted. A marine sediment investigation conducted by the EPA in 2003 determined that concentrations of contaminants in sediment along Gould Island's northeast, northwest, and south shorelines decreased with increasing water depth and distance from the island shoreline. Selection of samples for this investigation was therefore governed by water depth and distance from the shoreline. The number of sample locations also considered the inherent challenges of conducting a removal action of marine sediment. Removal actions conducted in offshore marine environments typically encompasses larger areas than those conducted in terrestrial environments. Cost considerations also affected the number of samples allocated for the Phase 2 RI. The number of samples collected in each area was considered by the planning team to yield a good spatial representation of the areas to be sampled for the available funding, with an emphasis on filling data gaps in those areas that were sparsely sampled in previous investigations.

A sample set of 20 locations was selected to adequately determine the extent of contamination detected during Phase 1 RI sampling in the Stillwater Area. Sample locations were spaced 60 feet apart next to the former rigging platform and 120 feet further out in Stillwater Area. Samples from four depths will be collected from each location as described in Worksheet 14.

A sample set of 30 was selected to investigation contamination detected at Phase 1 RI samples collected from the northeast and northwest shorelines. Of the 30 locations, 23 samples are designated for the northeast shoreline and seven are designated for the northwest shoreline. Phase 1 RI sampling included 13 locations along the northeast shoreline and 6 locations along the northwest shoreline.

Twenty-four sample locations will be selected for biota sampling and 30 locations will be selected for ecological sediment sampling. Based on the concentrations detected from the extent investigation, 24 locations will be selected over a concentration gradient (i.e., high, moderate, and low concentrations) to determine the relationship between sediment concentrations and toxicity or tissue concentrations with the goal of developing dose-response curves.

The measurement performance criteria for the laboratory analyses are described in Worksheet 12. Specific procedures for sample collection, analytical methods, and field and laboratory control samples are described in Worksheet 18.

Step 7 - Sampling Design

The DQO process presented in the EPA QA/G-4 document describes the use of various approaches for developing a data set. These approaches are based on contaminant distributions and outputs of the previous steps. The sampling design for the Phase 2 RI was based on the Phase 1 RI data and the data gaps that have been identified.

Soil, sediment, and biota samples will be collected by TtNUS personnel or TtNUS subcontractor personnel and analyzed off-site by subcontracted, Navy-approved fixed laboratories. Sample collection will be performed using hollow stem auger (HSA) drilling and split-barrel sampling techniques for on-shore soil investigations and a combination of vibracore and manual collection by divers for off-shore sediment and biota samples. Samples will be collected in accordance with TtNUS Standard Operating Procedures (SOPs).

7.1 Soil Investigations

Nine soil borings will be advanced in the southwest corner of the Site, near the coal bed area, and will be analyzed for PAHs, TAL metals, and TPH measured as GRO and DRO. Four samples will be collected from each boring at the following sample intervals: 0 to 6 inches, 12 to 18 inches, 24 to 30 inches, and 42 to 48 inches. Sample intervals may be adjusted if shallow bedrock is encountered (Figure 11-1). The sample interval for the deeper sample will be collected from the top of the saturated zone, as identified by the field geologist. Additionally, a third sample may be collected from any interval that shows signs of contamination.

Four soil borings will be advanced in the area near the former rigging platform and analyzed for PCBs, PAHs, and TAL metals. Two samples will be collected from each boring. One sample will be collected from 0 to 6 inches and the second at a deeper interval not to exceed 19.5 to 20 feet (Figure 11-1).

On-shore samples were arranged on the upgradient boundary of the site near the former coal pile. Sample locations proposed for the former rigging platform were placed along the southwestern perimeter of the former platform, in the vicinity of the PCB removal area.

7.2 Marine Sediment Extent of Contamination Investigations

Figure 11-4, Part 1 depicts the use of the data collected as part of “Extent of Contamination” sediment samples. Samples will be analyzed for bulk sediment chemistry only, and used to help select sample stations for “Ecological Sampling” described in Step 7.3.

Eighty sediment samples from the Stillwater Area, 23 sediment samples from the northeast shoreline, and seven sediment samples from the northwest shoreline will be collected and analyzed for PCBs, PAHs, and TAL metals to better define the extent of contamination for these chemicals (Figure 11-2).

Twenty sediment borings will be advanced in the Stillwater Area up to a maximum depth of four feet below sediment surface. Four samples will be collected from each boring at the following depth intervals: 0 to 6 inches, 6 to 12 inches, 12 to 24 inches, and 42 to 48 inches. Surface samples (0 to 6 inches) will collected from each northeast and northwest shoreline location.

By regulatory request, sediment borings will also be conducted at the locations of the phase 1 sample stations SD-304 and SD-311. Samples will be collected from six inch intervals to the depth of refusal of the vibracore device.

Marine sediment boring locations in the Stillwater Area were arranged in a grid pattern. Sediment sample locations along the northeast and northwest shorelines were arranged to compliment Phase 1 sediment locations so as to determine extent of contamination.

7.3 Ecological Investigation

After the analytical results are obtained from the sediment “extent of contamination” investigation, 30 locations will be selected for collecting biota tissue samples and additional sediment samples. This sampling will be performed to obtain data for the BERA. The sediment samples will undergo 28-day *Leptocheirus plumulosus* full-life-cycle chronic toxicity tests and will be analyzed for PCB homologs, metals, PAHs, TOC, and grain size. The biota tissue samples will be analyzed for PCB homologs, metals, and percent lipids.

Twenty-four locations will be selected for biota sampling and 30 locations will be selected for surface sediment (0 to 6 inches) sampling (six of these sediment locations will be selected in locations to be determined).

Ecological samples will be spatially located around the Site (Figure 11-3). Approximately 15 of the 24 locations will be located in the three areas of the Phase 2 sediment extent of contamination investigation. Three locations where the highest concentrations of cadmium, lead, and PCBs were detected during the Phase 1 RI (SD304, SD312, and SD317) will be sampled. Three new locations will be located on the southern end of Gould Island in order to further investigate an apparent gradient of decreasing concentrations from the Northeast Shoreline Area to the southern end of the island. Three locations will be reference locations from either Potter Cove or Cranston Cove.

Reference locations for ecological sampling will be selected from the six samples collected as part of the first step sediment sampling presented in Step 7.2. The selections will be made based on similarities of substrate to the site sample stations. The reference locations with the more similar substrate as deteremined by grain size analysis will be selected to represent reference conditions.

The ecological sediment locations will be distributed as follows (also, see Figure 11-3):

- Stillwater Area – 5 locations
- Northeast Shoreline – 5 locations
- Northwest Shoreline – 5 locations
- Locations previously found to contain elevated concentrations of contaminants – 3 locations (SD304, SD312, and SD317)
- South of Island – 3 locations
- Reference Area (Potter Cove or Cranston Cove, Jamestown) – 3 locations
- Locations to be determined – 6 locations

One sediment sample will be collected from each of these locations for toxicity testing and chemical analysis, for a total of 30 samples (plus field duplicates).

One co-located biota sample will be collected with sediment samples collected in the Stillwater Area, Northeast Shoreline, Northwest Shoreline, South of Island, and the Reference Area, if available, for a total of 24 samples plus field duplicates (biota samples will not be collected at the six locations to be determined). Biota species to be collected will include green crabs (*Carcinus maenas)* or Asian crabs (*Hemigrapsus sanguineus)*, clams (*Mercenaria mercenaria*) (where available) and mussels (*Mytilus edulis*) (if clams are not available). Sample locations are designated for the collection of crab samples. Green crabs will be given preference over Asian crabs. Separate locations will be designated for the collection of clams. If clams are not available, mussels will be collected. Biota samples will be submitted for PCB homologue and metals laboratory analysis.

Each clam, mussel, or crab sample will be co-located with a sediment sample within a 25-foot radius. The number and locations of bivalve and crab samples will be broken down as follows:

- Stillwater Area – 2 clam or mussel samples and 3 crab samples
- Northeastern Shoreline – 2 clam or mussel samples and 3 crab samples
- Northwestern Shoreline - 2 clam or mussel samples and 3 crab samples
- South of Island – 1 clam sample, 1 mussel sample, and 1 crab sample
- Reference location – 3 clam or mussel samples and 3 crab samples

Location and surveying data will be obtained to identify the station and depth of samples collected.

7.4 Tier 2 Sediment Transport Model

Additional sediment samples may be collected as part of the Tier 2 sediment transport model effort. A separate work plan document will be prepared that will contain all specific information on the methodology of the Tier 2 sediment transport model.

SAP Worksheet #12a -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Soil, Sediments, Biota - Field QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Temperature Blank	GRO/DRO/PAH/PCB/Metals	One per cooler	Accuracy/bias/ representativeness	4° C $\pm$ 2° C	S
Trip Blank	GRO	One per cooler	Bias/contamination	No target analytes $\geq$ ½ QL*	S
Rinsate Blank	GRO/DRO/PAH/PCB/Metals	One per 20 samples	Bias /contamination	No target analytes $\geq$ ½ QL*	S
Field Blank	GRO/DRO/PAH/PCB/Metals	One per water source	Bias/contamination	No target analytes $\geq$ ½ QL*	S
Field Duplicates	GRO/DRO/PAH/PCB	One per 10 samples	Precision/comparability	RPD ≤ 50%.If sample results are < 2x PQL, professional judgment is used.	S & A
Field Duplicates	Metals	One per 10 samples	Precision/comparability	RPD ≤ 50%.For results < 5x PQL, absolute difference < 4x PQL	S & A

GRO gasoline range organic
DRO diesel range organic
PAH polycyclic aromatic hydrocarbon
PCB polychlorinated biphenyl
RPD relative percent difference
QL quantitation limit
PQL project quantitation limit
^{o}C degree Celsius

* Except when sample concentration is < QL.

SAP Worksheet #12b -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Soil – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank*	GRO/DRO	One per analytical batch	Bias/contamination	No target compounds ≥ ½ QL	A
Surrogate Standards	GRO/DRO	Each field and QC sample	Accuracy/bias	GRO: BFB 60%-140% DRO: p-terphenyl 60%-140%	A
Laboratory Control Sample	GRO/DRO	One per analytical batch	Bias/contamination	GRO: gasoline spike 60%-140% DRO; DRCS spike: 60%-140%	A
Matrix Spike/Matrix Spike Duplicate	GRO/DRO	One per 20 samples	Accuracy/Precision	Recovery: 60-140% RPD ± 20%	A
All samples	GRO/DRO	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	GRO/DRO	All samples	Data completeness	95% overall	S & A

GRO gasoline range organics
DRO diesel range organics
QC quality control
DRCS diesel fuel number 2 composite sample
RPD relative percent difference
QL quantitation limit

* For common laboratory contaminants, no analytes detected > RL.

SAP Worksheet #12c -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Soil, Sediments (Extent Investigation) – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PAHs (8270C/8270C-SIM)	One per analytical batch	Bias/contamination	No target compounds ≥ QL	A
Surrogate Standards	PAHs (8270C/8270C-SIM)	Each field and QC sample	Accuracy/bias	*8270 soil:* 2-fluorophenol: 25-121% Phenol-d5: 24-113% Nitrobenzene-d5: 23-120% 2-fluorobiphenyl: 30-115% 2,4,6-tribromophenol: 19-122% Terphenyl-d14: 18-137% *8270 SIM soil:* 2-methylnaphthalene-d10 Fluorene-d10 Fluoranthene-d10 All recoveries 20-150%	A
Internal Standards	PAHs (8270C/8270C-SIM)	Each field and QC sample	Accuracy/Bias	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	A
Laboratory Control Sample	PAHs (8270C/8270C-SIM)	One per analytical batch	Bias/contamination	8270 soil: See NEL Organic SOP 3.3.15, pp. 30-31 8270 SIM soil: All analytes 30%-150%	A
Matrix Spike/ Matrix Spike Duplicate	PAHs (8270C/8270C-SIM)	One per 20 samples	Accuracy/Precision	Recoveries for scan and SIM: Same as LCS/LCSD RPDs for scan and SIM: 0-20%	A
All samples	PAHs (8270C/8270C-SIM)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	PAHs (8270C/8270C-SIM)	All samples	Data completeness	95% overall	S & A

PAH polycyclic aromatic hydrocarbon
SIM selected ion monitoring
QL quantitation limit
LCS laboratory control sample
LCSD laboratory control sample duplicate
CCV continuing calibration verification

SAP Worksheet #12d -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Soil, Sediments (Extent Investigation) – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PCB (8082)	One per analytical batch	Bias/contamination	No target compounds ≥ QL	A
Surrogate Standards	PCB (8082)	Each field and QC sample	Accuracy/bias	TCMX: 18-156% DCB: 25-158%	A
Laboratory Control Sample	PCB (8082)	One per analytical batch	Bias/contamination	AR1254: 50-150%	A
Matrix Spike/ Matrix Spike Duplicate	PCB (8082)	One per 20 samples	Accuracy/Precision	Rec AR1254: 50-150% RPD AR1254: 0-50%	A
All samples	PCB (8082)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	PCB (8082)	All samples	Data completeness	95 % overall	S & A

PCB polychlorinated biphenyl
QL quantitation limit
TCMX 2,4,5,6 tetrachlorometaxylene
DCB decachlorobiphenyl
RPD relative percent difference

SAP Worksheet #12e -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Soil, Sediments (Extent Investigation) – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	Metals (6010B/7471A)	One per analytical batch	Bias/contamination	No target metals ≥ QL	A
Laboratory Control Sample	Metals (6010B/7471A)	One per analytical batch	Bias/contamination	All target metals 85-115%	A
Matrix Spike	Metals (6010B/7471A)	One per 20 samples	Accuracy/bias	Recovery: 75-125%	A
Laboratory Duplicates	Metals (6010B/7471A)	One per 20 samples	Precision	+ 20% of the parent sample	A
ICP Serial Dilution	Metals (6010B/7471A)	One per analytical batch	Accuracy/bias	If original result s > 10xMDL an analysis of a 1:5 dilution should agree within +10% of the original value	A
All samples	Metals (6010B/7471A)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	Metals (6010B/7471A)	All samples	Data completeness	95 % overall	S & A

QL quantitation limit
MDL method detection limit
RPD relative percent difference
ICP inductively coupled plasma

SAP Worksheet #12f -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table –Sediments (Ecological Investigation) – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PAHs (8270C-SIM)	One per analytical batch	Bias/contamination	No target compounds ≥ QL	A
Surrogate Standards	PAHs (8270C-SIM)	Each field and QC sample	Accuracy/bias	See following table	A
Internal Standards	PAHs (8270C-SIM)	Each field and QC sample	Accuracy/Bias	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	A
Laboratory Control Sample	PAHs (8270C-SIM)	One per analytical batch	Bias/contamination	See Table 12-1	A
Matrix Spike/ Matrix Spike Duplicate	PAHs (8270C-SIM)	One per 20 samples	Accuracy/Precision	See Table 12-1	A
All samples	PAHs (8270C-SIM)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	PAHs (8270C-SIM)	All samples	Data completeness	95% overall	S & A

PAH polycyclic aromatic hydrocarbon
SIM selected ion monitoring
QL quantitation limit
IS internal standard
CCV continuing calibration verification

Table 12-1
Columbia Analytical Services Quality Control Limits for Organic Laboratory Control Samples and Matrix Spike Samples

Method	Analyte	LCS	MS
8270C	2,6-DIMETHYLNAPHTHALENE	50-150	50-150
8270C	ACENAPHTHENE	39-130	39-130
8270C	ACENAPHTHYLENE	44-130	44-130
8270C	ANTHRACENE	49-130	49-130
8270C	BENZO(A)ANTHRACENE	47-116	47-116
8270C	BENZO(A)PYRENE	27-124	27-124
8270C	BENZO(B)FLUORANTHENE	19-132	19-132
8270C	BENZO(G,H,I)PERYLENE	24-128	24-128
8270C	BENZO(K)FLUORANTHENE	41-123	41-123
8270C	CHRYSENE	45-117	45-117
8270C	DIBENZO(A,H)ANTHRACENE	29-129	29-129
8270C	FLUORANTHENE	51-124	51-124
8270C	FLUORENE	40-130	40-130
8270C	INDENO(1,2,3-CD)PYRENE	40-122	40-122
8270C	NAPHTHALENE	44-130	44-130
8270C	PHENANTHRENE	51-130	51-130
8270C	PYRENE	33-123	33-123
8270C	2-FLUOROBIPHENYL -SURR	23-120	23-120
8270C	NITROBENZENE-d5 -SURR	18-125	18-125
8270C	TERPHENYL-d14 -SURR	19-145	19-145
8270C	1,4-DIOXANE	31-80	31-80
8270C	1-METHYLNAPHTHALENE	50-150	50-150
8270C	2-METHYLNAPHTHALENE	42-130	50-150
8270C	BIS(2-ETHYLHEXYL)PHTHALATE	50-150	50-150
8270C	CARBAZOLE	40-150	40-150
8270C	DIBENZOFURAN	50-150	50-150
8270C	HEXACHLOROBENZENE	50-150	50-150
8270C	NITROBENZENE	50-150	50-150

SAP Worksheet #12g -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table – Sediments (Ecological Investigation), Biota – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PCB Homolog (680)	One per analytical batch	Bias/contamination	No target compounds ≥ QL	A
Surrogate Standards	PCB Homolog (680)	Each field and QC sample	Accuracy/bias	30-150%	A
Internal Standards	PCB Homolog (680)	Each field and QC sample	Accuracy/Bias/Precision	EICP area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	A
Laboratory Control Sample	PCB Homolog (680)	One per analytical batch	Bias/contamination	30-130%	A
Matrix Spike/ Matrix Spike Duplicate	PCB Homolog (680)	One per 20 samples	Accuracy/Precision	Recovery 30-130% RPD 30	A
All samples	PCB Homolog (680)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	PCB Homolog (680)	All samples	Data completeness	95 % overall	S & A

EICP extracted ion current profile
PCB polychlorinated biphenyl
QC quality control
QL quantitation limit

SAP Worksheet #12h -- Measurement Performance Criteria Table

(*UFP-QAPP Manual Section 2.6.2*)

Measurement Performance Criteria Table –Sediments (Ecological Investigation) – Analytical QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	Metals (6010B/7471A)	One per analytical batch	Bias/contamination	No target metals ≥ QL	A
Laboratory Control Sample	Metals (6010B/7471A)	One per analytical batch	Bias/contamination	Dependent on Lot of LCS Standard at time of analysis. Performance Acceptance Limits provided on Manufacturer's Certificate of Analysis. Current lot limits provided in Table 12-2	A
Matrix Spike	Metals (6010B/7471A)	One per 20 samples	Accuracy/bias	Recovery: 75-125%	A
Laboratory Duplicates	Metals (6010B/7471A)	One per 20 samples	Precision	Values ≥ 5xQL: RPD ± 20% Values ≤ 5xQL: Absolute Difference ≤ 2xQL	A
ICP Serial Dilution	Metals (6010B/7471A)	One per analytical batch	Accuracy/bias	If original result is > 50xMDL, the 5xdilution result must agree within ± 10 % of the original value	A
MRL Standard	Metals (6010B/7471A)	Once at the beginning and once at the end of the analytical run	Accuracy/bias	±50% of the true value	A
ICSA (ICP only)	Metals (6010B)	Once at the beginning and once at the end of the analytical run	Accuracy/bias	Results <MRL (<2x MRL for metals with MRL<10mg/L)	A
ICSB (ICP only)	Metals (6010B)	After each ICSA	Accuracy/bias	±20% of the true value	A
HLCCV1 (ICP only)	Metals (6010B)	Once during daily analysis	Accuracy/bias	±10% of the true value	A
HLCCV2 (ICP only)	Metals (6010B)	Once during daily analysis	Accuracy/bias	±10% of the true value	A
All samples	Metals (6010B/7471A)	All samples	Sensitivity	QL < project action limits listed in Worksheet # 15	A
All samples	Metals (6010B/7471A)	All samples	Data completeness	95 % overall	S & A

ICP	inductively coupled plasma
QL	quantitation limit
LCS	laboratory control sample
RPD	relative percent difference
MDL	method detection limit

Table 12-2

Columbia Analytical Services Quality Control Limits for Inorganic Laboratory Control Samples and Matrix Spike Samples

Analyte	LCS Limit
Aluminum	58-142
Antimony	50-150
Arsenic	79-120
Barium	82-118
Beryllium	82-118
Boron	56-144
Cadmium	82-119
Calcium	79-121
Chromium	78-122
Cobalt	82-118
Copper	82-118
Iron	50-150
Lead	81-119
Magnesium	77-123
Manganese	80-120
Molybdenum	79-121
Nickel	82-118
Potassium	70-130
Selenium	75-124
Silver	61-139
Sodium	56-145
Strontium	80-120
Thallium	76-124
Tin	70-130
Titanium	40-160
Vanadium	75-125
Zinc	79-121
Mercury	68-132

SAP Worksheet #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**
Phase I Gould Island RI	RI for Site 17: Building 32, Gould Island, December 2006	TtNUS	Selection of locations for Phase 2 sediment samples	None

SAP Worksheet #14 -- Summary of Project Tasks
(*UFP-QAPP Manual Section 2.8.1*)

The Phase 2 RI field activities consist of the following tasks:

- Mobilize/demobilize and Utility Clearance
- Sample collection supplies
- Conduct drilling and soil sampling for soil investigation
- Conduct sediment sampling for sediment investigation
- Conduct biota and sediment sampling for an ecological investigation,
- Conduct bathymetry mapping and sediment grain size analysis sampling (part of a Tier 2 sediment transport model)
- Decontamination
- IDW removal and disposal
- Analytical Tasks
- Data Management
- Assessment and Oversight
- Data Review
- Project Report

The SOPs and field documents referenced below and in the worksheets are included in Appendices A and B, respectively.

Mobilization/Demobilization and Utility Clearance

Mobilization includes procurement of field equipment, supplies, and subcontractors; mobilization of personnel, subcontractors, equipment, and supplies to the field; coordination with Dig-safe and the NAVSTA Environmental Office, a site walkover, field orientation meetings to prepare for field work, and brush clearing. Mobilization will be coordinated with the NAVSTA Environmental Office a minimum of 1 week in advance of the initiation of field activities. It is anticipated that transportation of equipment and facilities to the island will require a barge and tug boat. The barge contractor may need to repair the ramp area at Gould Island before mobilization efforts begin.

TtNUS will be responsible for obtaining clearance of all underground utilities at all sampling locations at least 72 hours prior to beginning on-site work. TtNUS will conduct a public and Navy Dig-safe clearance of the Site. TtNUS will coordinate with local utility contractors and the NAVSTA Environmental Office,

marking drilling locations on the ground with stakes, pin flags, and white spray paint, visiting the Site to meet with NAVSTA Environmental Office utility marking crews, and making adjustments as needed (to avoid overhead hazards, utility lines, etc). Utility clearance will be performed in accordance with TtNUS SOP HS-1.0. It is anticipated that brush clearing will need to be conducted to clear brush from work areas and provide access paths.

A field orientation meeting will be held prior to beginning the field program. The purpose of this meeting is to review the scope of the field study including site description, objectives of the field investigation, sampling locations, sampling methods, field QC samples, health and safety requirements, NAVSTA protocol and chain-of-command. The field orientation meeting will be attended by the field staff, project manager, lead chemist, and health and safety officer. Field team members will review this SAP, applicable SOPs (Appendix C), and applicable Field Documentation Forms (Appendix D) and they will document that they have read the QAPP by signing Worksheet #4. All subcontractors working at the Site will be provided with the site-specific health and safety plan, as well as their specifications and scopes of work.

Portable sanitary facilities, vehicles, and supply storage containers will be procured for the Site. In addition, a box truck will be procured for storage of supplies needed for drilling operations and ecological investigations that are not conducted on board vessels. Drillers will bring a drill rig, support truck, and a water trailer to the Site for decontamination purposes. Drums (55-gallon) will be brought to the site to hold drill cuttings. It is assumed that it will be acceptable to store drums outside and not require a storage box at this site.

Demobilization includes removing field equipment and supplies from the Site, returning rented equipment, managing IDW, performing general site cleanup, organizing and finalizing field paperwork, and entering field records/data into the Site database.

General Sample Collection Provisions – Sample design and rationale is presented in Worksheet 17. Worksheet 19 lists the types of sample containers, preservatives, and holding times for each sample matrix and analysis. The sample collection documentation, handling, tracking, and custody procedures are presented in Worksheet 27.

Drilling and Soil Sampling Procedures

Borings will be advanced to refusal or bedrock (anticipated to be relative shallow) using hollow stem auger (HSA) drilling techniques. Continuous split-barrel samples will be retrieved at 2-foot intervals, and surface and subsurface samples will be sent for laboratory analysis.

Drilling will be performed by a subcontractor according to the procedures in SOP GH-1.3. All down-hole drilling equipment will be steam-cleaned before use at each boring. Borings will be advanced using HSA drilling techniques. Continuous split-barrel samples will be retrieved at 2-foot intervals throughout the length of the boring using a 2-inch ID, stainless steel split-barrel sampler, and all required information will be recorded on the boring log sheet (Appendix D) according to SOP GH-1.5. The bedrock depth encountered during the Phase 1 RI ranged from 7 to 15 feet below ground surface (bgs) at the southeast portion of the Site. The bedrock depth in the north portion of the Site is believed to be up to approximately 50 feet bgs. It is presumed that most of the overburden is a dense till, as evidenced by borings conducted during the Phase 1 RI. No bedrock coring will be conducted.

Soil samples will be collected in accordance with SOP SA-1.3. As the split-barrel samplers are retrieved from the borehole, they will be opened, visually inspected, and scanned for VOCs using a photo-ionization detector (PID) according to SOP ME-12 or a flame-ionization detector (FID) according to SOP ME-15. For samples collected from the PCB release area, which will be analyzed for PCBs, PAHs, and metals, two sample intervals will be collected: 0 to 6 inches and the second at a deeper interval not to exceed 19.5 to 20 feet. The limit placed on the deep sample interval is based on the maximum depth contaminated subsurface soil may be potentially be impacting marine sediments in the Stillwater Area. The sampling procedures below for “non-volatile parameters” will then be followed.

For samples collected near the former coal pile area, which will be analyzed for PAHs, TAL metals, DRO, and GRO, four sample intervals will be collected: 0 to 6 inches, 12 to 18 inches, 24 to 30 inches, and 42 to 48 inches.

Soil Sampling Procedures for GRO Laboratory Samples (Grab)

Each soil sample for GRO analysis will be collected using a cut syringe or equivalent device and placed in a methanol-preserved vial with a septa cap, according to the SW-846 Method 5035A (July 2002). The methanol-preserved vial will be maintained at 4°C for up to 14 days. The following procedure should be followed for the soil GRO sample collection:

1. Label two 40-mL amber vials containing 5 mL of methanol with the sample location number and a bottle letter such as A, B.

2. Collect approximately 5 grams of sample by coring or stabbing the soil with a 10-mL pre-cut syringe. If non-aqueous phase liquids (NAPL) is noted within the soils, then a reduced volume of approximately 1 to 2 grams should be collected as a separate "medium concentration" (NAPL) sample. Extrude the sample into the 40-mL GRO vial. The soil must be immersed in the methanol; re-collect the sample using a smaller volume if necessary. Avoid touching the threads on the vial's neck or loss of methanol by evaporation. Cap the vial and invert it several times to mix the sample.

3. Weigh each sample vial to the nearest 0.01 gram and record the weight on the field log sheet. Repeat the sample collection procedure for the second vial. Pack and ship to the laboratory. Include the field log sheet containing the sample weight information with the samples.

Field duplicate samples will also be collected. Following the collection of the first set of GRO containers, collect the field duplicate from the same sampling interval. No additional vials are necessary for laboratory QC samples.

<u>Soil Sampling Procedures for Non-Volatile Parameters</u>

1. Record all required data on the boring log, which will also serve as the soil sample log sheet (Appendix D). Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. Use the boring log to record soil descriptions, depth of strata changes, and sample depth intervals. Classify the soil sample visually using the Unified Soil Classification System (ASTM D-2488-98).

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Transfer the soil from the split-barrel sampler to a decontaminated stainless-steel bowl using only decontaminated stainless steel trowels, and homogenize the sample.

4. If there is insufficient sample volume to fill all the containers for analyses, an equal amount of material from the intervals immediately above and below the selected sample interval may be used to supplement the composite sample to ensure sufficient sample quantity for all analyses.

5. Remove any large particles such as twigs, gravel or artificial fill too large to be sent for analysis. Note the removal of material on the boring log.

6. Fill the appropriate sample containers with the soil sample.

7. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.

8. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the chain-of-custody procedures described in Worksheet 27 are followed. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.

9. Decontaminate the sampling equipment before reuse.

Care should be taken in handling all soil samples to ensure that the exterior of the sample containers are clean and free of soils before shipping to the laboratory.

Extent of Contamination Sediment Sampling Procedures

Marine sediment sampling at the Site will be conducted in accordance with SOP SA-1.2 by a subcontractor assisted and overseen by TtNUS. Samples will be collected in the Stillwater area up to a maximum depth of 4 feet. Sediment samples collected from the northeast shoreline and northwest shoreline will be collected from 0 to 6 inches below the sediment surface.

Samples from the Stillwater Area will be collected with a vibracore device mounted on a barge. Cores will be advanced to a maximum depth of 4 feet below sediment surface or refusal, whichever is encountered first. Samples will be collected from four intervals: 0 to 6 inches, 6 to 12 inches, 18 to 24 inches, and 42 to 48 inches.

For stations where surficial samples will be collected, samples will be collected by a diver using an acetate lined coring device and/or a Smith-MacIntyre grab sampler (or equivalent) device capable of providing adequate quantities of sediment from that interval. Sampling device selection will be based on sediment type (i.e. soft, hard), sampling efficiency for sediment type, and required volume of sediment needed for analyses.

When core tubes are selected, each will be fitted with a cutting head and core catcher if needed. The cores will be driven by hand or pneumatic hammer to achieve the sampling depth. Six-inch and/or 1-foot long acetate tubes will be used to ensure that excess sea water is not gathered along with the sediment sample. The sediment top and bottom of the core tubes will be labeled, and they will be stored upright until recontainerization or until samples are composited. If refusal is encountered at any depth above the target sample depth for that location, the sampler will move to a suitable location. When possible, the sampler will minimize the horizontal distance from the original sampling point when moving to obtain a sample. If refusal above the target depth is encountered after four attempts, then the samples from the core with the maximum depth achieved will be selected for analysis.

As each of the samples for a given depth at a particular station is brought to the surface, the contents will be transferred into a clean, stainless-steel mixing bowl in order to form a single composite sample for each sampling location. Large stones and shells and other similar objects too large to be analyzed and not representative of the desired sample will be removed and discarded. The remaining material will be slowly decanted and then mixed until homogeneous and transferred to the sample containers for shipment to the laboratory. The laboratory will be instructed to decant any additional water before sample preparation.

Water depths at sample stations will be recorded and normalized to tide to provide elevations of the ground surface in the underwater portion of the Site. Adjustments to tide will be accomplished by referencing local tide gauging stations at Newport, and subtracting the tide height from the water depth at the given time. A written report will be provided from the subcontractor which describes the sediment conditions at each of the sample stations, coordinates (horizontal and vertical) of the sample stations, and the velocity and direction of the currents encountered relative to the tide cycle at the seafloor at each of the sample stations.

It is expected that the majority of sediment samples will be collected from areas with water depths between 5 and 30 feet. Some locations may be collected from areas with water depths less that 5 feet or more than 30 feet

Ecological Investigation Sediment Sampling Procedures

Sediment samples for the ecological investigation will be collected in the same manner as the surficial extent of contamination sediment samples.

Biota Sampling Procedures

Biota samples will target the following species: hardshell clams (*Mercenaria mercenaria*), blue mussels (*Mytilus edulis*) (if clams are not available), green crabs (*Carinus maenas*), and Asian crabs (*Hemigrapsus sanguineus*) (if green crabs are not available). Bivalve samples will be collected by divers and crab traps will be stationed and baited for a period of one week to collect green crabs. Locations where biota samples are to be collected will be based on the analytical results from the previous sediment sample collection effort. Biota samples will be collected from a selection of sediment sample locations that exhibit a concentration gradient from high, medium, and low contaminant concentrations.

A minimum of 10 bivalves and crabs will be required for each sample, regardless of species. A sample group of 20 individuals within a single size range is optimal. All collected biota samples will be characterized according to species, length and total weight. For bivalve samples, shell length will be measured using dial calipers in millimeters. For crab samples, carapace width will be measured using dial calipers in millimeters (to the nearest whole millimeter). Biota samples will be analyzed for PCB homologues, TAL metals, and percent lipids.

- Bivalves/crabs selected for analysis will be cleaned with a brush and local bay seawater to remove sediment and other material on the shell. Samples will be identified by species and size, and all shells of the same species and size range placed with shells intact into one plastic bag or plastic container per sample station. One sample consisting of multiple organisms will be collected per sampling station. Biota samples will be shipped overnight to NEL laboratory in sealed plastic bags, cooled to 4 degrees C. The laboratory will homogenize biota samples prior to analysis using the following procedure as per the National Status and Trends Mussel Watch Program (NOAA, 1993):

- Remove the tissue from all bivalves for one individual sample and composite. Crab samples will be processed with the shell intact. Any liquid residue in the biota sample containers will also be processed with the samples.

- Homogenize each composite biota sample using a stainless steel tissue mixer and transfer to a clean sample container for PCB congeners, percent lipids, and metal analyses.

- Decontaminate sample processing equipment prior to processing each sample to avoid cross contamination.

Quality Control Tasks – Field QC samples will be collected as part of the investigation, including field duplicates, trip blanks, equipment rinsate blanks, and field blanks. Also, samples will be assigned and additional sample volume collected for the laboratory to perform matrix spike analysis. Worksheet #20 presents the field QC sample summary.

- Field QC Samples

  Field duplicates are used to assess the combined field and laboratory precision. Field duplicates for the samples will be collected by alternately filling sample bottles from the source being sampled. Field duplicate samples will be collected at a frequency of one for every 10 or fewer investigative samples. Field duplicate samples will be shipped blind to the laboratories.

  Trip blanks are used to assess the potential for cross-contamination during shipment and storage of GRO samples. Trip blanks associated with the solid samples for GRO analysis will consist of one 40-mL VOC vial containing 5 mL of methanol. Trip blanks will be prepared at the rate of one per cooler shipment.

  Equipment rinsate blanks are used to assess the effectiveness of decontamination procedures. Rinsate blanks are obtained under representative field conditions by running analyte-free deionized/distilled water (provided by the laboratory) through sample collection equipment after decontamination and prior to use, and placing it in the appropriate sample containers for analysis. Rinsate blanks will be collected at a frequency of 1 for every 20 or fewer investigative samples on each piece of decontaminated equipment.

  Field blanks are used to determine the quality of the source water used for decontamination. These samples will consist of the contaminant-free deionized water used in the last step of decontamination. At a minimum, field blanks will be prepared at the rate of one per sampling event or one per source.

- Laboratory QC Samples

  Laboratory matrix spikes provide information about the effect of the sample matrix on the preparation and measurement methodology. Matrix spikes for organic analyses are performed in duplicate and are hereinafter referred to as MS/MSD samples. A single MS analysis will be performed for inorganic parameters. The laboratory will perform the MS/MSD and laboratory duplicate analyses at a frequency of one for every 20 or fewer investigative samples per sample

matrix. The field sampler will assign samples for MS/MSD analysis at this frequency on the chain-of-custody form and sample log sheet. No extra volume for soil samples and double volume for sediment samples (due to high percent moisture content) should be collected.

Laboratory Control Samples provide information about the condition of the analytical process under ideal conditions and without introduction of variables associated with the environmental samples to be analyzed. LCSs will be analyzed prior to the start of routine analyses to verify that the analytical processes are under control.

Decontamination Procedures

All non-disposable equipment that comes in contact with the sample medium will be decontaminated to prevent cross-contamination between sampling points. This includes equipment such as stainless steel bowls, scoops, as well as heavy equipment. Personnel decontamination is discussed in the HASP.

Standard Operating Procedures for decontamination are provided in the TtNUS SOP SA-7.1 (Appendix C). All heavy equipment, including the drilling rig, rods and augers, and other downhole equipment used during site investigation activities, will be decontaminated prior to beginning work and between all boreholes using a high-pressure steam wash. The water to be used for steam-cleaning will be from a potable source.

Investigation Derived Waste Management

It is anticipated that waste materials will be generated during the field investigation. These materials include:

- Decontamination fluid;
- Used personal protective equipment (PPE);
- Used sampling equipment;
- Drill cuttings and excess soil samples;

Investigation-derived waste will be managed as described below, in accordance with RIDEM regulations (Rules and Regulations for Hazardous Waste Management, DEM OWM-HW01-07).

Visibly clear phosphate-free detergent wash water and rinse water decontamination fluids from sampling equipment will be released to the ground upon rinsing, in the immediate vicinity of its point of generation. The decontamination rinse water will be contained in 55-gallon drums or bulk containers.

Used PPE, such as sampling gloves, Tyvek coveralls, paper towels, or other materials will be bagged and sealed prior to disposal as general refuse. If PPE becomes grossly contaminated, it will be segregated from other PPE, labeled and staged as "contaminated material." Contaminated material will be drummed and staged in the IDW area designated by the Navy. TtNUS will arrange for off-site disposal of drums by a licensed waste hauler at an approved facility.

Used disposable sampling equipment, which generally has minor contamination, will be disposed of with the PPE as general refuse. Grossly contaminated disposable equipment, as determined at the discretion of the sampler, will require segregation from other equipment and proper disposal.

Drill cuttings and excess soil samples will be contained in 55-gallon drums or bulk containers and staged at a secure area. Any drums used for storage will be clearly marked with a grease pencil or other water-resistant marker to indicate the borehole from which the cuttings were removed. The word "soil" will be used to differentiate between drummed cuttings and drummed purge water or development water. The drums or bulk containers will be staged in an orderly fashion, with proper spacing, in an area designated by the Navy. Composite samples will be collected from the filled drums to characterize the waste generated. Data from the analysis of these composite samples will be used for characterization of the materials, and, based on these analyses, the material will be manifested for shipment off-site to a disposal facility. Waste pickup and disposal will be conducted in coordination with the NAVSTA waste management office, who takes ownership of the waste after characterization.

TtNUS will be responsible for arranging the removal and proper disposal of all accumulated waste materials following completion of the Phase 2 RI program. Manifests and shipping papers will be signed by a representative of the NAVSTA waste management office. Disposal will be arranged with licensed waste haulers at approved receiving facilities. Characterization analyses will be conducted by the waste disposal subcontractor.

Analytical Tasks

Chemical analysis for soil samples (GRO, DRO, PAHs, PCBs, and metals) and extent of contamination sediment samples (PAHs, PCBs, and metals) will be performed by a subcontracted laboratory, Mitkem Laboratories Services, Inc. (NEL). NEL is approved by the Naval Facilities Engineering Service Center

(NFESC). Analyses will be performed in accordance with the analytical method identified Worksheet #23 and the requirements of the technical specifications for laboratory services developed by TtNUS for this work (Appendix E). The subcontracted laboratory will meet the PQLs specified in Worksheet #15.

The technical specification details the analytical requirements, number of samples, matrix, analytical method to be performed, sample containers, preservatives, holding times, the quantitation limits required for the project, and data deliverables. NEL will perform the chemical analyses following laboratory-specific SOPs developed based on the methods listed in Worksheet #23. Copies of the SOPs are included in Appendix E on the attached CD.

The laboratory QC sample Method/SOP acceptance limits to be met for each analysis listed in Worksheet #28. Instrument calibration will meet the QC criteria required by the EPA Methods. The procedures and acceptance criteria for laboratory analytical instrument calibration are presented in Worksheet #24, and for maintenance and inspection in Worksheet #25.

<u>Data Management</u>

- Project documentation and records
  - Field sample collection and field measurement records - See Worksheets #27 and #29.
  - Laboratory data package deliverables - See Worksheet #30.
  - Data assessment documents and records – See Worksheet #29.

- Data recording is described in Worksheet #27.

- Data handling and management - After the field investigation is completed, the field sampling log sheets will be organized by date and media 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 the laboratories will meet the requirements of the technical specifications. The electronic data results will be automatically downloaded into the TtNUS database in accordance with proprietary TtNUS processes.

- Data tracking and control. The TtNUS Project Manager (or designee) is responsible for the overall tracking and control of data generated for the project.

  - Data Tracking. Data is tracked from its generation to its archiving in the TtNUS project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contract laboratories. Upon receipt of the data packages from the analytical laboratories, the 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 the analytical laboratory.

  - Data Storage, Archiving, and Retrieval. The data packages received from the subcontract laboratories are tracked in the data validation log book. After the data are validated, the data packages are entered into the TtNUS 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 FOL to be entered into the 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 TtNUS.

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

Assessment and Oversight – See Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

Data Review

- Data verification – See Worksheet #34
- Data validation – See Worksheets #35 and #36
- Usability assessment – See Worksheet #37

Project Report

Draft and Final versions of project reports will be prepared and submitted to the Navy, EPA, and RIDEM for review. The reports will include the following sections:

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

- Introduction and Background – includes a description of the history of operations and activities at the Site and a summary of previous investigations and removal actions.

- Description of Field Investigations – includes a summary of the work planned and described in this SAP and any field modification as documented by the field geologist and engineers. This section will include maps showing the sampling station locations and well installations and tables summarizing the data collected.

- Data quality – includes a summary of quantitative analytical performance indicators such as completeness, precision, bias and sensitivity, as well as qualitative indicators such as representativeness and comparability. Also includes a reconciliation of project data with the DQOs and an identification of deviations from this SAP. Minimum requirements follow:

    - Precision for field duplicate samples and laboratory duplicates such as MS/MSD or LCS/LCSD pairs will be computed as follows:

$$RPD = \frac{(\mathrm{Re}sult1 - \mathrm{Re}sult2)}{(\mathrm{Re}sult1 + \mathrm{Re}sult2)} x200\%$$

    - Accuracy for matrix spikes and LCSs will be computed in terms of percent recoveries as follows:

$$LCSPercent\mathrm{Re}covery = \frac{Actual\mathrm{Re}sult}{Expected\mathrm{Re}sult} x100\%$$

$$MatrixSpikePercent\mathrm{Re}covery = \frac{SSR - SR}{S} x100\%$$

where: SSR = Spiked Sample Result
SR = Sample Result
S = Spike Concentration

    - Bias will be compute as 100% - Percent Recovery.

- Completeness will be estimated as the number of samples of each population that were collected versus the targeted number, as well as the total number of valid results for each chemical compared to the total number of possible valid results. These quantities will be computed as follows:

$$SampleCompleteness = \frac{Number\ of\ Samples\ Analyzed}{Number\ of\ Samples\ Targeted\ for\ Analysis} x100\%$$

$$Analytical\ Completness = \frac{(Number\ of\ Possible\ Valid\ Results - Number\ of\ Rejected\ Results)}{Number\ of\ Possible\ Valid\ Results} x100\%$$

where rejected results are those results that are rejected during validation (the impact of rejected data will be evaluated during data usability assessment).

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 #37.

- Physical Features of the Site – this section will describe the topographical features of the Site augmenting the information provided in the Phase 1 report. Particularly, this section will show the sediment topography seaward of the island shoreline and the types of sediment present, as well as physical features found on shore at the south west corner of the site. Additional descriptions of ecological setting of the Site will be provided.

- Nature of Contamination – includes the contamination detected in each medium sampled in relation to the conceptual model of the site. This section will resolve the extent of the contamination in sediments near outfalls and in the Stillwater areas at the north of the Site. Also the extent of the petroleum contamination at the southwest corner of the site will be discussed. Detected contaminant concentrations will be tabulated for each medium and depicted on maps.

- Contaminant Fate and Transport – includes a description of the contaminants detected and their behavior in the soil and sediment, particularly with emphasis on the future migration of these contaminants either through the food chain or other medium.

- Baseline Ecological Risk Assessment (BERA) – the BERA will included an evaluation of the data collected during the Phase 2 investigation as well as data previously collected at the site. As part of the BERA, plots showing the results of the toxicity testing will be generated, as well as figures presenting the benthic sampling results. Tables and figures will represent the data points of the chemical concentrations that are most significant risk drivers.

- Summary and Conclusions – this section will summarize the findings, describe the need for additional ecological assessments, identify additional data collection needs, and conclude if a Remedial Investigation will be required to protect receptors.

TtNUS will develop response to comments received on the draft reports. TtNUS will submit the draft reports before the next sampling begins. The final version of the report will submitted in hardcopy and electronic format to the project stakeholders.

SAP Worksheet #15a -- Reference Limits and Evaluation Table

Matrix: Soil Samples

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
PAHs						
2-Methylnaphthalene	91-57-6	10000	RIDEM-DEC	0.330	0.330	0.071
Acenaphthene	83-32-9	10000	RIDEM-DEC	0.330	0.330	0.057
Acenaphthylene	208-96-8	10000	RIDEM-DEC	0.330	0.330	0.062
Anthracene	120-12-7	10000	RIDEM-DEC	0.330	0.330	0.075
Benzo(a)anthracene	56-55-3	7.8	RIDEM-DEC	0.330	0.330	0.049
Benzo(a)pyrene	50-32-8	0.8	RIDEM-DEC	0.330	0.330	0.041
Benzo(b)fluoranthene	205-99-2	7.8	RIDEM-DEC	0.330	0.330	0.054
Benzo(g,h,i)perylene	191-24-2	10000	RIDEM-DEC	0.330	0.330	0.046
Benzo(k)fluoranthene	207-08-9	78	RIDEM-DEC	0.330	0.330	0.050
Chrysene	218-01-9	780	RIDEM-DEC	0.330	0.330	0.045
Dibenzo(a,h)anthracene	53-70-3	0.8	RIDEM-DEC	0.330	0.330	0.048
Fluoranthene	206-44-0	10000	RIDEM-DEC	0.330	0.330	0.065
Fluorene	86-73-7	10000	RIDEM-DEC	0.330	0.330	0.072
Indeno(1,2,3-c,d)pyrene	193-39-5	7.8	RIDEM-DEC	0.330	0.330	0.050
Naphthalene	91-20-3	10000	RIDEM-DEC	0.330	0.330	0.068
Phenanthrene	85-01-8	10000	RIDEM-DEC	0.330	0.330	0.073
Pyrene	129-00-0	10000	RIDEM-DEC	0.330	0.330	0.058
TAL Metals						
Aluminum	7429-90-5	--	-	20	20	2.22
Antimony	7440-36-0	820	RIDEM-DEC	6	6	0.73
Arsenic	7440-38-2	7.0	RIDEM-DEC	1	1	0.35
Barium	7440-39-3	10000	RIDEM-DEC	20	20	0.22
Beryllium	7440-41-7	1.3	RIDEM-DEC	0.5	0.5	0.030
Cadmium	7440-43-9	1000	RIDEM-DEC	0.5	0.5	0.06
Calcium	7440-70-2	--	-	500	500	10.95
Chromium	7440-47-3	10000[3]	RIDEM-DEC	1	1	0.17
Cobalt	7440-48-4	--	-	5	5	0.32
Copper	7440-50-8	10000	RIDEM-DEC	3.4	3.4	1.69

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
Iron	7439-89-6	--	-	10	10	2.39
Lead	7439-92-1	500	RIDEM-DEC	1	1	0.18
Magnesium	7439-95-4	--	-	500	500	7.04
Manganese	7439-96-5	10000	RIDEM-DEC	1.5	1.5	0.15
Mercury	7439-97-6	610	RIDEM-DEC	0.1	0.1	0.0064
Nickel	7440-02-0	10000	RIDEM-DEC	4	4	0.70
Potassium	7440-09-7	--	-	500	500	4.26
Selenium	7782-49-2	10000	RIDEM-DEC	3.5	3.5	0.44
Silver	7440-22-4	10000	RIDEM-DEC	1	1	0.12
Sodium	7440-23-5	--	-	500	500	37.0
Thallium	7440-28-0	140	RIDEM-DEC	2.5	2.5	0.36
Vanadium	7440-62-2	10000	RIDEM-DEC	5	5	0.23
Zinc	740-66-6	10000	RIDEM-DEC	6	6	2.77
PCBs						
Aroclor-1016	12674-11-2	10[4]	RIDEM-DEC	0.033	0.033	0.0015
Aroclor-1221	11104-28-2	10[4]	RIDEM-DEC	0.033	0.033	0.0075
Aroclor-1232	11141-16-5	10[4]	RIDEM-DEC	0.033	0.033	0.010
Aroclor-1242	53469-21-9	10[4]	RIDEM-DEC	0.033	0.033	0.011
Aroclor-1248	12672-29-6	10[4]	RIDEM-DEC	0.033	0.033	0.0081
Aroclor-1254	11097-69-1	10[4]	RIDEM-DEC	0.033	0.033	0.0027
Aroclor-1260	11096-82-5	10[4]	RIDEM-DEC	0.033	0.033	0.0011
Total PCBs	--	10	RIDEM-DEC	0.033	0.033	--
Petroleum Hydrocarbons						
GRO	-	2500	RIDEM-DEC	250	250	0.67
DRO	-	2500	RIDEM-DEC	250	250	0.18

1. The project action limits for all chemicals except GRO and DRO represent the Rhode Island Department of Environmental Management (RIDEM) Industrial/Commercial Direct Exposure Criteria. For GRO and DRO, the action limit represents the RIDEM Method 1 Industrial/Commercial TPH Direct Exposure Criterion. Source: RIDEM (2004).
2. QLs and MDLs from Mitkem Laboratories Services
3. Criterion is for "Chromium III"
4. Criterion is for "Polychlorinated biphenyls (PCBs)

mg/Kg - milligrams per kilogram (dry weight)
PCBs - Polychlorinated biphenyls
GRO – gasoline range organics
DRO – Diesel range organics
- - Criterion not available

SAP Worksheet #15b -- Reference Limits and Evaluation Table

(*UFP-QAPP Manual Section 2.8.1*)

Matrix: Sediment Samples -- Extent Investigation

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
PAHs						
2-Methylnaphthalene	91-57-6	0.07	ER-L[3]	0.0064	0.0064	0.00317
Acenaphthene	83-32-9	0.016	ER-L[3]	0.0075	0.0075	0.00376
Acenaphthylene	208-96-8	0.044	ER-L[3]	0.008	0.0080	0.00391
Anthracene	120-12-7	0.0853	ER-L[3]	0.0033	0.0033	0.00296
Benzo(a)anthracene	56-55-3	0.261	ER-L[3]	0.0033	0.0033	0.00186
Benzo(a)pyrene	50-32-8	0.43	ER-L[3]	0.0033	0.0033	0.00120
Benzo(b)fluoranthene	205-99-2	1.8	AET[5]	0.0033	0.0033	0.00230
Benzo(g,h,i)perylene	191-24-2	0.67	AET[5]	0.0033	0.0033	0.00180
Benzo(k)fluoranthene	207-08-9	1.8	AET[5]	0.0033	0.0033	0.00195
Chrysene	218-01-9	0.384	ER-L[3]	0.0033	0.0033	0.00133
Dibenzo(a,h)anthracene	53-70-3	0.0634	ER-L[3]	0.0033	0.0033	0.00141
Fluoranthene	206-44-0	0.6	ER-L[3]	0.012	0.012	0.00552
Fluorene	86-73-7	0.019	ER-L[3]	0.0033	0.0033	0.00200
Indeno(1,2,3-c,d)pyrene	193-39-5	0.6	AET[5]	0.0033	0.0033	0.00144
Naphthalene	91-20-3	0.16	ER-L[3]	0.0033	0.0033	0.00121
Phenanthrene	85-01-8	0.24	ER-L[3]	0.0033	0.0033	0.00287
Pyrene	129-00-0	0.665	ER-L[3]	0.0033	0.0033	0.00326

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
TAL Metals						
Aluminum	7429-90-5	18000	AET[5]	20	20	2.22
Antimony	7440-36-0	2	ER-L[4]	1.5	1.5	0.73
Arsenic	7440-38-2	8.2	ER-L[3]	1	1	0.35
Barium	7440-39-3	48	AET[5]	20	20	0.22
Beryllium	7440-41-7	-	-	0.5	0.5	0.030
Cadmium	7440-43-9	1.2	ER-L[3]	0.5	0.5	0.06
Calcium	7440-70-2	-	-	500	500	10.95
Chromium	7440-47-3	81	ER-L[3]	1	1	0.17
Cobalt	7440-48-4	10	AET[5]	5	5	0.32
Copper	7440-50-8	34	ER-L[3]	3.4	3.4	1.69
Iron	7439-89-6	220000	AET[5]	10	10	2.39
Lead	7439-92-1	46.7	ER-L[3]	1	1	0.18
Magnesium	7439-95-4	-	-	500	500	7.04
Manganese	7439-96-5	260	AET[5]	1.5	1.5	0.15
Mercury	7439-97-6	0.15	ER-L[3]	0.1	0.1	0.0064
Nickel	7440-02-0	20.9	ER-L[3]	4	4	0.70
Potassium	7440-09-7	-	-	500	500	4.26
Selenium	7782-49-2	1	AET[5]	0.88	0.88	0.44
Silver	7440-22-4	0.5	LEL[6]	0.3	0.3	0.12
Sodium	7440-23-5	-	-	500	500	37.0
Thallium	7440-28-0	-	-	2.5	2.5	0.36
Vanadium	7440-62-2	57	AET[5]	5	5	0.23
Zinc	7440-66-6	150	ER-L[3]	6	6	2.77

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
PCBs						
Aroclor-1016	12674-11-2	0.0227	ER-L[3,7]	0.01	0.01	0.00149
Aroclor-1221	11104-28-2	0.0227	ER-L[3,7]	0.01	0.01	0.00750
Aroclor-1232	11141-16-5	0.0227	ER-L[3,7]	0.01	0.01	0.000827
Aroclor-1242	53469-21-9	0.0227	ER-L[3,7]	0.01	0.01	0.000889
Aroclor-1248	12672-29-6	0.0227	ER-L[3,7]	0.016	0.016	0.00812
Aroclor-1254	11097-69-1	0.0227	ER-L[3,7]	0.01	0.01	0.00273
Aroclor-1260	11096-82-5	0.0227	ER-L[3,7]	0.01	0.01	0.00115
Total PCBs	--	0.0227	ER-L[3,7]	0.01	0.01	--

Notes:

1. The project action limit represents the selected ecological benchmark
2. QLs and MDLs from Mitkem Laboratories
3. ER-L – Effects-Range Low (Long, et al., 1995)
4. ER-L – Effects-Range Low (Long and Morgan, 1991)
5. AET – Apparent Effects Threshold (Buchman, 2006
6. LEL – Lowest Effects Level (Persaud, et al., 1993)
7. Total PCB value

mg/Kg - milligrams per kilogram (dry weight)
PCBs - Polychlorinated biphenyls
- -Criterion not available

SAP Worksheet #15c -- Reference Limits and Evaluation Table

Matrix: Sediment Samples -- Ecological Investigation

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
PAHs						
2-Methylnaphthalene	91-57-6	0.07	ER-L[3]	0.0033	0.0033	0.0011
Acenaphthene	83-32-9	0.016	ER-L[3]	0.0033	0.0033	0.00069
Acenaphthylene	208-96-8	0.044	ER-L[3]	0.0033	0.0033	0.00089
Anthracene	120-12-7	0.0853	ER-L[3]	0.0033	0.0033	0.00087
Benzo(a)anthracene	56-55-3	0.261	ER-L[3]	0.0033	0.0033	0.0018
Benzo(a)pyrene	50-32-8	0.43	ER-L[3]	0.0033	0.0033	0.0013
Benzo(b)fluoranthene	205-99-2	1.8	AET[5]	0.0033	0.0033	0.0013
Benzo(g,h,i)perylene	191-24-2	0.67	AET[5]	0.0033	0.0033	0.0015
Benzo(k)fluoranthene	207-08-9	1.8	AET[5]	0.0033	0.0033	0.00093
Chrysene	218-01-9	0.384	ER-L[3]	0.0033	0.0033	0.0017
Dibenzo(a,h)anthracene	53-70-3	0.0634	ER-L[3]	0.0033	0.0033	0.00098
Fluoranthene	206-44-0	0.6	ER-L[3]	0.0092	0.0092	0.0046
Fluorene	86-73-7	0.019	ER-L[3]	0.0033	0.0033	0.00076
Indeno(1,2,3-c,d)pyrene	193-39-5	0.6	AET[5]	0.0033	0.0033	0.0012
Naphthalene	91-20-3	0.16	ER-L[3]	0.0033	0.0033	0.0018
Phenanthrene	85-01-8	0.24	ER-L[3]	0.0033	0.0033	0.0031
Pyrene	129-00-0	0.665	ER-L[3]	0.0033	0.0033	0.0028
TAL Metals						
Aluminum	7429-90-5	18000	AET[5]	20	10	4.1
Antimony	7440-36-0	2	ER-L[4]	2	2	0.80
Arsenic	7440-38-2	8.2	ER-L[3]	1	1	0.27
Barium	7440-39-3	48	AET[5]	20	2	0.51
Beryllium	7440-41-7	-	-	0.5	0.5	0.017

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
Cadmium	7440-43-9	1.2	ER-L[3]	0.5	0.5	0.017
Calcium	7440-70-2	-	-	500	100	13.5
Chromium	7440-47-3	81	ER-L[3]	1	1	0.048
Cobalt	7440-48-4	10	AET[5]	5	5	0.021
Copper	7440-50-8	34	ER-L[3]	2.5	2	0.13
Iron	7439-89-6	220000	AET[5]	10	10	0.92
Lead	7439-92-1	46.7	ER-L[3]	1	0.5	0.15
Magnesium	7439-95-4	-	-	500	100	2.7
Manganese	7439-96-5	260	AET[5]	1.5	1	0.31
Mercury	7439-97-6	0.15	ER-L[3]	0.1	0.02	0.0021
Nickel	7440-02-0	20.9	ER-L[3]	4	4	0.043
Potassium	7440-09-7	-	-	500	200	100
Selenium	7782-49-2	1	AET[5]	1	1	0.29
Silver	7440-22-4	0.5	LEL[6]	0.5	1*	0.089
Sodium	7440-23-5	-	-	500	100	21.9
Thallium	7440-28-0	-	-	2.5	1	0.3
Vanadium	7440-62-2	57	AET[5]	5	5	0.035
Zinc	7440-66-6	150	ER-L[3]	6	2	0.32
PCB Homologs						
Monochlorobiphenyls	27323-18-8	0.0227	ER-L[3,7]	0.002	0.002	0.000042
Dichlorobiphenyls	25512-42-9	0.0227	ER-L[3,7]	0.002	0.002	0.000048
Trichlorobiphenyls	25323-68-6	0.0227	ER-L[3,7]	0.002	0.002	0.00037
Tetrachlorobiphenyls	26914-33-0	0.0227	ER-L[3,7]	0.002	0.002	0.00013
Pentachlorobiphenyls	25429-29-2	0.0227	ER-L[3,7]	0.002	0.002	0.00015
Hexachlorobiphenyls	26601-64-9	0.0227	ER-L[3,7]	0.002	0.002	0.00011
Heptachlorobiphenyls	28655-71-2	0.0227	ER-L[3,7]	0.002	0.002	0.00012
Octachlorobiphenyls	31472-83-0	0.0227	ER-L[3,7]	0.002	0.002	0.000094
Nonachlorobiphenyls	53742-07-7	0.0227	ER-L[3,7]	0.002	0.002	0.00014
Decachlorobiphenyl	2051-24-3	0.0227	ER-L[3,7]	0.002	0.002	0.00020

Analyte	CAS Number	Project Action Limit[1] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
Total PCBs	-	0.0227	ER-L[3,7]	0.02	0.02	--
Total Organic Carbon				200	200	

* The QL exceeds the PQL and project action limit (PAL). However, the MDL is well below the PAL. If necessary, the laboratory has the capability to use Method 6020 (ICP-MS) to achieve a lower MDL and QL.

(1). The project action limit represents the selected ecological benchmark

(2). QLs and MDLs from Columbia Analytical Services

(3). ER-L – Effects-Range Low (Long, et al., 1995)

(4). ER-L – Effects-Range Low (Long and Morgan, 1991)

(5). AET – Apparent Effects Threshold (Buchman, 2006

(6). LEL – Lowest Effects Level (Persaud, et al., 1993)

(7). Total PCB value

mg/Kg - milligrams per kilogram (dry weight)

PCBs - Polychlorinated biphenyls

- Criterion not available

SAP Worksheet #15d -- Reference Limits and Evaluation Table

Matrix: Biota (clam, mussel, and crab) Samples

Analyte	CAS Number	Project Action Limit[(1)] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
TAL Metals						
Aluminum	7429-90-5	30	See Table 15-1	20	10	4.1
Antimony	7440-36-0	0.9	See Table 15-1	0.8	2*	0.80
Arsenic	7440-38-2	16	See Table 15-1	1	1	0.27
Barium	7440-39-3	575	See Table 15-1	20	2	0.51
Beryllium	7440-41-7	8.2	See Table 15-1	0.5	0.5	0.017
Cadmium	7440-43-9	12	See Table 15-1	0.5	0.5	0.017
Calcium	7440-70-2	--	See Table 15-1	500	100	13.5
Chromium	7440-47-3	37	See Table 15-1	1	1	0.048
Cobalt	7440-48-4	114	See Table 15-1	5	5	0.021
Copper	7440-50-8	90	See Table 15-1	2.5	2	0.13
Iron	7439-89-6	774	See Table 15-1	10	10	0.92
Lead	7439-92-1	45	See Table 15-1	1	0.5	0.15
Magnesium	7439-95-4	--	See Table 15-1	500	100	2.7
Manganese	7439-96-5	797	See Table 15-1	1.5	1	0.31
Mercury	7439-97-6	0.18	See Table 15-1	0.1	0.02	0.0021
Nickel	7440-02-0	26	See Table 15-1	4	4	0.043
Potassium	7440-09-7	--	See Table 15-1	500	100	100
Selenium	7782-49-2	3.1	See Table 15-1	1.5	1	0.29
Silver	7440-22-4	56	See Table 15-1	1	1	0.089
Sodium	7440-23-5	--	See Table 15-1	500	100	21.9
Thallium	7440-28-0	0.1	See Table 15-1	0.1	0.1	0.030
Vanadium	7440-62-2	9.5	See Table 15-1	5	5	0.035
Zinc	7440-66-6	1168	See Table 15-1	6	2	0.32

Analyte	CAS Number	Project Action Limit[(1)] (mg/Kg)	Project Action Limit Reference[1]	Project Quantitation Limit Goal (mg/Kg)	Laboratory-specific[2]	
					QLs	MDLs
PCB Homologs						
Monochlorobiphenyls	27323-18-8	--	See Table 15-1	0.02	0.02	0.00042
Dichlorobiphenyls	25512-42-9	--	See Table 15-1	0.02	0.02	0.00048
Trichlorobiphenyls	25323-68-6	--	See Table 15-1	0.02	0.02	0.0037
Tetrachlorobiphenyls	26914-33-0	--	See Table 15-1	0.02	0.02	0.0013
Pentachlorobiphenyls	25429-29-2	--	See Table 15-1	0.02	0.02	0.0015
Hexachlorobiphenyls	26601-64-9	--	See Table 15-1	0.02	0.02	0.0011
Heptachlorobiphenyls	28655-71-2	--	See Table 15-1	0.02	0.02	0.0012
Octachlorobiphenyls	31472-83-0	--	See Table 15-1	0.02	0.02	0.00094
Nonachlorobiphenyls	53742-07-7	--	See Table 15-1	0.02	0.02	0.0014
Decachlorobiphenyl	2051-24-3	--	See Table 15-1	0.02	0.02	0.0020
Total PCBs	-	1.1	See Table 15-1	0.02	0.02	--

* The analytical laboratory will make every effort to obtain a lower reporting limit for this element in order to meet the project action limit.

1. Biota Project Action Limits are calculated as described in Table 15-1 below.
2. QLs and MDL from Columbia Analytical Services

\- -Criterion not available

Table 15-1

Calculation of Project Action Limits in Tissue Samples

Parameter	Toxicity Reference Values[1]		Project Action Limits[2,3]	
	Mammal (mg/kg-day)	Bird (mg/kg-day)	Raccoon (mg/kg)	Herring Gull (mg/kg)
Aluminum	1.93	110	30	3034
Antimony	0.059	NV	0.9	NV
Arsenic	1.04	2.24	16	62
Barium	51.8	20.8	802	575
Beryllium	0.532	NV	8.2	NV
Cadmium	0.77	1.47	12	41
Calcium	NV	NV	NV	NV
Chromium	2.40	2.66	37	74
Cobalt	7.33	7.61	114	210
Copper	5.82	4.05	90	112
Iron	50	100	774	2766
Lead	4.7	1.63	73	45
Magnesium	NV	NV	NV	NV
Manganese	51.5	179	797	4951
Mercury	0.032	0.0064	0.5	0.18
Nickel	1.7	6.71	26	186
Potassium	NV	NV	NV	NV
Selenium	0.2	0.4	3.1	11
Silver	6.02	2.02	93	56
Sodium	NV	NV	NV	NV
Thallium	0.0074	NV	0.1	NV
Vanadium	4.16	0.344	64	9.5
Zinc	75.4	66.1	1168	1828
Total PCBs	0.068	0.18	1.1	5.0

(1) - The Toxicity Reference Values are No Observed Adverse Effects Level (see Appendix B)
(2) - The Project Action Limits are chemical concentrations in the tissue samples that would result in an EEQ that is equal to one in the conservative food chain model. It is calculated as presented below.
(3) - The shaded cell is the lower of the raccoon or herring gull Project action Limit.

$$PAL = \frac{BW*TRV*EEQ}{If}$$

Exposure Inputs	Raccoon	Herring Gull	Units
Body Weight = BW	3.670E+00	9.51E-01	kg
Food Ingestion Rate = If	2.370E-01	3.44E-02	kg/day

PAL = Project Action Limit
TRV = Toxicity Reference Value
EEQ - Ecological Effects Quotient = 1.0

The sources of the exposure parameters are presented in Appendix B.

SAP Worksheet #16 -- Project Schedule / Timeline Table

(*UFP-QAPP Manual Section 2.8.2*)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Extent of Contamination Marine Sediment Sampling	TtNUS	September 24, 2009	October 30, 2009	Technical Memorandum	December 30, 2009
Soil Sampling	TtNUS	April 23, 2010	May 21, 2010	Phase 2 RI Report	December 29, 2010
Ecological Sampling – Sediment, Biota, and Toxicity Samples	TtNUS	April 26, 2010	June 18, 2010	Phase 2 RI Report	December 29, 2010
Tier 2 Sediment Transport Model	TtNUS	February 22, 2010	November 12, 2010	Phase 2 RI Report	December 29, 2010

SAP Worksheet #17 -- Sampling Design and Rationale
(*UFP-QAPP Manual Section 3.1.1*)

Soil Samples

Nine soil borings will be advanced using hollow stem auger (HAS) drilling techniques in the southwest corner of the Site, near the coal bed area. Samples will be submitted to Mitkem Laboratories and will be analyzed for PAHs, TAL metals, and total petroleum hydrocarbons (TPH) measured as gasoline range organic compounds (GRO) and diesel range organic compounds (DRO). Four samples will be collected from each boring at the following sample intervals: 0 to 6 inches, 12 to 18 inches, 24 to 30 inches, and 42 to 48 inches. Sample intervals may be adjusted if shallow bedrock is encountered. Samples will be analyzed for TPH because “hot spots” of petroleum were detected downgradient of this area during the Phase 1 RI, but the source of petroleum was not identified (see Figure 11-1). Where possible, borings will be advanced into the water table to determine the presence of NAPL. If NAPL is present a sample will be collected and analyzed for VOCs and PCBs.

Four soil borings will be advanced in the area near the former rigging platform and analyzed for PCBs, PAHs, and Target Analyte List (TAL) metals. Continuous split barrel soil samples will be collected at two foot intervals throughout the soil column at these borings, which will be advanced to a depth of 20 feet below ground surface. This depth will reach well below the vadose zone, and below the adjacent sediment/water interface. Samples will be selected for laboratory analysis at a minimum as follows: 1) one sample will be selected from the ground surface, from a depth of 0-1 foot. 2) one sample will be selected from the top of the saturated zone, as identified by the field geologist. 3) any soil sample with oil staining as identified by the field geologist will also be selected for laboratory analysis. The collection of these samples will identify whether PCBs, PAHs, and metals are present in soil in this area that may be contributing to marine sediment contamination detected during the Phase 1 RI in the Stillwater Area (see Figure 11-1).

See Table 17-1 for a summary of borings to be completed. The coal pile area samples were arranged along the upgradient edge of the site boundary and along the former roadway to the west. Sample locations proposed for the former rigging platform were placed along the southwestern perimeter of the former platform, in the vicinity of the PCB removal area. These locations are expected to have the greatest chance of locating residual contamination if it is present.

Sediment Samples

Twenty sediment borings will be advanced using vibracore equipment or similar tools in the Stillwater Area up to a maximum depth of four feet below sediment surface. Four samples will be collected from each boring at the following depth intervals: 0 to 6 inches, 6 to 12 inches, 12 to 24 inches, and 42 to 48 inches. Water depth in the Stillwater Area is approximately 20 feet as published by NOAA.

Surfical samples (0 to 6 inches) will collected using ponar grab tools or similar dredge/grab samplers from 23 northeast shoreline locations and seven northwest shoreline locations.

All sediment samples will be analyzed by Mitkem Laboratories for PCBs, PAHs, and TAL metals to better define the extent of contamination for these chemicals.

See Figures 11-2 for the proposed sample locations. Offshore sample locations in the Stillwater Area were arranged in a grid pattern. The grid locations were based on an approximate 60 foot grid in the area extending approximately 120 feet north of the shoreline where the former rigging platform was located, with additional locations based on a 120-foot grid throughout the Stillwater Area to investigate the extent of contamination in marine sediments. Sediment sample locations along the northeast and northwest shorelines were arranged to compliment Phase 1 sediment locations so as to determine extent of contamination. See Table 17-2 for a summary of the extent of contamination sediment samples to be collected.

Ecological Samples

An ecological investigation will be conducted to provide additional data needed to complete a BERA for the Site. After chemical data have been obtained from the sediment samples collected during the Phase 2 RI sediment investigation, locations will be selected for the collection of sediment grab samples and biota. The sediment samples will undergo 28-day *Leptocheirus plumulosus* full-life-cycle chronic toxicity tests and will be analyzed by Columbia Analytical Services for PCB homologues, metals, PAHs, TOC, and grain size. The biota tissue samples will be analyzed by Columbia Analytical Services for PCB homologues, metals, and percent lipids. The data will be used to assess risk to benthic invertebrates, shellfish, mammals, and birds.

Twenty-four locations will be selected for biota sampling and 30 locations will be selected for surficial sediment (0 to 6 inches) sampling (six of these sediment locations will be selected in locations to be determined). Based on the concentrations detected from the extent investigation, they will be selected

over a concentration gradient (i.e., high, moderate, and low concentrations) to determine the relationship between sediment concentrations and toxicity or tissue concentrations with the goal of developing dose-response curves.

Ecological samples will be spatially located around the Site. Approximately 15 of the 24 locations will be located in the three areas of the Phase 2 sediment extent of contamination investigation. The three areas are locations where the highest concentrations of cadmium, lead, and PCBs were detected during the Phase 1 RI (SD304, SD312, and SD317). Three new locations will be located on the southern end of Gould Island in order to further investigate an apparent gradient of decreasing concentrations from the Northeast Shoreline Area to the southern end of the island. Three locations will be reference locations from Potter Cove. The ecological sediment locations will be distributed as follows (also, see Figure 11-3):

- Stillwater Area – 5 locations
- Northeast Shoreline – 5 locations
- Northwest Shoreline – 5 locations
- Locations previously found to contain elevated concentrations of contaminants – 3 locations (SD304, SD312, and SD317)
- South of Island – 3 locations
- Reference Area (Potter Cove or Cranston Cove, Jamestown) – 3 locations selected as described in Worksheet 11, Step 7.1.
- Locations to be determined – 6 locations

One sediment sample will be collected from each of these locations for toxicity testing and chemical analysis, for a total of 30 samples (plus field duplicates).

One co-located biota sample will be collected with sediment samples collected in the Stillwater Area, Northeast Shoreline, Northwest Shoreline, South of Island, and the Reference Area, if available, for a total of 24 samples plus field duplicates. Biota species to be collected will include green crabs (*Carcinus maenas)* or Asian crabs (*Hemigrapsus sanguineus)*, clams (*Mercenaria mercenaria*) (where available) and mussels (*Mytilus edulis*) (if clams are not available). Sample locations are designated for the collection of crab samples. Green crabs will be given preference over Asian crabs. Separate locations will be designated for the collection of clams. If clams are not available, mussels will be collected. Biota samples will be submitted for PCB homologue and metals laboratory analysis.

Each clam, mussel, or crab sample will be co-located with a sediment sample within a 25-foot radius. The number and locations of bivalve and crab samples will be broken down as follows:

- Stillwater Area – 2 clam or mussel samples and 3 crab samples
- Northeastern Shoreline – 2 clam or mussel samples and 3 crab samples
- Northwestern Shoreline - 2 clam or mussel samples and 3 crab samples
- South of Island – 1 clam sample, 1 mussel sample, and 1 crab sample
- Reference location – 3 clam or mussel samples and 3 crab samples

Location and surveying data will be obtained to identify the station and depth of samples collected.

Biota species to be collected will include green crabs (*Carcinus maenas)* or Asian crabs (*Hemigrapsus sanguineus)*, clams (*Mercenaria mercenaria*) (where available) and mussels (*Mytilus edulis*) (if clams are not available). Sample locations are designated for the collection of crab samples. Green crabs will be given preference over Asian crabs. Separate locations will be designated for the collection of clams. If clams are not available, mussels will be collected. Biota samples will be submitted for PCB homologue and metals laboratory analysis.

The locations will be selected to represent a range of PCB and metals concentrations in order to correlate toxicity and biota contamination with sediment concentrations. Five locations will be selected from each Phase 2 RI sediment investigation area (Stillwater, Northeast Shoreline, and Northwest Shoreline).

Tier 2 Sediment Transport Model

Additional sediment samples may be collected as part of the Tier 2 sediment transport model effort. A separate work plan document will be prepared that will contain all specific information on the methodology of the Tier 2 sediment transport model.

Table 17-1

Summary of Proposed Soil Borings

Boring/ Well ID	Location	Justification
GI-SB401	Southwest corner of site, northeast of former coal pile	Determine presence and type of petroleum contamination in soils upgradient of the southwest corner of former Building 32, and determine presence of elevated metals at the upgradient boundary of the site near the former dust collection building and former acid storage shed.
GI-SB402	Southwest corner of site, east of former coal pile	
GI-SB403		
GI-SB404		
GI-SB405	Southwest corner of site, southeast of former coal pile	
GI-SB406	Southwest corner of site, south of former Building 33 (Power Plant) and north of the former coal pile	
GI-SB407	Southwest corner of site, west of the former Building 33 (Power Plant).	
GI-SB408	Southwest corner of site, south of former Building 33 (Power Plant) and north of the former coal pile	
GI-SB409	Southwest corner of site, west of former Building 33 (Power Plant) and north of the former coal pile.	
GI-SB410	Northern end of site, adjacent to the former rigging platform	Determine if remnant PCBs, as well as PAHs and metals, are present in the soil in the vicinity of the former PCB release area near the former rigging platform.
GI-SB411		
GI-SB412		
GI-SB413		

Table 17-2

Summary of Proposed Sediment and Biota Samples

Sample ID	Location	Justification
EXTENT OF CONTAMINATION SAMPLES		
Stillwater Area		
GI-SD401	Stillwater Area, adjacent to the former rigging platform. These locations are all part of a 60 foot grid.	Determine the extent of previously detected PAHs, PCBs, and metals in the Stillwater Area. All samples are part of a series of samples that are arranged into a grid to determine the extent of contamination in sediment where rigging and heavy boat traffic operations formerly occurred.
GI-SD402		
GI-SD403		
GI-SD404		
GI-SD405		
GI-SD406		
GI-SD407		
GI-SD408		
GI-SD409		
GI-SD410		
GI-SD411		
GI-SD412		
GI-SD413	Stillwater Area. This location is part of a 120 foot grid.	This location is one of a series of samples that are arranged into a grid to determine the extent of potential contamination in sediment throughout the Stillwater Area.
GI-SD414		
GI-SD415		
GI-SD416		
GI-SD417		
GI-SD418		
GI-SD419		
GI-SD420		

Sample ID	Location	Justification
EXTENT OF CONTAMINATION SAMPLES		
Northeast Shoreline		
GI-SD421	Northeast shoreline, adjacent to Phase 1 RI sediment location SD317	To determine the extent of cadmium contamination previously detected at location SD317
GI-SD422	Northeast shoreline, adjacent to Phase 1 RI sediment location SD317	To determine the extent of cadmium contamination previously detected at location SD317
GI-SD423	Northeast shoreline, adjacent to Phase 1 RI sediment location SD317	To determine the extent of cadmium contamination previously detected at location SD317
GI-SD424	Northeast shoreline, adjacent to Phase 1 RI sediment location SD310	To resolve the extent of PAH, PCB, and metals contamination at a former outfall areas as well as off-shore of and between Phase 1 sediment sample stations on the northeast shoreline.
GI-SD425	Northeast shoreline, offshore from the Phase 1 RI sediment location SD310	
GI-SD426	Northeast shoreline, between Phase 1 RI sediment locations SD310 and SD309.	
GI-SD427	Northeast shoreline, between Phase 1 RI sediment locations SD310 and SD309.	
GI-SD428	Northeast shoreline, between Phase 1 RI sediment locations SD310 and SD309.	
GI-SD429	Northeast shoreline, offshore from the Phase 1 RI sediment location SD309.	
GI-SD430	Northeast shoreline, between Phase 1 RI sediment locations SD309 and SD306.	
GI-SD431	Northeast shoreline, between Phase 1 RI sediment locations SD309 and SD306.	
GI-SD432	Northeast shoreline, offshore from Phase 1 RI sediment locations SD306 and SD305.	
GI-SD433	Northeast shoreline, offshore from Phase 1 RI sediment location SD305.	

Sample ID	Location	Justification
EXTENT OF CONTAMINATION SAMPLES		
Northeast Shoreline		
GI-SD434	Northeast shoreline, between Phase 1 RI sediment locations SD305 and SD304.	To resolve the extent of PAH, PCB, and metals contamination at a former outfall areas as well as off-shore of and between Phase 1 sediment sample stations on the northeast shoreline.
GI-SD435	Northeast shoreline, offshore from Phase 1 RI sediment location SD304.	
GI-SD436		
GI-SD437	Northeast shoreline, south of Phase 1 RI sediment location SD304.	
GI-SD438	Northeast shoreline, south of Phase 1 RI sediment location SD304.	
Northwest Shoreline		
GI-SD439	Northwest shoreline between Phase 1 RI sediment locations SD313 and SD313F.	To resolve the extent of PAH and metals contamination at a former outfall areas as well as offshore of, and between Phase 1 sediment sample locations on the northwest shoreline.
GI-SD440	Northwest shoreline, offshore from Phase 1 RI sediment location SD313.	
GI-SD441	Northwest shoreline, south of Phase 1 RI sediment location SD313.	
GI-SD442	Northwest shoreline, south of Phase 1 RI sediment location SD302F.	
GI-SD443	Northwest shoreline, south of Phase 1 RI sediment location SD308.	
GI-SD444	Northwest shoreline, south of Phase 1 RI sediment location SD302.	
GI-SD445	Northwest shoreline, offshore from Phase 1 RI sediment location SD302.	
Near Shore Locations		
GI-SD446	Northeast shoreline, east of former outfall area	To resolve the nearshore extent of PAH and metals contamination at a suspected former source area on the northeast shoreline.
GI-SD447	Northeast shoreline, east of the former Bldg 54	To resolve the nearshore extent of PAH and metals contamination at a suspected former source area on the northeast shoreline.

Sample ID	Location	Justification
EXTENT OF CONTAMINATION SAMPLES (cont)		
Near Shore Locations (cont)		
GI-SD448	Northeast shoreline, east of former Bldg 38	To resolve the nearshore extent of PAH and metals contamination at a suspected former source area on the northeast shoreline.
GI-SD449	Northeast shoreline, east of former Bldg 50	To resolve the nearshore extent of PAH and metals contamination at a suspected former source area on the northeast shoreline.
GI-SD450	Northeast shoreline, east of former Riggers Storage Bldg	To resolve the nearshore extent of PAH and metals contamination at a suspected former source area on the northeast shoreline.
Reference Locations		
GI-JCC-01	Cranston Cove, Jamestown	Candidate Reference Location
GI-JCC-02	Cranston Cove, Jamestown	Candidate Reference Location
GI-JCC-04	Cranston Cove, Jamestown	Candidate Reference Location
GI-JPC-01	Potter Cove, Jamestown	Candidate Reference Location
GI-JPC-02	Potter Cove, Jamestown	Candidate Reference Location
GI-JPC-03	Potter Cove, Jamestown	Candidate Reference Location
Other Locations		
GI-SD304	Northeast Shoreline	Deep core sample to establish vertical extent by regulatory request – anticipate four samples at six inch vertical intervals
GI-SD311	South Area	Deep core sample to establish vertical extent by regulatory request – anticipate four samples at six inch vertical intervals

Sample ID	Location	Justification
ECOLOGICAL SEDIMENT SAMPLES		
Stillwater Area		
GI-SD501	Stillwater Area, specific locations will be determined based on analytical results from extent of contamination sampling	To be used in the Baseline Ecological Risk Assessment
GI-SD502		
GI-SD503		
GISD504		
GI-SD505		
Northeast Shoreline		
GI-SD506	Northeast Shoreline, specific locations will be determined based on analytical results from extent of contamination sampling	To be used in the Baseline Ecological Risk Assessment
GI-SD507		
GI-SD508		
GI-SD509		
GI-SD510		
GI-SD511	Northeast Shoreline, adjacent to Phase 1 sample SD-XXX	To be used in the Baseline Ecological Risk Assessment
GI-SD512	Northeast Shoreline, adjacent to Phase 1 sample SD-XXX	To be used in the Baseline Ecological Risk Assessment
GI-SD513	Northeast Shoreline, adjacent to Phase 1 sample SD-XXX	To be used in the Baseline Ecological Risk Assessment
Northwest Shoreline		
GI-SD514	Northwest Shoreline, specific locations will be determined based on analytical results from extent of contamination sampling	To be used in the Baseline Ecological Risk Assessment
GI-SD515		
GI-SD516		
GI-SD517		
GI-SD518		
South end of Island		
GI-SD519	South end of Island	To be used in the Baseline Ecological Risk Assessment
GI-SD520		
GI-SD521		

Sample ID	Location	Justification
ECOLOGICAL SEDIMENT SAMPLES (con't)		
Potter Cove or Cranston Cove, Jamestown		
GI-SD522	Potter Cove or Cranston Cove, Jamestown	To be used in the Baseline Ecological Risk Assessment as a reference condition, see Figure 11-4.
GI-SD523		
GI-SD524		
Location to be determined		
GI-SD525	Locations to be determined	To be used in the Baseline Ecological Risk Assessment
GI-SD526		
GI-SD527		
GI-SD528		
GI-SD529		
GI-SD530		

Sample ID	Location	Justification
ECOLOGICAL TISSUE SAMPLES		
Stillwater Area		
GI-ET501	Stillwater Area, locations to be determined.	To be used in the Baseline Ecological Risk Assessment
GI-ET502		
GI-ET503		
GI-ET504		
GI-ET505		
Northeast Shoreline		
GI-ET506	Northeast Shoreline, locations to be determined.	To be used in the Baseline Ecological Risk Assessment
GI-ET507		
GI-ET508		
GI-ET509		
GI-ET510		
Northwest Shoreline		
GI-ET511	Northwest Shoreline, specific locations will be determined.	To be used in the Baseline Ecological Risk Assessment
GI-ET512		
GI-ET513		
GI-ET514		
GI-ET515		
South end of island		
GI-ET516	South end of Island, locations to be determined.	To be used in the Baseline Ecological Risk Assessment
GI-ET517		
GI-ET518		
Potter Cove or Cranston Cove, Jamestown		
TBD	Locations to be determined, east shoreline of Jamestown Island.	Three reference stations to be selected from the group of six as stated in Worksheet 11, Step 7.1. The data from the three selected stations will be used as a baseline reference condition for the site, and site biota data will be compared to the samples from these reference locations as described in Figure 11-4.
TBD		
TBD		

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

(*UFP-QAPP Manual Section 3.1.1*)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
SB-401 through SB-409	Soil	four intervals (surface to refusal)	PAHs, metals, GRO and DRO	36 samples, 4 duplicates	Soil Sampling, SA-1.3 (Revision 7, 9/03)
SB-410 through SB-413	Soil	two intervals (surface and up to 20 feet bgs)	PCBs, PAHs, and metals	8 samples, 1 duplicate	Soil Sampling, SA-1.3 (Revision 7, 9/03)
SD-401 through SD-420	Sediment	four intervals (surface to 4 feet bgs)	PCBs, PAHs, and metals	80 samples, 8 duplicates	Surface Water and Sediment Sampling, SA-1.2 (Revision 5, 9/03)
SD-421 through SD-450	Sediment	surface (0 to 6 inches bgs)	PCBs, PAHs, and metals	30 samples, 3 duplicates	Surface Water and Sediment Sampling, SA-1.2 (Revision 5, 9/03)
ET-501 through ET-524	Ecological tissue	NA	PCB homologues and metals	24 samples 3 duplicates	Worksheet 14
SD-501 through SD-530	Sediment	surface (0 to 6 inches bgs)	toxicity test, PCB homologues, PAHs, metals, TOC, and grain size	30 samples, 3 duplicates	Surface Water and Sediment Sampling, SA-1.2 (Revision 5, 9/03)

SAP Worksheet #19 -- Analytical SOP Requirements Table

(*UFP-QAPP Manual Section 3.1.1*)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference	Containers (number, size, and type)	Sample volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation / analysis)
Aqueous Rinsate and Field Blanks	GRO	SW-846 8015B/3.3.17	2-40 mL VOC vials	2-40 mL VOC vials	HCl to pH < 2, Cool to 4°C	14 Days to Analysis
	DRO	SW-846 3510C/8015B/3.3.18	4-1 Liter amber class bottles	1 liter per analysis	Cool to 4°C	7 Days (Extraction) / 40 Days (Analysis)
	PAHs	SW-846 3520C/ 8270C/3.3.15 & 3.3.46 (SIM)				
	PCBs	SW-846 3510C/8082/3.3.16				
	PCB Homologs[2]	SW-846 3510C/680				
	Metals	SW-846 3020A/6010B/7470A/3.2.1 & 3.2.3	1-500-mL polyethylene bottle	100 mL	Nitric Acid to pH < 2, Cool to 4°C	6 Months (Analysis), 28 Days for mercury
Soil (coal pile area)	PAHs	SW-846 3541 or 3550B/ 8270C/3.3.15 & 3.3.31	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A/3.2.64, 3.2.1 & 3.2.4	1-4 oz wide mouth jar	10 g	Cool to 4°C	6 Months (Analysis), 28 Days for mercury
	GRO	SW-846 5030/8015B/3.3.17	2-40 mL VOC vials	2-40 mL VOC vials	5 mL methanol, Cool to 4°C	14 Days to Analysis
	DRO	SW-846 3541 or 3550B/8015B/3.3.18	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
Soil (former rigging platform area)	PAHs	SW-846 3541 or 3550B/ 8270C/3.3.15 & 3.3.31	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	PCBs	SW-846 3541 or 3550B/8082/3.3.35 &3.3.16	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A/3.2.64, 3.2.1 & 3.2.4	1-4 oz wide mouth jar	10 g	Cool to 4°C	6 Months (Analysis), 28 Days for mercury
Sediments – Extent Investigation	PAHs	SW-846 3541 or 3550B/ 8270C-SIM/3.3.31 & 3.3.46	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	PCBs	SW-846 3541 or 3550B/8082/3.3.35 & 3.3.16	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A/3.2.64, 3.2.1 & 3.2.4	1-4 oz wide mouth jar	10 g	Cool to 4°C	6 Months (Analysis), 28 Days for mercury
Clams/Mussels	PCB Homologs	SW-846 3550B/680 EXT-3550 SOC-680	Ziplock bags	30g of tissue	Cool to 4°C Freeze upon receipt	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A MET-3050 MET-200.7/6010 MET-7471/245.5		10 g	Cool to 4°C Freeze upon receipt	6 Months (Analysis), 28 Days for mercury

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference	Containers (number, size, and type)	Sample volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation / analysis)
Crabs	PCB Homologs	SW-846 3550B/680 EXT-3550 SOC-680	Ziplock bags	30g of tissue	Cool to 4°C Freeze upon receipt	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A MET-3050 MET-200.7/6010 MET-7471/245.5		10 g	Cool to 4°C Freeze upon receipt	6 Months (Analysis), 28 Days for mercury
Ecological Sediments	PAHs	SW-846 3550B/ 8270C-SIM EXT-3550 SOC-8270C	1-4 oz wide mouth jar	30g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	PCB Homolog	SW-846 3550B/680 EXT-3550 SOC-680	1-4 oz wide mouth jar	20g	Cool to 4°C	14 Days (Extraction) / 40 Days (Analysis)
	Metals	SW-846 3050B/6010B/7471A MET-3050 MET-200.7/6010 MET-7471/245.5		10 g	Cool to 4°C	6 Months (Analysis), 28 Days for mercury
	Toxicity testing	ASTM E 1367-03	Ziplock bags or lab-specified plastic container	1 gallon	Cool to 4°C	2 weeks to test

[1] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

[2] Rinsate blanks for shellfish samples will be generated by at the laboratory processing the shellfish samples prior to analysis.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(*UFP-QAPP Manual Section 3.1.1*)

Matrix	Analytical Group	No. of Samples	No. of Field Duplicates	No. of MS/MSDs[1]	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	Total No. of Samples to Lab[2]
Soil (coal pile area)	PAHs	36	4	2	1	2		43
	Metals	36	4	2	1	2		43
	GRO	36	4	2	1	2	6	49
	DRO	36	4	2	1	2	-	43
Soil (PCB release area)	PAHs	8	1	1	1	1	-	11
	PCBs	8	1	1	1	1	-	11
	Metals	8	1	1	1	1	-	11
Sediment	PAHs	118	12	6	1	6	-	138
	PCBs	118	12	6	1	6	-	138
	Metals	118	12	6	1	6	-	138
Crabs	PCB Homologs	13	2	1	-	1[3]	-	16
	Metals	13	2	1	-	1[3]	-	16
	Percent lipids	13	2	1	-	1[3]	-	16
Clams/Mussels	PCB Homologs	11	2	1	-	1[3]	-	14
	Metals	11	2	1	-	1[3]	-	14
	Percent lipids	11	2	1	-	1[3]	-	14
Sediment Ecological	PCB Homologs	30	3	2	1	2	-	36
	PAHs	30	3	2	1	2	-	36
	Metals	30	3	2	1	2	-	36
	TOC	30	3	2	-	-	-	33
	Grain size	30	3	-	-	-	-	33
	Toxicity	30	1	-	-	-	-	31

[1] MS/MSD for organic and MS/Lab Dup for metals.
[2] Total number of samples does not include the MS/MSD
[3] Equipment blanks for biota samples will be generated at the laboratory processing the shellfish samples prior to analysis.

Notes:
(1) Collect 1 rinsate blank per 20 field samples.
(2) Collect 1 duplicate per 10 field samples.
(3) Collect 1 field blank per water source (e.g. decontamination rinse water).
(4) Collect 1 trip blank per cooler containing GRO samples.
(5) Assign one lab QC sample per 20 samples – Organics – Matrix Spike and a Matrix Spike Duplicate, Inorganics – Matrix Spike and Laboratory Duplicate. The Lab QC samples are not included in the total sample count.

SAP Worksheet #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
S1	Site Reconnaissance, GH-1.1 (6/99, Revision 1)	TtNUS	NA	N	
S2	Utility Locating and Excavation Clearance, HS-1.0 (12/03, Revision 2)	TtNUS	NA	N	
S3	Soil and Rock Drilling Methods, GH-1.3	TtNUS	Drilling rig and accessories	N	
S4	Borehole and Sample Logging, GH-1.5 (6/99, Revision 1)	TtNUS	Geological logging	N	
S5	Soil Sampling, SA-1.3 (9/03, Revision 7)	TtNUS	Soil sampling	N	Additional details are described in Worksheet 14
S6	Photovac 2020 Photoionization Air Monitor, ME-12	TtNUS	VOC scanning	N	
S7	Photovac MicroFid Handheld Flame Ionization Detector, ME-15	TtNUS	VOC scanning	N	
S8	Surface Water and Sediment Sampling, SA-1.2 (9/03, Revision 5)	TtNUS	Sediment sampling	N	Additional details are described in Worksheet 14
S9	NA		Biota sampling		Biota sampling procedures are described in Worksheet 14
S10	Sample Nomenclature, CT-04 (9/03, Revision 1)	TtNUS	NA	N	
S11	Database Records and Quality Assurance, CT-05 (1/01, Revision 2)	TtNUS	NA	N	
S12	Non-radiological Sample Handling, SA-6.1 (2/04, Revision 3)	TtNUS	Sample bottleware, packaging material, shipping materials	N	
S13	Field Documentation, SA-6.3 (9/03, Revision 2)	TtNUS	Field logbook, field sample forms, boring logs	N	
S14	Decontamination of Field Equipment and Waste Handling, SA-7.1 (9/03, Revision 3)	TtNUS	Decon equipment, scrub brushes, phosphate free detergent, DI water	N	

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

(*UFP-QAPP Manual Section 3.1.2.4*)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Photovac 2020 Photoionization Detector (PID)	Calibration	Prior to use; factory inspection and calibration once a year or when malfunctioning	Instrument indicates calibration is complete	repair or use alternate equipment	project field personnel	S6	Rental field equipment will be used
Photovac 2020 Photoionization Detector (PID)	Maintenance	Wipe down after each use; clean UV light source every 24 hours of operation; change sample inlet filter weekly or as required; charge battery after each use; clean ionization chamber monthly			project field personnel	S6	
Photovac MicroFID Flame Ionization Detector (FID)	Calibration	Prior to use	Instrument indicates calibration is complete	repair or use alternate equipment	project field personnel	S7	
Photovac MicroFID Flame Ionization Detector (FID)	Maintenance	Charge battery prior to use (8 hours); empty hydrogen cylinder prior to transport and refill on arrival; replace sample inlet filter weekly or when pump sounds labored			project field personnel	S7	

SAP Worksheet #23a -- Analytical SOP References Table – Mitkem Laboratories

(*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)
1	70.0033:Semivolatiles by Method 8270D SIM, Rev 5, 12/07	Definitive	Solid /PAHs (SIM)	GC/MS	Mitkem Laboratories	N
2	60.0003: PCBs by Method 8082, Rev. 8, 3/08	Definitive	Solid /PCB Aroclors	GC/ECD	Mitkem Laboratories	N
3	90.0038: Gasoline Range Organics by GC/FID using Method SW-846 8015 Rev 10, 1/09	Definitive	Solid/GRO	GC/FID	Mitkem Laboratories	N
4	60.0050: TPH by GC/FID using Method SW-846 8015B, Rev 10, 2/07	Definitive	Solid /ExTPH	GC/FID	Mitkem Laboratories	N
5	100.0111: Metals by Method 6010C, Rev 12, 2/09	Definitive	Solid/ICP Metals	ICP-AES	Mitkem Laboratories	N
6	110.0110: Metals in Water and Soils by Method 6020A ICP-MS, Rev 0, 3/09	Definitive	Solid/ICP Metals	ICP-MS	Mitkem Laboratories	N
7	100.0012: Mercury by Method 7470A/7471B, Rev 9, 11/08	Definitive	Solid /Mercury	CVAA	Mitkem Laboratories	N
8	110.0038: Percent Moisture, Rev 7, 2/09	Definitive	Solid/Percent Moisture	Oven	Mitkem Laboratories	N
9	50.0052, Organic Preparation of Soil Samples by Sonication, Method 3550B, Rev 2, 1/08	Definitive	Solid/PAH, PCB, ExTPH	NA	Mitkem Laboratories	N
10	50.0053, Organic Preparation of Soil Samples by Soxhlet, Method 3540C, Rev 2, 1/08	Definitive	Solid/ PAH, PCB, ExTPH	NA	Mitkem Laboratories	N
11	50.0100, Organic Preparation of Soil Samples by MSE, Method 3570, Rev 1, 1/08	Definitive	Solid/ PAH, PCB, ExTPH	NA	Mitkem Laboratories	N
12	50.0054, Organic Extract Filtration and Concentration Techniques, Rev 1, 2/09	Definitive	Solid/ PAH, PCB, ExTPH	NA	Mitkem Laboratories	N
13	100.0104, Sample Preparation of Soils by Acid Digestion for ICP, Method 3050B, Rev 7, 3/09	Definitive	Solid/ICP Metals	NA	Mitkem Laboratories	N

SAP Worksheet #23b -- Analytical SOP References Table – Columbia Analytical Services

(*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)
MET-200.7/6010	Determination of Metals and Trace Elements by Inductively Coupled Plasma Atomic Emission Spectrometry, Rev. 9, 7/12/06	Definitive	Sediments, Biota - Metals	ICP-AES	Columbia Analytical Services	N
MET-3050	Metals Digestion , Soil, Sediment, sludge for ICP-AES, ICP-MS and GFAA Analysis, Rev. 3, 1/15/03	Preparation	Sediments, Biota - Metals	Hotblock	Columbia Analytical Services	N
MET-7471/245.5	Mercury in Solid or Semisolid Waste by Cold Vapor Atomic Absorption Spectrometry, Rev. 4, 12/9/05	Definitive	Sediments, Biota - Metals	CVAA	Columbia Analytical Services	N
SOC-680	PCBs by GC/MS, Rev. 2, 4/6/04	Definitive	Biota, Ecological Sediment – PCB Homolog	GC/MS	Columbia Analytical Services	N
SOC-8270C	Semivolatile Organic Compounds by GC/MS, Rev. 6, 3/31/06	Definitive	Sediments – PAHs (SIM)	GC/MS	Columbia Analytical Services	N
EXT-3550	Ultrasonic Extraction, Rev. 2, 4/3/02	Preparation	Biota, Ecological Sediment – PCB Homolog Sediments - PAHs	Sonicators	Columbia Analytical Services	N

SAP Worksheet #24a -- Analytical Instrument Calibration Table – Mitkem Laboratories

(*UFP-QAPP Manual Section 3.2.2*)

Instrument	Calibration Procedure	Frequency of Calibration[1]	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GCMS - PAHs - SIM	Initial Calibration	After major instrument maintenance or when CCV fails	%RSD <20% with a maximum of 10% of the target analytes and/or surrogate compounds allowed %RSD <50%. Relative Retention Times must meet ±0.06 RRT units for each compound and surrogate. Minimum RRFs are suggested in Table 4 of the method.	Check instrument performance, perform corrective maintenance, recalibrate	Mitkem Department Supervisor	1
	Second Source Initial Calibration Verification (ICV)	After each initial calibration	Recovery within 80-120%	Investigate problem, verify second source standard. Reinject standard. Reanalyze initial calibration.	Mitkem Department Supervisor	
	Continuing Calibration Verification (CCV)	Every 12 hours	%D <20% with a maximum of 20% of the target analytes and/or surrogate compounds allowed %D < 50% D. Minimum RRFs are suggested in Table 4 of the method.	Check instrument performance, perform corrective maintenance, recalibrate and reanalyze all samples analyzed since the last successful calibration verification.	Mitkem Department Supervisor	
	DFTPP Tune	Every 12 hours	Ion abundance within acceptance limits listed in Table 3 of test method.	Check instrument performance, perform maintenance, retune the instrument and reanalyze tuning solution.	Mitkem Department Supervisor	
GC-PCBs	Initial calibration (5-level calibration)	After major instrument maintenance	%Relative Standard Deviation <20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	2
	Continuing calibration (middle range calibration)	Every 10 samples and at end of run	%Difference < 20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration[1]	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC - GRO, ExTPH	Initial Calibration	After major instrument maintenance, or when CCV fails	5 point calibration curve for individual compounds, with %Relative Standard Deviation for analytes <20%	Check instrument performance, maintenance, recalibrate	Mitkem Department Supervisor	3, 4
	Continuing Calibration	Every 12 hours and/or every 20 samples	%Difference for analytes <20% (GRO), <25% (ExTPH)	Check instrument performance, maintenance, recalibrate	Mitkem Department Supervisor	
ICP	Initial calibration (3-level)	Every analytical run	Initial Calibration: correlation coefficient >=0.995	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	
	Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	
	Continuing calibration verification (CCV)	Every 10 samples and at end of run	Continuing Calibration: all analytes within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	
	Continuing calibration blank	Before beginning a sample run, every 10 samples and at end of run	No analytes > quantitation limit	Reanalyze	Mitkem Department Supervisor	
ICP-MS	Tune	Daily for each instrument run.	Measured mass within 0.05 amu of actual mass, resolution <0.9 amu full width at 10% peak height.	Check instrument performance, perform maintenance, retune the instrument and reanalyze tuning solution	Mitkem or Columbia Department Supervisor	6 or 20
	Initial calibration (3-level)	Every analytical run	Initial Calibration: correlation coefficient >=0.995	Check instrument performance, perform maintenance, recalibrate	Mitkem or Columbia Department Supervisor	
	Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Columbia Department Supervisor	
	Continuing calibration verification (CCV)	Every 10 samples and at end of run	Continuing Calibration: within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Columbia Department Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration[1]	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	Continuing calibration blank	Before beginning a sample run, every 10 samples and at end of run	No analytes > quantitation limit	Reanalyze	Columbia Department Supervisor	
Mercury analyzer	Initial calibration (5-level)	Every analytical run	Initial Calibration: correlation coefficient >=0.995	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	7
	Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	All analytes within +/- 10%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	
	Continuing calibration verification (CCV)	Every 10 samples and at end of run	Continuing Calibration: within +/- 20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	
	Continuing calibration blank	Before beginning a sample run, every 10 samples and at end of run	No analytes > quantitation limit	Reanalyze	Mitkem Department Supervisor	
GC-PCBs	Initial calibration (5-level calibration)	After major instrument maintenance	%Relative Standard Deviation <20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	2
	Continuing calibration (middle range calibration)	Every 10 samples and at end of run	%Difference < 20%	Check instrument performance, perform maintenance, recalibrate	Mitkem Department Supervisor	

(1) "As needed" is defined by each method. NEL policy is to analyze an initial calibration upon: (1) change in instrumentation (major maintenance, including source cleaning, upgrade or use of a different component (e.g. purge and trap apparatus), or capillary column change; and, (2) CCV does not meet initial curve even after routine maintenance (changing liner, cleaning injection port, etc.).

SAP Worksheet #24b -- Analytical Instrument Calibration Table – Columbia Analytical Services

(*UFP-QAPP Manual Section 3.2.2*)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference
ICP-AES	ICAL - Three point calibration	At the beginning of each day or if QC is out of criteria.	Analytes run at their calibration levels must fall within 90-110% of True Values. Correlation Coefficient ≥0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	MET-200.7/6010B
ICP-AES	CCV	One for every 10 client samples	90-110% Recovery	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	MET-200.7/6010B
CVAA	ICAL - Minimum 5 point (plus a blank)	At the beginning of each day or if QC is out of criteria	ICV 90-110% Recovery Correlation Coefficient ≥0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	MET-7471A/245.5
CVAA	CCV	One for every 10 client samples	90-110% Recovery	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	MET-7471A/245.5
GC/MS - LVI	ICAL - Minimum five point calibration for all analytes	Instrument receipt, instrument change (new trap, column, etc.), or when CCV does not meet criteria	RSD for each CCC < 30%, minimum mean RF for each SPCC as noted in 7.3.5.4 of method 8270C. If RSD for an analyte is > 15% apply linear (r2 > 0.99) method for quantitation ICV recovery within 70-130%.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/Supervisor	SOC-8270C
GC/MS - LVI	CCV	Each 12 hour window	%D for each CCC ≤ 20%, minimum RF for each SPCC as noted in SOP and Method 8270C	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	SOC-8270C
GC/MS - SIM	ICAL	Instrument receipt, instrument change (new trap, column, etc.), or when CCV does not meet criteria	RSD for each target < 20%, If RSD for an analyte is > 20% apply linear (r2 > 0.99) method for quantitation ICV recovery within 70-130%	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	SOC-680
GC/MS - SIM	CCV	Each 12 hour window	%D for each ≤ 20% of the mean,	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/Supervisor	SOC-680

SAP Worksheet #25a -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – Mitkem Laboratories

(*UFP-QAPP Manual Section 3.2.3*)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GCMS - PAH (SIM)	Check for leaks, replace gas line filters, recondition or replace trap, replace column, clean injection port/liner and replace septum as needed, replace Electron Mulitplier	Tune (BFB or DFTPP), Continuing Calibration Verification	Monitor instrument performance via tuning mass criteria, and Continuing Calibration Verification	Tune daily, CCV after every 12 hours of operation	Ion abundance within acceptance limits for tune, CCV %D ≤20%	As needed, replace connections, gas line filters, trap, or GC column. Clip column, replace injection port liner, clean injection port, clean source. Repeat tune, calibration or CCV and any affected samples. See Attachment in SOP for more details.	Mitkem Department Supervisor	1
ICP-AES	Perform leak test, change pump tubing, change torch and window, clean filters	Calibration Verification and Calibration Blank	Monitor instrument performance viaCalibration Verification andCalibration Blank	Daily, after every 10 samples	CV: all analytes ±10% CB: no analytes > quantitation limit	Check/replace pump tubing, clean torch and window, clean all filters. Repeat calibration or CCV and any affected samples. See section 6.2 of SOP for more details.	Mitkem Department Supervisor	5
ICPMS	Perform leak test, change pump tubing, remove and clean cone, extraction lens and ion lens stack	Calibration Verification and Initial Calibration Blank	Monitor instrument performance viaCalibration Verification andCalibration Blank	Daily, after every 10 samples	CV: all analytes ±10% CB: no analytes > quantitation limit	Check/replace pump tubing, clean torch and window, clean all filters. Repeat calibration or CCV and any affected samples. See section 6.2 of SOP for more details.	Mitkem or Columbia Department Supervisor	6 or 20
Mercury Analyzer	Perform leak test, change tubing, clean window, clean filters	Initial Calibration Verification and Initial Calibration Blank	Monitor instrument performance via Initial Calibration Verification and Initial Calibration Blank	Daily, after every 10 samples	ICV: Hg within ±10% CCV: Hg within ±20% ICB: Hg < QL	Replace connections, replace pump tubing, clean all filters. Repeat calibration or CCV.	Mitkem Department Supervisor	7

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/ECD – PCBs; GC/FID – GRO, ExTPH	Check for leaks, replace gas line filters, clip end of column, recondition or replace column, clean injection port/liner, replace septum	Continuing Calibration Verification	Monitor instrument performance via Continuing Calibration Verification	Daily, after every 10 Samples	%D <20% (GRO, PCB), <25% (ExTPH)	As needed, check GC conditions, check for leaks, clip column, clean/replace injection port/ liner. Repeat calibration or CCV and affected samples. See Attachment 1 of SOP for more details.	Mitkem Department Supervisor	2, 3, 4

SAP Worksheet #25b -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – Columbia Analytical Services

(*UFP-QAPP Manual Section 3.2.3*)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed	PAH/PCB Homolog Analysis	Initial/ Continuing Calibration	Instrument receipt, instrument change (new trap, column, etc.), when CCal does not meet criteria or when manual tune.	Same as ICAL/CCal	Same as ICAL/CCal	Analyst/Supervisor	SOC-8270C SOC-680
ICP-AES	Check pressure and gas supply daily. Clean pump tubing daily. Clean nebulizer, spray chamber and torch every 2 weeks. Replace water filters quarterly. Replace vacuum filters monthly.	Trace Metals	Initial/Continuing Calibration	Instrument receipt, instrument change (new parts) When calibration criteria not met.	Same as ICAL/CCal	Same as ICAL/CCal	Analyst/Supervisor	MET-200.7/6010B
CVAA	Check pressure and gas supply daily. Check/change aspiration tubing daily. Clean burner head daily. Clean optic every 3 months. Clean furnace windows monthly.	Mercury	Initial/Continuing Calibration	Instrument receipt, instrument change (new parts) When calibration criteria not met.	Same as ICAL/CCal	Same as ICAL/CCal	Analyst/Supervisor	MET-7471A/245.5

SAP Worksheet #26a -- Sample Handling System – Mitkem Laboratories

(*UFP-QAPP Manual Appendix A*)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operations Leader, Tetra Tech NUS
Sample Packaging (Personnel/Organization): Field Operations Leader, Tetra Tech NUS
Coordination of Shipment (Personnel/Organization): Field Operations Leader, Tetra Tech NUS
Type of Shipment/Carrier: Laboratory courier service (Mitkem)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Custodians, Mitkem Laboratories
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff (organic / inorganic), Mitkem Laboratories
Sample Determinative Analysis (Personnel/Organization): GC/MS, Metals Laboratory Staff, Mitkem Laboratories
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will be shipped or delivered to the laboratory 1 or 2 days from collection.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 6 months from submittal of final data report
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL (Intact leftover samples)
Personnel/Organization: Sample Custodians, Mitkem Laboratories
Number of Days from Analysis: 60 days from submittal of final data report

SAP Worksheet #26b -- Sample Handling System – Columbia Analytical Services

(*UFP-QAPP Manual Appendix A*)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operations Leader/Tetra Tech NUS, Inc.
Sample Packaging (Personnel/Organization): Field Operations Leader/Tetra Tech NUS, Inc.
Coordination of Shipment (Personnel/Organization): Field Operations Leader/Tetra Tech NUS, Inc.
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Management Analyst/Columbia Analytical Services
Sample Custody and Storage (Personnel/Organization): See above
Sample Preparation (Personnel/Organization): Organic and/or inorganic analysts- Columbia Analytical Services -See preparation SOPs referenced on Worksheet #23
Sample Determinative Analysis (Personnel/Organization): Organic and/or inorganic analysts - Columbia Analytical Services -See preparation SOPs referenced on Worksheet #23
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): See Worksheet #19 and NEL SOP # 3.7.11
Sample Extract/Digestate Storage (No. of days from extraction/digestion): See Worksheet #19 and SOC-680, MET-3050
Biological Sample Storage (No. of days from sample collection): See Worksheet #19 and SOC-680, MET-3050
SAMPLE DISPOSAL
Personnel/Organization: Technical Assistant/Columbia Analytical Services
Number of Days from Analysis: See Columbia Analytical Services SOP – SMO-SPLDIS.

SAP Worksheet #27 – Sample Custody Requirements Table

(*UFP-QAPP Manual Section 3.3.3*

Sample Designation and Tracking System

Each sample collected will be assigned a unique sample tracking number used to catalog the results. The sample tracking number will consist of alpha-numeric characters identifying the Site, sample medium, location, and depth or date. Any other pertinent information regarding sample identification will be recorded on the sample logsheets, chains of custody forms or in the field logbooks.

The alpha-numeric (A-N) coding to be used in the sample system is detailed below and in the subsequent definitions.

AAA	-	AANNN	-	(AAAA) or (NNNN)
(Site ID)	-	(Medium & Location)	-	Depth/Date/Species

Site identifier: "GI" for Site 17, Gould Island

Medium identifier: "SB" for soil boring samples,
"SD" for sediment samples, and
"ET" for ecological tissue of the biota samples

Sample location identifier: Each sample station will be assigned a unique location identifier composed of numeric characters, in sequence beginning with "01". 400-series samples will include extent of contamination sediment samples. 500-series samples will include ecological sediment and biota samples.

Depth/Date/Species: For soil and sediment sample locations, this portion of the sample tracking number will represent the depth in feet below ground surface (bgs) from which the sample was collected; e.g., for soil samples collected from 2 to 4 feet bgs, this portion of the sample tracking number will be "0204". For groundwater samples, this portion of the sample tracking number will be the date collected (Month, Day, Year), and for tissue samples, this portion will represent the species (IBM = Indigenous Blue Mussels, HSC = Hard Shell Clam, and GCR = Green Crab).

For example, a soil sample from soil boring SB01 from the 10 to 12 feet interval will be labeled GI-SB401-1012. A sediment sample collected at the SD401 location from the 6 to 12 inches interval will be labeled GI-SB401-0612.

Quality Control Samples (QC) samples collected during the Phase 2 RI will use the same coding system as for the environmental samples. Field QC sample types are presented in Section 3.4. Field QC designations will conform with the following formats:

- Field Duplicates: Blind field duplicate samples will be designated such that the location designation will be replaced with "DUP" followed by a sequential value (the nth duplicate sample collected during that sampling event) and a chronological value (MMDDYY). The sample log sheet will note which sample location the duplicate was collected from. The first sediment field duplicate sample collected on October 5, 2007 will be labeled GI-SD-DUP01-100507 and the second field duplicate will be GI-SD-DUP02-100507. The first soil field duplicate collected will be labeled GI-SB-DUP01-100507.

- Field Blanks: Field blank sample identifiers will consist of the "FB" label, the medium, and a chronological value (MMDDYY). Example: GI-SD-FB01-100507.

- Rinsate Blanks: Rinsate blank sample identifiers will consist of the "RB" label, the medium, and a chronological value (MMDDYY). Example: GI-SD-RB01-100507.

  Laboratory QC samples (matrix spike and laboratory duplicate samples) have no separate sample identifier codes, but are noted on the chain-of-custody record and sample log sheet.

Sample Handling and Chain-of Custody Procedures

Custody of samples must be maintained and documented at all times. To ensure the integrity of a sample from collection through analysis, an accurate written record is necessary to trace the possession and handling of the sample. This documentation is referred to as the "chain of custody" form. Chain of custody begins when samples are collected in the field, and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be accompanied by a chain-of-custody form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

Following collection, samples will be placed on ice in a secure cooler and attended by TtNUS personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. Chain of custody procedures are described in further detail in the following TtNUS SOPs (presented in Appendix C):

- SA-6.3 Field Documentation
- SA-6.1 Non-Radiological Sample Handling

The samples will be shipped to the laboratories in coolers packed with ice and bubble wrap, or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain of custody is maintained. The chain-of-custody forms are shipped to the laboratory with the samples.

Samples will be shipped to the laboratories by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times before sample extraction, digestion, or analysis are presented in Worksheet Number 19. Saturday deliveries will be coordinated by the FOL or his designee with the laboratory. This table also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

Each sample collected will be assigned a unique sampling tracking number. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the chain-of-custody form. Preservatives used will be stated on the sample label and the chain of custody form. The chain-of-custody form is a two-part form: the original accompanies the samples to the analytical laboratory, and the copy is retained by the sampling staff until it is submitted to the project manager and data validators.
One copy of the chain-of-custody form will be kept by archived in the project files. Information to be recorded on the chain-of-custody form should include:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- Quality control (QC) sample designation
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

The field crew will attempt to identify any potentially high concentration samples on the chain-of-custody form. Groundwater sample salinity will be noted on the form to aid the analytical laboratory in establishing dilution requirements for metals analyses where salinity could present an interference concern.

SAP Worksheet #28a -- Laboratory QC Samples Table

(*UFP-QAPP Manual Section 3.4*)

Matrix	Soil
Analytical Group	GRO/DRO
Analytical Method / SOP Reference	GRO-SW846 8015B/ Lab SOP 3 DRO- 3541 or 3550B/8015B/Lan SOP 3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	< 1/2QL	Investigate source of contamination. Rerun method blank. Re-extract associated batch of samples unless ND for the contaminant.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	< 1/2QL
Laboratory Control Sample (LCS)	One per prep batch of twenty or fewer samples of similar matrix	80-120 %R	Reanalyze once. If the LCS recoveries are high but the sample results are <QL narrate. Flag with * on Form 3	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120 %R
Laboratory Control Sample Duplicate (LCSD)	One per prep batch of twenty or fewer samples of similar matrix	80-120 %R RPD ≤ 20%	Reanalyze once. If the LCS recoveries are within QC limts, narrate. Flag with * on Form 3	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	80-120 %R RPD ≤ 20%
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	60-140 %R	If recoveries are outside limits and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable check standard prep. Speak with PM regarding further action. Flag outliers with * on Form 3	Analyst, Supervisor, QA Manager	Accuracy/Bias	60-140 %R
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	60-140 %R RPD ≤ 30%	Same as MS	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	60-140 %R RPD ≤ 30%
Surrogates	1 Per Sample	Bromofluorobenzene, 79-118 %R	Unless obvious chromatographic interference, if sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag with * on Form 2.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Bromofluorobenzene, 79-118 %R

Matrix	Soil
Analytical Group	GRO/DRO
Analytical Method / SOP Reference	GRO-SW846 8015B/ Lab SOP 3 DRO- 3541 or 3550B/8015B/Lan SOP 3

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	< 1/2QL	Investigate source of contamination. Rerun method blank. Re-extract associated batch of samples unless ND for the contaminant.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	< 1/2QL

SAP Worksheet #28b -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	PAHs
Analytical Method / SOP Reference	SW846 8270D/ Lab SOP 1

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No target analytes >one half of Quantitation Limit except for common lab contaminants not > QL	Investigate source of contamination. Rerun method blank. No samples may be run until an acceptable method blank has been run.	Laboratory Supervisor	Accuracy/bias-Contamination	No target analytes > ½ QL (> QL for common laboratory contaminants), unless target analytes in field samples are > 10x those in method blank.
Matrix Spike	One per batch of twenty or fewer samples of similar matrix	Recovery within lab statistical QC limits	If recoveries are outside limits and surrogate and LCS criteria are met, note in narrative. If both the LCS and MS/MSD are unacceptable reprepare the samples and QC. Check standard prep. Flag outliers with * on Form 3.	Laboratory Supervisor / Data Validator	Accuracy/bias	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of twenty or fewer samples of similar matrix	Recovery within lab statistical QC limits; 40% RPD	Same as MS.	Laboratory Supervisor / Data Validator	Accuracy/bias & Precision	Recovery within lab statistical QC limits; 40% RPD
LCS/LCS Duplicate	One per batch of twenty or fewer samples of similar matrix. Include LCSD if prep batch does not include MS/MSD.	Recovery within lab statistical QC limits; 40% RPD	Reanalyze once. If the LCS recoveries are high but the sample results are <QL narrate. Flag with * on Form 3	Laboratory Supervisor / Data Validator	Accuracy/bias	Recovery within lab statistical QC limits; 40% RPD
Internal Standards (IS)	Each field and QC sample	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	Reanalyze affected samples. If similar results, report both runs. Flag outliers with an * on Form 8.	Laboratory Supervisor / Data Validator	Accuracy	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT
Surrogates	1 Per Sample	Benzo(e)pyrene-d12, 45% - 135%	If sample volume available, re-extract. Report both if second successful analysis is outside Holding Time or both fail QC criteria. Flag with * on Form 2.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Benzo(e)pyrene-d12, 45% - 135%

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	PAHs
Analytical Method / SOP Reference	SW846 8270D/ Lab SOP 1

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

SAP Worksheet #28c -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	PCBs
Analytical Method / SOP Reference	SW846 8082/Mitkem SOP 2

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No target analytes > one half of Quantitation Limit	First re-analyze, possibly re-extract batch, qualify data	Laboratory Supervisor	Accuracy/bias- Contamination	No target analytes > ½ QL unless target analytes in field samples are > 10x those in method blank.
Matrix Spike	One per batch of twenty or fewer samples of similar matrix	Recovery within lab statistical QC limits	Check LCS and instrument performance, qualify data	Laboratory Supervisor / Data Validator	Accuracy/bias	Recovery within lab statistical QC limits
Matrix Spike Duplicate	One per batch of twenty or fewer samples of similar matrix	Recovery and RPD within lab statistical QC limits	Check LCS and instrument performance, qualify data	Laboratory Supervisor / Data Validator	Accuracy/bias & Precision	Recovery and RPD within lab statistical QC limits
LCS/LCS Duplicate	One per prep batch of twenty or fewer samples of similar matrix. Include LCSD if prep batch does not include MS/MSD.	Recovery and RPD within lab statistical QC limits	Check instrument performance, reanalyze, if recovery is high and sample is <QL narrate	Laboratory Supervisor / Data Validator	Accuracy/bias	Recovery and RPD within lab statistical QC limits
Surrogates	Two per sample	Within lab statistical QC limits for at least one surrogate DCB: 60-125% TCX: 27-120%	Check instrument performance, re-analyze and qualify data	Laboratory Supervisor / Data Validator	Accuracy/bias	Recovery within lab limits
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	PCBs
Analytical Method / SOP Reference	SW846 8082/Mitkem SOP 2

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No target analytes > one half of Quantitation Limit	First re-analyze, possibly re-extract batch, qualify data	Laboratory Supervisor	Accuracy/bias- Contamination	No target analytes > ½ QL unless target analytes in field samples are > 10x those in method blank.

SAP Worksheet #28d -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	Metals
Analytical Method / SOP Reference	SW846 6010C/Lab SOP 5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Preparation Blank	One per prep batch of twenty or fewer samples of similar matrix	No target analytes > ½ Quantitation Limit	Redigest and reanalyze, if sample concentration is >10X blank concentration narrate.	Laboratory Supervisor	Accuracy/bias-	No target analytes > ½ QL (> QL for common laboratory contaminants), unless target analytes in field samples are > 10x those in method blank.
Interference Check Solutions (ICS-A and ICS-B)	At beginning and end of instrument run and after every 20 samples	ICS-A: Unspiked analytes < 2x MDL (unless they are a verified trace impurity from one of the spike analytes) ICS-AB: %Recovery 80% - 120%	Reanalyze samples analyzed after last acceptable ICSA/ICSAB	Laboratory Supervisor		ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.
Serial Dilution	One per prep batch of twenty or fewer samples of similar matrix	%Difference 90% - 110%	Check instrument performance, Qualify data	Laboratory Supervisor / Data Validator	Accuracy/Bias, Precision	If original sample result is at least 50x MDL, 5-fold dilution must agree within ± 15% of the original result.
Laboratory Control Sample	One per prep batch of twenty or fewer samples of similar matrix	%Recovery 80% - 120%	Redigest and reanalyze, if recovery is high and sample is <QL narrate	Laboratory Supervisor	Accuracy/Bias	80-120 % recovery.
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	RPD <=20%	Check instrument performance, Qualify data	Laboratory Supervisor / Data Validator	Precision	Values ≥ 5xQL: RPD ± 35% Values < 5xQL: Absolute Difference ≤ 2xQL
Matrix Spike Sample	One per prep batch of twenty or fewer samples of similar matrix	%Recovery 75% - 125%	Perform post-digestion spike analysis, qualify data	Laboratory Supervisor / Data Validator	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value

Matrix	Soil/Sediment (Extent Investigation)
Analytical Group	Metals
Analytical Method / SOP Reference	SW846 6010C/Lab SOP 5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Post-Digestion Spike	For elements outside of QC limits in Matrix Spike	%Recovery 75% - 125%	Check instrument performance, Qualify data	Laboratory Supervisor / Data Validator	Accuracy/Bias	Recovery ± 25 % of true value
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C
Preparation Blank	One per prep batch of twenty or fewer samples of similar matrix	No target analytes > ½ Quantitation Limit	Redigest and reanalyze, if sample concentration is >10X blank concentration narrate.	Laboratory Supervisor	Accuracy/bias-	No target analytes > ½ QL (> QL for common laboratory contaminants), unless target analytes in field samples are > 10x those in method blank.
Interference Check Solutions (ICS-A and ICS-B)	At beginning and end of instrument run and after every 20 samples	ICS-A: Unspiked analytes < 2x MDL (unless they are a verified trace impurity from one of the spike analytes) ICS-AB: %Recovery 80% - 120%	Reanalyze samples analyzed after last acceptable ICSA/ICSAB	Laboratory Supervisor		ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.

SAP Worksheet #28e -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Soil/Sediment
Analytical Group	PAHs (Ecological Investigation)
Analytical Method / SOP Reference	8270/8270C-SIM SOC-8270C

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/20 samples or one every batch if less than 20	Positive detects less than the ½ QL	Reanalyze affected samples if they have positive detects of the same analyte	Analyst	Bias/contamination	Positive detects < ½ QL
Laboratory Control Sample (LCS)	1/20 samples or one every batch if less than 20	See Worksheet 12f	Reanalyze affected samples	Analyst	Accuracy	See Worksheet 12f
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1/20 samples or one every batch if less than 20	See Worksheet 12f	Flag data	Analyst	Accuracy/precision/Matrix Interference	See Table
Surrogate Standards	Each field and QC sample	See Worksheet 12f	Reanalyze affected sample	Analyst	Accuracy	See Table
Internal Standards (IS)	Each field and QC sample	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	Reanalyze affected samples	Analyst	Accuracy	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT
Temperature Blank	One per cooler	above freezing to 6° C	Laboratory will notify TtNUS of temperatures outside criteria. TtNUS will respond whether to proceed with analysis and will decide whether to resample. Data may be qualified.	Laboratory project manager, TtNUS Project Manager, data validator	Bias/representativeness	Above freezing to 6° C
Rinsate Blank	1/20 field samples	Positive detects less than the ½ QL	Qualify affected data results	Data validator	Bias/contamination	Positive detects less than the ½ QL
Field Blank	One per water source	Positive detects less than the ½ QL	Data validator notify FOL	Data validator	Bias/contamination	Positive detects less than the ½ QL

SAP Worksheet #28f -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Sediment/Biota (Ecological Investigation)
Analytical Group	Metals
Analytical Method / SOP Reference	6010B/7471A MET-200.7/6010B MET-7471A/245.5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/20 samples or one every batch if less than 20	Positive detects less than the ½ QL	Reanalyze affected samples if they have positive detects of the same analyte	Analyst	Bias/contamination	Positive detects < ½ QL
Laboratory Control Sample (LCS)	1/20 samples or one every batch if less than 20	Dependent on Lot of LCS Standard at time of analysis. Performance Acceptance Limits provided on Manufacturer's Certificate of Analysis. Current lot limits provided in Table	Reanalyze affected samples	Analyst	Accuracy	Dependent on Lot of LCS Standard at time of analysis. Performance Acceptance Limits provided on Manufacturer's Certificate of Analysis. Current lot limits provided in Table
Matrix Spike (MS)	1/20 samples or one every batch if less than 20	Recovery 75-125%	Flag Samples	Analyst	Bias/Matrix Interference	Recovery: 75-125%
Laboratory Duplicate	1/20 samples or one every batch if less than 20	Values ≥ 5xQL: RPD ± 20%6010B/35%7471A Values ≤ 5xQL: Absolute Difference ≤ 2xQL	Reanalyze affected samples	Analyst	Precision	Values ≥ 5xQL: RPD ± 20%6010B/35%7471A Values ≤ 5xQL: Absolute Difference ≤ 2xQL
ICP Serial Dilution	1/20 samples or one every batch if less than 20	If original result is > 50xMDL, the 5xdilution result must agree within ± 10 % of the original value	Reanalyze affected samples	Analysts	Accuracy/bias	If original result is > 50xMDL, the 5xdilution result must agree within ± 15 % of the original value
Temperature Blank	One per cooler	Above freezing to 6° C	Laboratory will notify TtNUS of temperatures outside criteria. TtNUS will respond whether to proceed with analysis and will decide whether to resample. Data may be qualified.	Laboratory project manager, TtNUS Project Manager, data validator	Bias/representativeness	Above freezing to 6° C

Matrix	Sediment/Biota (Ecological Investigation)
Analytical Group	Metals
Analytical Method / SOP Reference	6010B/7471A MET-200.7/6010B MET-7471A/245.5

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Rinsate Blank	1/20 field samples	Positive detects less than the ½ QL	Qualify affected data results	Data validator	Bias/contamination	Positive detects less than the ½ QL
Field Blank	One per water source	Positive detects less than the ½ QL	Data validator notify FOL	Data validator	Bias/contamination	Positive detects less than the ½ QL
MRL Standard	Once at the beginning and once at the end of the analytical run	±50% of the true value	Reanalyze affected samples	Analyst	Accuracy/bias	±50% of the true value
ICSA (ICP only)	Once at the beginning and once at the end of the analytical run	Results <MRL (<2x MRL for metals with MRL<10mg/L)	Reanalyze affected samples	Analyst	Accuracy/bias	Absolute value of concentration for all non-spiked analytes < 2x MDL (unless they are a verified trace impurity from one of the spike analytes)
ICSB (ICP only)	After each ICSA	±20% of the true value	Reanalyze affected samples	Analyst	Accuracy/bias	±20% of the true value
HLCCV1 (ICP only)	Once during daily analysis	±10% of the true value	Reanalyze affected samples	Analyst	Accuracy/bias	±10% of the true value
HLCCV2 (ICP only)	Once during daily analysis	±10% of the true value	Reanalyze affected samples	Analyst	Accuracy/bias	±10% of the true value

SAP Worksheet #28g -- Laboratory QC Samples Table
(*UFP-QAPP Manual Section 3.4*)

Matrix	Sediment/Biota (Ecological Investigation)
Analytical Group	PCB Homologs
Analytical Method / SOP Reference	680 SOC-680

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1/20 samples or one every batch if less than 20	Positive detects less than the ½ QL	Reanalyze affected samples if they have positive detects of the same analyte	Analyst	Bias/contamination	Positive detects < ½ QL
Laboratory Control Sample (LCS)	1/20 samples or one every batch if less than 20	30-130%	Reanalyze affected samples	Analyst	Accuracy	30-130%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	1/20 samples or one every batch if less than 20	30-130%	Flag samples	Analyst	Accuracy/precision	30-130%
Surrogate Standards	Each field and QC sample	30-150%	Reanalyze affected sample	Analyst	Accuracy	30-150%
Internal Standards (IS)	Each field and QC sample	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT	Reanalyze affected samples	Analyst	Accuracy/precision	IS area -50% to +100% compared to IS from CCV; IS RT window ± 0.5 minutes compared to CCV RT
Temperature Blank	One per cooler	Above freezing to 6° C	Laboratory will notify TtNUS of temperatures outside criteria. TtNUS will respond whether to proceed with analysis and will decide whether to resample. Data may be qualified.	Laboratory project manager, TtNUS Project Manager, data validator	Bias/representativeness	Above freezing to 6° C
Rinsate Blank	1/20 field samples	Positive detects less than the ½ QL	Qualify affected data results	Data validator	Bias/contamination	Positive detects less than the ½ QL
Field Blank	One per water source	Positive detects less than the ½ QL	Data validator notify FOL	Data validator	Bias/contamination	Positive detects less than the ½ QL

SAP Worksheet #29 -- Project Documents and Records Table

(*UFP-QAPP Manual Section 3.5.1*)

Document	Where Maintained
Sample Collection Documents and Records: • Field logbook (and sampling notes) • Field sample forms • Chain of custody records • Sample shipment airbills • Equipment calibration logs • Photographs • Field Task Modification Forms • Sampling and Analysis Plan • Field Sampling SOPs	TtNUS project file, results will be discussed in subject document
Laboratory Documents and Records: • Sample receipt/login form • Sample storage records • Sample preparation logs • Standard traceability logs • Equipment Calibration logs • Sample analysis run logs • Equipment maintenance, testing, and inspection logs • Corrective action forms • Reported field sample results • Reported results for standards, quality control checks, and quality control samples • Data completeness checklists • Sample storage and disposal records • Telephone logs • Extraction/clean-up records • Raw data	TtNUS Project File, Long term data package storage at third party professional document storage firm (BRM), results will be discussed in subject document.
Data Assessment Documents and Records: • Field Sampling Audit Checklist (if an audit is conducted) • Analytical Audit Checklist (if an audit is conducted) • Data Validation Memorandum	TtNUS project file, results will be discussed in subject document.

SAP Worksheet #30 -- Analytical Services Table

(*UFP-QAPP Manual Section 3.5.2.3*)

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization[1] (name and address, contact person and telephone number)
Soil/Sediment	GRO, DRO, PAHs, PCBs, Metals	GI-SB401 to 413 GI-SD401 to 450	GRO -5030/8015B DRO- 3541 or Metals-3550B/8015B PAHs-8270/8270C-SIM PCBs-8082	21 days	Mitkem Laboratories 175 Metro Center Boulevard Warwick, Rhode Island 02886-1755 Contact: Edward Lawler Laboratory Operations Manager 401-732-3400, ext. 315	Not applicable
Sediment/Biota	PCB homologues, Metals	GI-ET401 to 424 GI-SD501 to 530	Metals-6010B/7471A PCB homologue - 680	21 days	Columbia Analytical Services 1 Mustard Street, Suite 250 Rochester, NY 14609 585-288-5380 Mark Wilson	Not applicable

Data package deliverables are presented in Table 30-1 below. Data packages will be provided as both hardcopy and portable document format (.PDF). All laboratories will provide a Naval Installation Restoration Information Solutions (NIRIS) compatible electronic data deliverable (EDD). Data packages will be contract laboratory program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). The standard turnaround time for analytical services is 21 calendar days. Turnaround time will be measured from the laboratory receipt of the last samples in a sample delivery group (SDG). SDGs must contain 20 samples (no more than 20 and only less if the entire sampling event was comprised of less than 20 samples). Data will be stored by the analytical laboratory for five years.

Table 30-1

Laboratory Data Package Elements

Data Package Elements	PAHs	PCBs	GRO/DRO	Metals
♦ **INVENTORY SHEET (Org. and Inorg. DC-2 Form)**	X	X	X	X
♦ **NARRATIVE (Org. Narrative, Inorg. Cover Page)**	X	X	X	X
♦ **EPA SHIPPING/RECEIVING DOCUMENTS AND INTERNAL LABORATORY COC RECORDS:**				
- Airbills	X	X	X	X
- Chain-of-Custody Records/Forms (Traffic Report)	X	X	X	X
- Sample Log-In Sheet (Org. and Inorg. DC-1 Form)	X	X	X	X
- Miscellaneous Shipping/Receiving Records	X	X	X	X
- Internal Lab. Sample Transfer Records and Tracking Sheets	X	X	X	X
♦ **SAMPLE DATA:**				
- Tabulated Summary Form for Field Sample and PE Sample Results (Org. and Inorg. Form I)	X	X	X	X
- Tentatively Identified Compounds Tabulate Summary Form (Org. Form I TIC)	X			
- Reconstructed Total Ion Chromatogram (RIC) for each sample	X			
- Raw spectra of target compound and background subtracted spectrum of target compound for each sample	X			
- Mass spectra of all reported TICs/three best library matches for each sample	X			
- Chromatograms from both columns for each sample	X	X	X	
- GC Integration report or data system printouts and calibration plots for each sample	X	X	X	
- PCB Identification Tabulated Summary Form (Org. Form X)		X		
- For PCB confirmed by GC/MS, copies of raw spectra and background subtracted spectrum of target compounds				
- Manual worksheets	X	X	X	X
- Sample preparation/extraction/digestion log (Inorg. Form XIII) and logbook pages	X	X	X	X
- Sample analysis run log (Inorg. Form XIV) and logbook pages	X	X	X	X
- ICP Raw Data				X
- Mercury Raw Data				X
- Other Analytical Raw Data	X	X	X	X

Data Package Elements	PAHs	PCBs	GRO/DRO	Metals
♦ **STANDARDS DATA:**				
- Initial Calibration Tabulated Summary Form (Org. Form VI, Inorg. Form IIA)	X	X	X	X
- Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)	X	X	X	X
- Recontracted Ion Chromatograms and Quantitation Reports for all GC/MS standards	X			
- GC Chromatograms and data system printouts for all GC standards		X	X	
- Instrument Detection Limits Tabulated Summary Form (Inorg. Form X)				X
- ICP Interelement Correction Factors Tabulated Summary Form (Inorg. Form XIA and XIB)				X
- ICP Linear Ranges Tabulated Summary Form (Inorg. Form XII)				X
- CRDL Standards for AA and ICP Tabulated Summary Form (Inorg. Form IIB)				X
- Standards preparation logbook pages	X	X	X	X
♦ **QC DATA:**				
- Tuning and Mass Calibration Tabulated Summary Form (Org. Form V)	X			
- Surrogate Percent Recovery Tabulated Summary Form (Org. Form II)	X	X	X	
- MS/MSD Recovery Tabulated Summary Form (Org. Form III)	X	X	X	
- Method Blank Tabulated Summary Form (Org. Form IV and Inorg. Form III)	X	X	X	X
- Internal Standard Area and RT Tabulated Summary Form (Org. Form VIII)	X			
- QC Raw Data – RICs, Chromatograms, Quan Reports, Integration Reports, Mass Spectra, etc.	X	X	X	X
- Spike Sample Recovery Tabulated Summary Form (Inorg. Form IV)				X
- Duplicates Tabulated Summary Form (Inorg. Form VI)				X
- Internal Laboratory Control Sample Tabulated Summary Form (Inorg. Form VII)				X
- Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)	X	X	X	X
- Standard Addition Results Tabulated Summary Form (Inorg. Form VIII)				X
- ICP Serial Dilutions Tabulated Summary Form (Inorg. Form IV)				X
- QC Raw Data – ICP, Furnace, Mercury computer printouts, etc.				X
- QC sample preparation logbook pages	X	X	X	X

Data Package Elements	PAHs	PCBs	GRO/DRO	Metals
♦ **MISCELLANEOUS DATA:**				
- Original preparation and analysis forms or copies of preparation and analysis logbook pages	X	X	X	X
- Screening records	X	X	X	X
- All instrument output, including strip charts from screening activities	X	X	X	X
- Preparation Logs Raw Data	X	X	X	X
- Percent Solids Determination Log	X	X	X	X
- Other Records (ex. Telephone Communication Log)	X	X	X	X

Notes:

Metals = Target Analyte List Metals
GRO = Gasoline Range Organics
DRO = Diesel Range Organics
PCB = Polychlorinated biphenyls
( ) = Contract Laboratory Program Form Number

SAP Worksheet #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 Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Field Supervision	Daily	Internal	TtNUS	FOL, TtNUS	FOL	FOL and Field Crew	FOL, SM, PM, QA Manager (Kelly Carper)
Project Supervision	Monthly	Internal	TtNUS	PM (Steve Parker), TtNUS	SM (Tom Campbell), TtNUS	SM (Tom Campbell) and FOL	PM (Steve Parker) SM (Tom Campbell), and Program Manager (John Trepanowski), TtNUS
Field Sampling System Audit	1 per contract year	Internal	TtNUS	TBD	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS	Auditor, SM, and PM	TtNUS QA Manager (Kelly Carper)
Laboratory System Audit	Every 18 months	External	NFESC	TBD, NFESC	Laboratory QA Manager or Laboratory Manager	Laboratory Manager and QA Manager	NFESC

- System audits will be performed as appropriate to ensure that the work is being implemented in accordance with the approved project SOPs and in an overall satisfactory manner.
  - The TtNUS FOL will supervise the field operations. The FOL will perform a daily check to ensure that the field instruments are calibrated, equipment is properly decontaminated, samples are collected and handled properly, and the fieldwork is accurately and neatly documented. Corrective actions will be implemented immediately if any con-compliance is detected. The FOL will also check records for reasonable results to ensure that the field crew has time to double check apparent anomalies, if they occur.
  - System audits for the laboratory will be performed regularly and in accordance with NFESC guidance and Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM) (DoD, 2006), as provided in the Laboratory Quality Assurance Plan (LQAP).
  - The data validator(s) will review the data to ensure that the analytical results were obtained through the approved methodology, and that the appropriate levels of QC were followed. The data review effort will be supervised by the TtNUS Project Chemist.

The Project Manager will oversee the FOL and data reviewer, and check that management of the acquired data proceeds in an organized and expeditious manner.

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

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

Performance audits of laboratories are coordinated through NFESC and are conducted every 18 months by NFESC's independent quality assurance contractor.

Assessment findings that require corrective action initiate a sequence of events that include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action effectiveness. The procedures for handling any QAPP deviations and project deficiencies that are identified through the planned project assessments are summarized in Worksheet #32.

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

SAP Worksheet #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**
Field Supervision	Site Log Book	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS	Immediately	Entry in site log book	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS	24 hours
Project Supervision	Written report	Program Manager (John Trepanowski), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Monthly	Written memo	PM (Steve Parker)	Within a week of notification
Field Sampling System Audit	Audit checklist and written audit finding summary	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS FOL TtNUS, Program Manager (John Trepanowski), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Dependant on the finding, if major, a stop work may be issue immediately; however, if minor, within 1 week of audit	Written memo	QAM (Kelly Carper),TtNUS Auditor, TtNUS Program Manager, TtNUS	Within 48 hours of notification
Laboratory System Audit	Written audit report	Laboratory Manager, QA Manager	Not specified by NFESC	Letter	NFESC	Specified by NEFSC

SAP Worksheet #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 SDG	Within 3 weeks	Project Chemist (Lucy Guzman), TtNUS	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS Project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	immediately	QA Manager (TtNUS)	PM (TtNUS), QAM (TtNUS), Program Manger (TtNUS), project file
Project monthly progress report	Monthly for duration of the project	monthly	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	every day that field sampling is occurring	FOL (TtNUS)	PM (Steve Parker), TtNUS SM (Tom Campbell), TtNUS
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	immediately	Subcontracted laboratories	TtNUS, project file

SAP Worksheet #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	Chain-of-custody forms will be reviewed internally and checked against the samples collected to verify complete and accurate information. The forms will be signed by the sampler and a copy will be retained for the project file, the Site Manager, and the data validators. See SOP SA-6.3.	Internal	TtNUS sampler and FOL
SAP Sample tables	Verify that all proposed samples listed in the SAP tables have been collected	Internal	TtNUS, FOL or designee
Sample log sheets	Verify that information recorded in the log sheets is accurate and complete	Internal	TtNUS, FOL or designee
Sample coordinates	Verify that sample locations are correct and in accordance with the QAPP proposed locations.	Internal	TtNUS, FOL or designee
Analytical data packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QA Manager will sign the case narrative for each data package.	External	Laboratory QAM
Analytical data packages	The data packages will be verified for completeness by TtNUS upon data package receipt. Missing information will be requested from the laboratory and validation will be suspended until missing data are received.	Internal	TtNUS data validators (Ann Franke, Paula DiMattei)
Data results	Results in summary forms will be verified against the raw data. Calculations will be checked for accuracy.	External	Laboratory QAM
Electronic data deliverables	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness	Internal	TtNUS data validators (Ann Franke, Paula DiMattei)

Verification includes field data verification and laboratory data verification. Verification inputs as per SAP Worksheet 34 will be checked.

SAP Worksheet #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[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs	Ensure that all sampling SOPs were followed	TtNUS PM, FOL, or designee
IIa	Analytical SOPs	Ensure that the laboratory followed the analytical SOPs cited in the SAP with no modification	TtNUS chemist or data validators
IIa/IIb	Laboratory data packages for accuracy and precision	The validator will ensure that the measurement performance criteria listed in Worsheets 12 were met for all samples and analyses. This includes verification that the specified QC samples were collected and analyzed and verification that the analytical quality control criteria set up for this project were met	TtNUS, data validators (Ann Franke, Paula DiMattei)
IIa/IIb	Laboratory data packages for completeness representativeness	Ensure that the chain-of-custody forms were signed by the samplers and the laboratory sample custodian. Verify that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from collection to analysis	TtNUS, data validators (Ann Franke, Paula DiMattei)
IIb	Project Quantitation Limits for sensitivity	Verify that the project quantitation limits listed in Worksheet #15 were achieved.	TtNUS, data validators (Ann Franke, Paula DiMattei)
IIa/IIb	Project action limits	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as no-detected. Document this usability issue and inform the project manager.	TtNUS Chemist, Lucy Guzman or designee
IIb	Analytical data Deviations	Determine the impact of deviation from the analytical method requirements and matrix interferences effect on the analytical results.	TtNUS Chemist, Lucy Guzman or designee

Step II: Validation

Validation of field measurements and laboratory analytical data is discussed in this section. Validation of field data will be limited to real-time checks in the field as data are generated, whereas laboratory analytical data will be validated in accordance with current applicable USEPA guidance.

Step IIa: Validation Activities

One hundred percent of the laboratory data from chemical analyses will be validated to assure appropriate quality for use in risk assessment and for remedial decisions. Validation of analytical data will be conducted by the TtNUS or data validation subcontractor. Final review and approval of validation deliverables will

be completed by the project chemist. All parameters will be reviewed using applicable sections of the aforementioned guidelines and the laboratory SOPs. Each analysis will be validated in accordance with SAP Worksheet 36.

As part of the validation process, the validator will check that the laboratory has provided all the documentation required to support the reported analytical results. If any documentation is missing from the data package, the data validator will contact the laboratory to request a resubmittal. If the laboratory fails to resubmit the requested information, the data validator will note this on the data validation cover letter. The usability of such data will then be determined by the PM and the Navy.

Data validation will be completed to ensure that the data are of evidentiary quality. Particular emphasis will be placed on holding time compliance, equipment calibration, spike recoveries, and blank results although all required elements of the validation process will be considered.

SAP Worksheet #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	Soil (coal pile area)	DRO/GRO	Tier II data validation. Laboratory-developed criteria for SW-846 8015B or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (U.S. EPA, 1996).	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Soil (coal pile area)	PAHs	Tier II data validation. Laboratory-developed criteria for SW-846 8270/8270C-SIM or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (U.S. EPA, 1996)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Soil (coal pile area)	Metals	Tier II data validation. Laboratory-developed criteria for SW-846 6010B/7471A or Region I, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, February 1989 (U.S. EPA, 1989)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Soil (former rigging platform area) and sediments	PAHs	Tier II data validation. Laboratory-developed criteria for SW-846 8270/8270C-SIM or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (U.S. EPA, 1996)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Soil (former rigging platform area) and sediments	Metals	Tier II data validation. Laboratory-developed criteria for SW-846 6010B/7471A or Region I, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, February 1989 (U.S. EPA, 1989)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Soil (former rigging platform area) and sediments	PCBs	Tier II data validation. Tier II data validation. Laboratory-developed criteria for SW-846 8082 or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (U.S. EPA, 2004)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Biota (clams, mussels, and crabs)	PCB Homologs	Tier II data validation. Laboratory-developed criteria for EPA method 680 or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (U.S. EPA, 2004)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Biota (clams, mussels, and crabs)	Metals	Tier II data validation. Laboratory-developed criteria for SW-846 6010B/7471A or Region I, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, February 1989 (U.S. EPA, 1989)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Sediments (ecological)	PCB Homologs	Tier II data validation. Laboratory-developed criteria for EPA method 680 or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (U.S. EPA, 2004)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Sediments (ecological)	PAHs	Tier II data validation. Laboratory-developed criteria for SW-846 8270C-SIM or Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (U.S. EPA, 1996)	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Sediments (ecological)	Metals	Tier II data validation. Laboratory-developed criteria for SW-846 6010B/7471A or Region I, EPA-NE Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, February 1989 (U.S. EPA, 1989)	TtNUS, Project Chemist (L. Guzman and staff chemists)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Sediments (ecological)	Toxicity Testing	Method specific QC criteria	TtNUS ecological risk assessment staff

SAP Worksheet #37 -- Usability Assessment

(*UFP-QAPP Manual Section 5.2.3*)

Usability Assessment
Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
o Evaluate if the project required quantitation limits listed in Worksheet #15 were achieved for non-detected site contaminants. If no detectable results were reported and data are acceptable for the verification and validation steps, the data will be evaluated further to assess their usefulness.
o If detectable concentrations are reported and the verification and validation steps are acceptable, the data will be evaluated further to assess their usefulness.
o If verification and validation are not acceptable the data are qualified, the data are estimated (J, UJ) for minor QC deviations that do not affect the data usability, or the data are rejected for major QC deviations affecting data usability, the impact of rejected data will be evaluated and re-sampling may be necessary. The use of estimated data will be discussed in the project report.
o For statistical comparisons and mathematical manipulations, non-detect values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate will be used to represent the concentration at that sample location.
o Statistical calculation of the mean and lowest and highest detected concentration will be presented in summary tables in the final Phase 2 RI report.
o Statistical tests will be conducted to identify potential outliers. Potential outliers will be removed if a review of the field and laboratory documentation indicates that the results are true outliers.
o Tabulate unusable data (i.e., data qualified as “R”).
o Evaluate project assumptions that form the basis of the QAPP.
o Identify and evaluate potential data outliers (using statistical tests at the 5 percent significance level, if applicable based on statistician assessment).
o Ensure completion of corrective actions.
o Evaluate effects of deviations from planned procedures and processes on the interpretation and utility of the data.

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

After completion of the data validation and review of the data quality indicators, the data will be reconciled with the method performance criteria (MPC) to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and statistical analyses will be performed to estimate the set characteristics. The statistic evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the project manager 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 also be evaluated to determine whether missing or rejected data and be compensated by other data.

Identify the personnel responsible for performing the usability assessment:

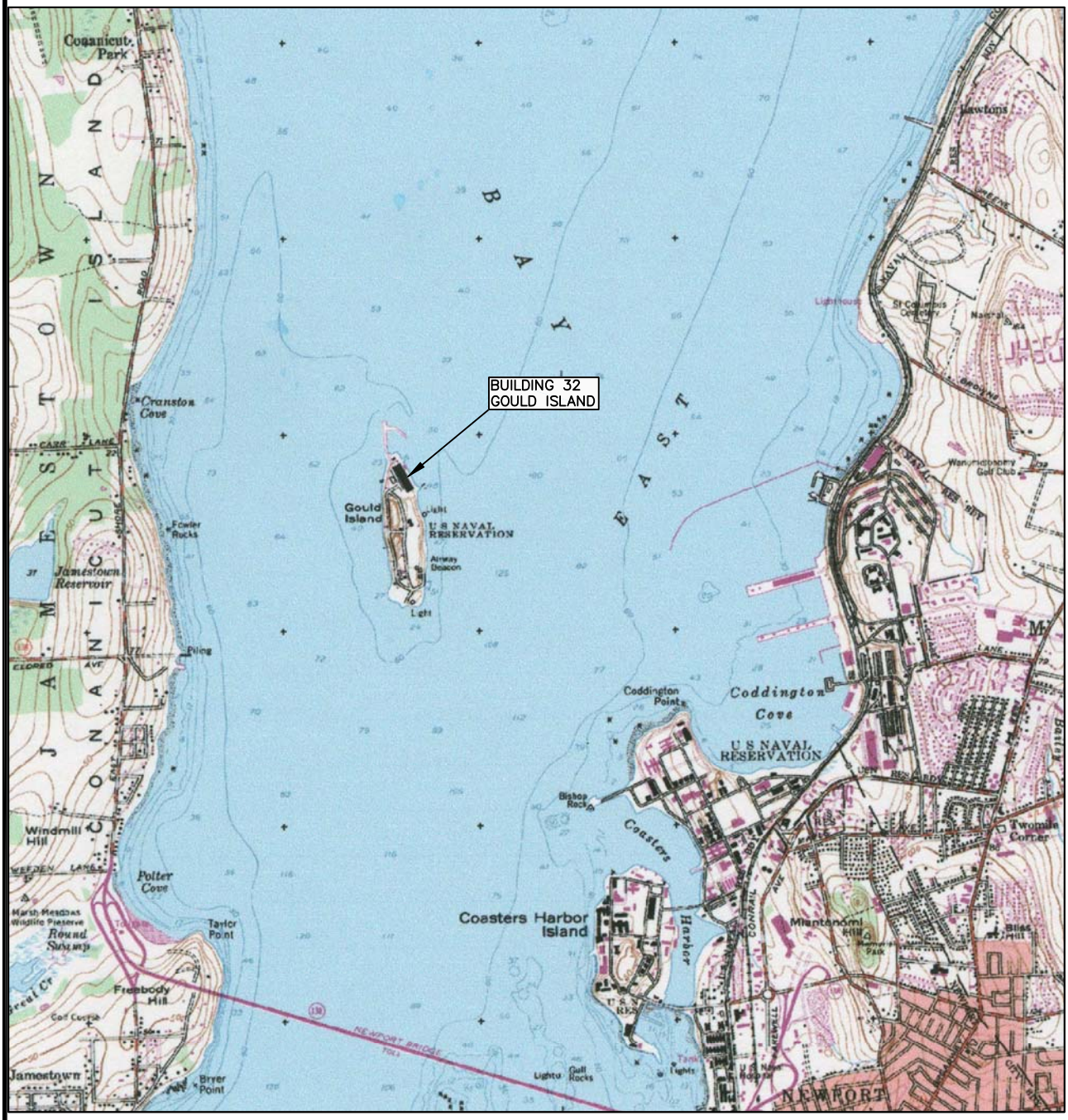
Navy RPM, Federal regulators and their contractors, State regulators and their contractors, TtNUS Project Manager, Project Chemist, Project Geologist, Risk Assessors and others as designated by the Project Manager.

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

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

FIGURES

ACAD: 00949\0210\USGS.DWG 10/20/07 DWM



BASE MAP IS A PORTION OF THE FOLLOWING 7.5 X 15 MINUTE U.S.G.S. QUADRANGLE:
PRUDENCE ISLAND, RHODE ISLAND, 1955, PHOTOREVISED 1970 AND 1975

GRAPHIC SCALE
0 0.5 MILE 1 MILE

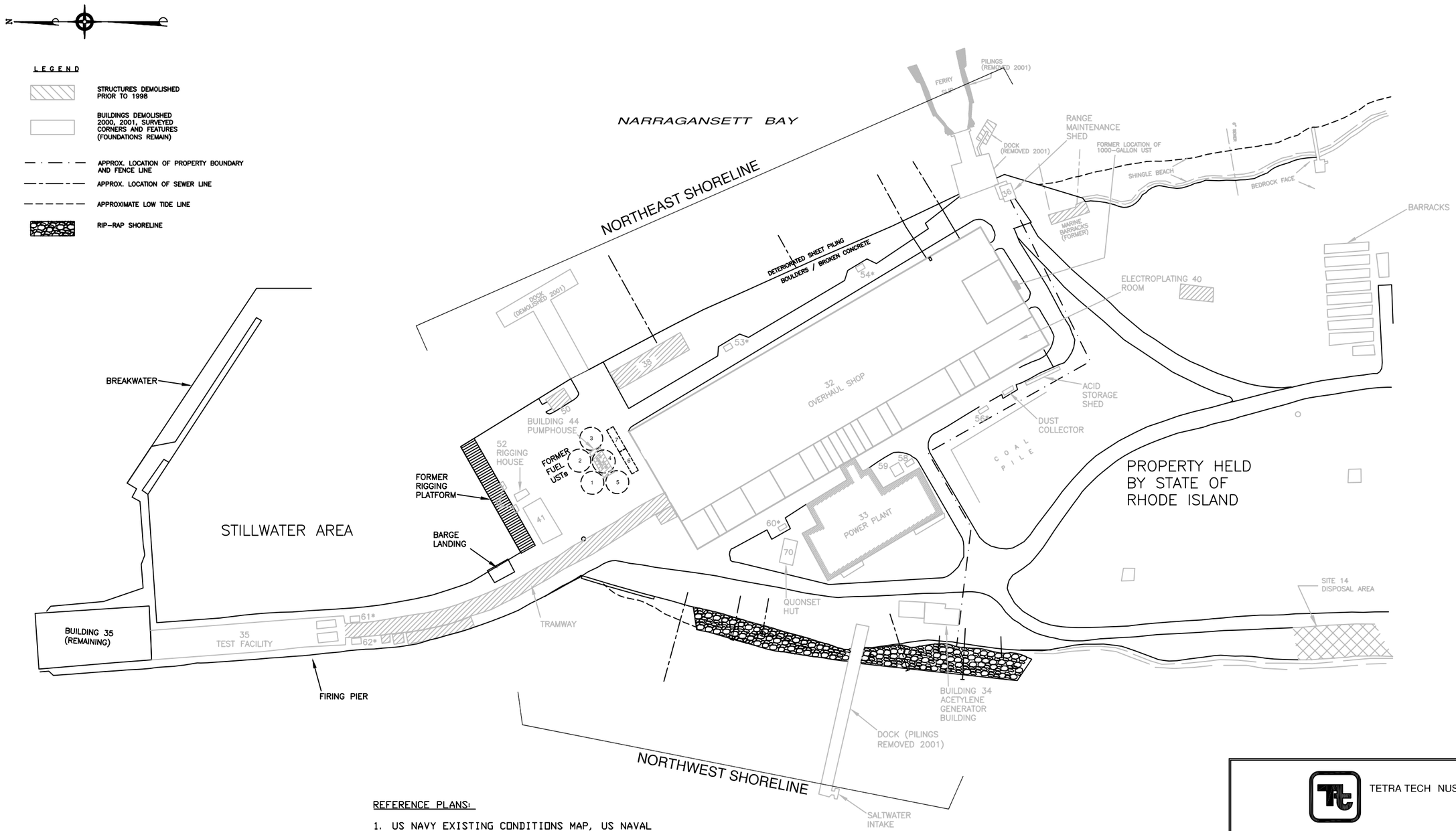
QUADRANGLE LOCATION

TETRA TECH NUS, INC.

SITE LOCUS
SITE 17: BUILDING 32, GOULD ISLAND
PHASE 2 REMEDIAL INVESTIGATION SAMPLING AND ANALYSIS PLAN
NAVSTA NEWPORT, RHODE ISLAND

SCALE	AS NOTED
FILE	00949\0210\USGS.DWG
REV	0
DATE	10/20/07
FIGURE NUMBER	10–1

ACAD: \00949\0230\BASE_MAP.DWG 12/29/09 DWM



NOTES:

1. PLAN NOT TO BE USED FOR DESIGN.
2. ALL LOCATIONS TO BE CONSIDERED APPROXIMATE.
3. PHYSICAL FEATURES SHOWN MAY NOT DEPICT CURRENT CONDITIONS

REFERENCE PLANS:

1. US NAVY EXISTING CONDITIONS MAP, US NAVAL TORPEDO STATION, NEWPORT, RHODE ISLAND, 6/30/47
2. DRAINS, TRENCHES, ETC. GOULD ISLAND BUILDING 32 SITE, LOUIS FEDERICI & ASSOCIATES FOR TETRA TECH, NUS INC. CTO 286, LFA#970706, 4/25/00
3. GEOTECHNICAL SURVEY PLAN AT FORMER BUILDING 44 AND 32, US NAVAL BASE ON GOULD ISLAND, LOUIS FEDERICI & ASSOCIATES FOR BROWN & ROOT ENVIRONMENTAL, 9/8/97, DWG#970706-03
4. LOCATION OF MONITORING WELLS AT BUILDING 32 - GOULD ISLAND, U.S. NAVAL BASE NEWPORT, RHODE ISLAND, LOUIS FEDERICI & ASSOCIATES FOR TETRA TECH, NUS INC., 5/9/00

GRAPHIC SCALE

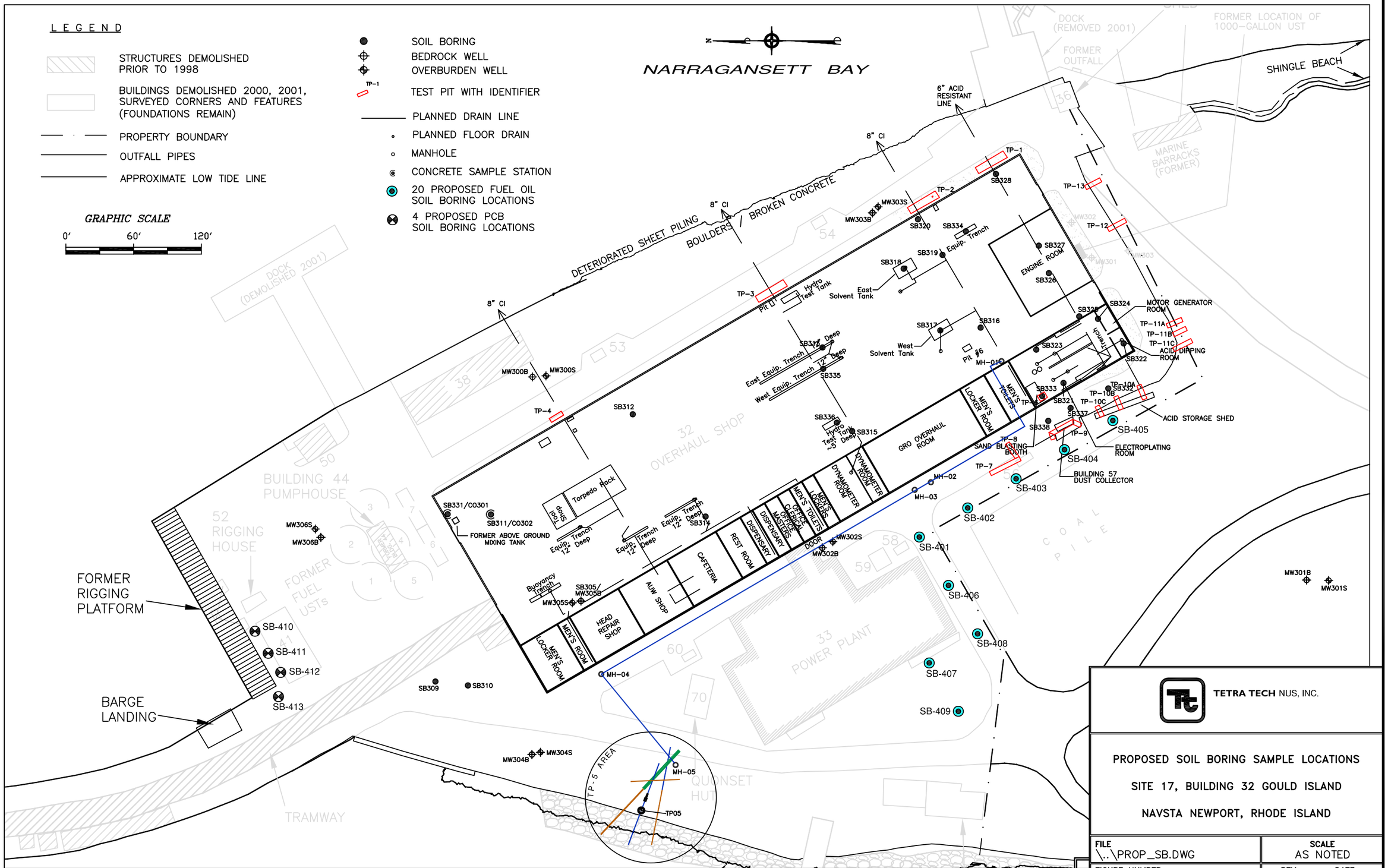
0' 150' 300'

TETRA TECH NUS, INC.

BASE MAP
SITE 17: BUILDING 32, GOULD ISLAND
PHASE 2 REMEDIAL INVESTIGATION SAMPLING AND ANALYSIS PLAN
NAVSTA NEWPORT, RHODE ISLAND

FILE	SCALE
\..\BASE_MAP.DWG	AS NOTED
FIGURE NUMBER 10-2	REV 0 / DATE 12/29/09

ACAD: \00949\0210\PROP_SB.DWG 09/22/09 DWM



TETRA TECH NUS, INC.

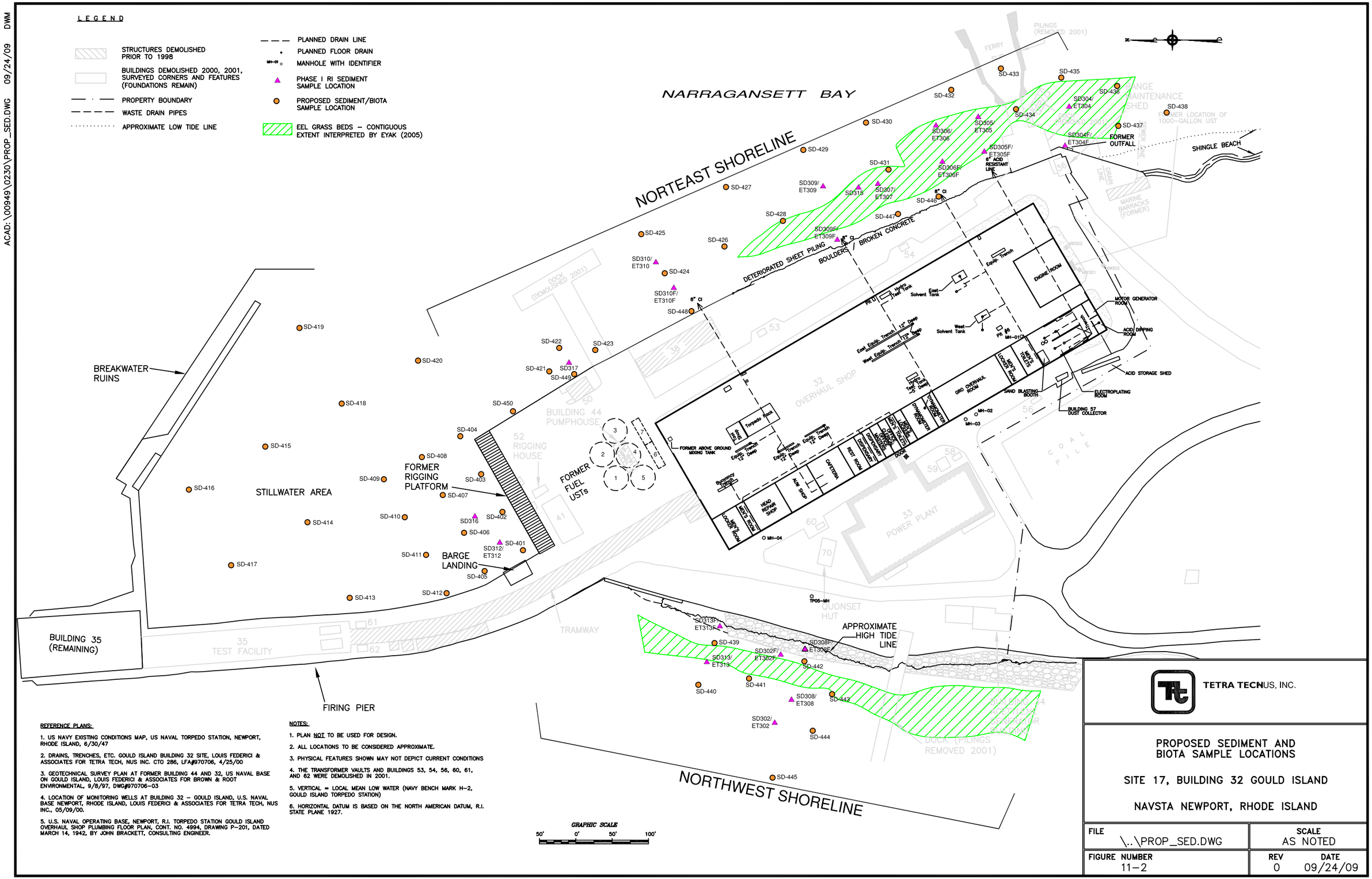
PROPOSED SOIL BORING SAMPLE LOCATIONS

SITE 17, BUILDING 32 GOULD ISLAND

NAVSTA NEWPORT, RHODE ISLAND

FILE	SCALE
\..\PROP_SB.DWG	AS NOTED
FIGURE NUMBER 11-1	REV 0 / DATE 09/22/09

ACAD: \00949\0230\PROP_SED.DWG 09/24/09 DWM



REFERENCE PLANS:

1. US NAVY EXISTING CONDITIONS MAP, US NAVAL TORPEDO STATION, NEWPORT, RHODE ISLAND, 6/30/47
2. DRAINS, TRENCHES, ETC. GOULD ISLAND BUILDING 32 SITE, LOUIS FEDERICI & ASSOCIATES FOR TETRA TECH, NUS INC. CTO 286, LFA#970706, 4/25/00
3. GEOTECHNICAL SURVEY PLAN AT FORMER BUILDING 44 AND 32, US NAVAL BASE ON GOULD ISLAND, LOUIS FEDERICI & ASSOCIATES FOR BROWN & ROOT ENVIRONMENTAL, 9/8/97, DWG#970706-03
4. LOCATION OF MONITORING WELLS AT BUILDING 32 – GOULD ISLAND, U.S. NAVAL BASE NEWPORT, RHODE ISLAND, LOUIS FEDERICI & ASSOCIATES FOR TETRA TECH, NUS INC., 05/09/00.
5. U.S. NAVAL OPERATING BASE, NEWPORT, R.I. TORPEDO STATION GOULD ISLAND OVERHAUL SHOP PLUMBING FLOOR PLAN, CONT. NO. 4994, DRAWING P-201, DATED MARCH 14, 1942, BY JOHN BRACKETT, CONSULTING ENGINEER.

NOTES:

1. PLAN NOT TO BE USED FOR DESIGN.
2. ALL LOCATIONS TO BE CONSIDERED APPROXIMATE.
3. PHYSICAL FEATURES SHOWN MAY NOT DEPICT CURRENT CONDITIONS
4. THE TRANSFORMER VAULTS AND BUILDINGS 53, 54, 56, 60, 61, AND 62 WERE DEMOLISHED IN 2001.
5. VERTICAL = LOCAL MEAN LOW WATER (NAVY BENCH MARK H-2, GOULD ISLAND TORPEDO STATION)
6. HORIZONTAL DATUM IS BASED ON THE NORTH AMERICAN DATUM, R.I. STATE PLANE 1927.

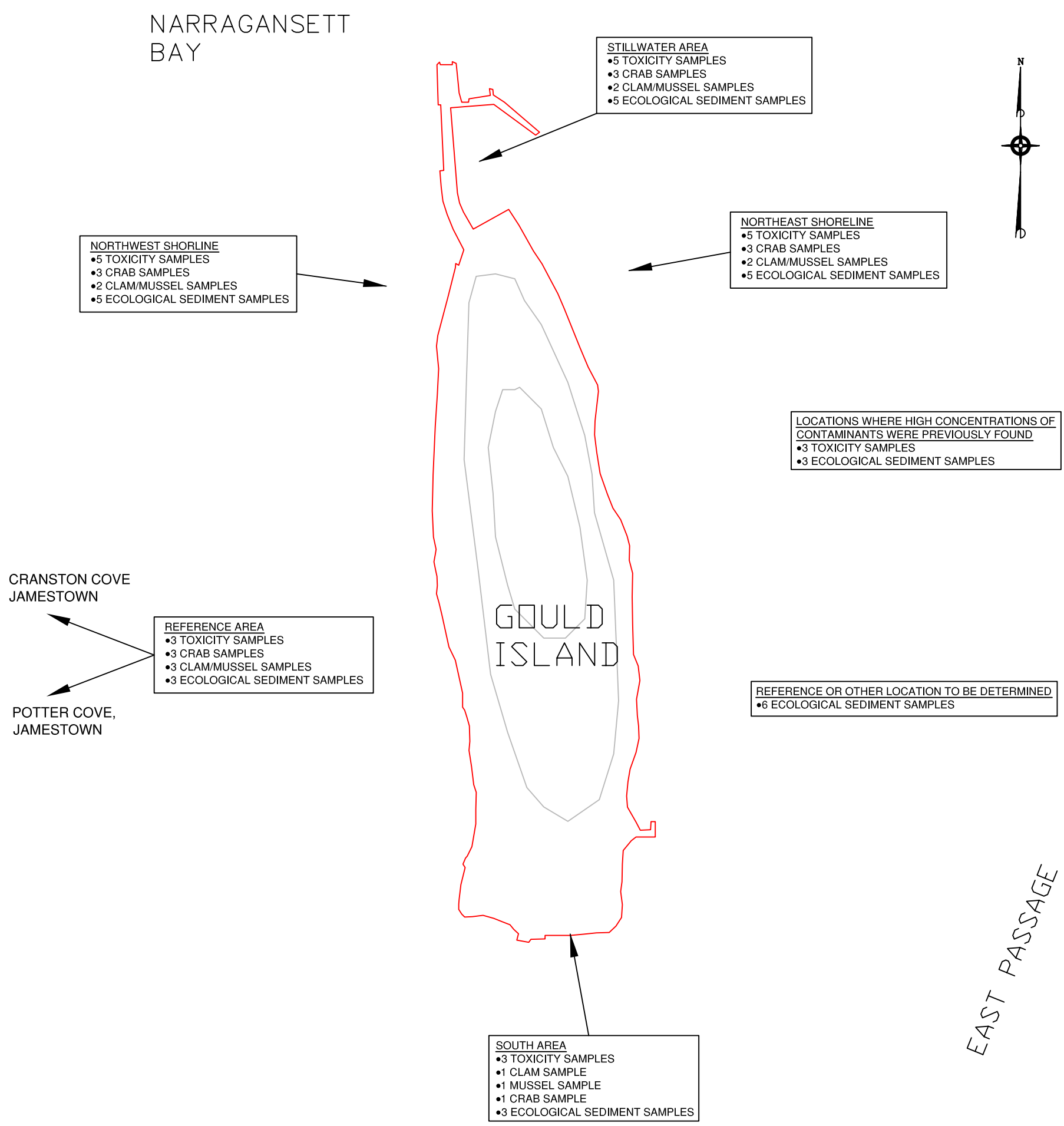
TETRA TECHNUS, INC.

PROPOSED SEDIMENT AND BIOTA SAMPLE LOCATIONS

SITE 17, BUILDING 32 GOULD ISLAND

NAVSTA NEWPORT, RHODE ISLAND

FILE	SCALE
\..\PROP_SED.DWG	AS NOTED
FIGURE NUMBER 11-2	REV 0 / DATE 09/24/09



LEGEND

SHORELINE

SURFICIAL CONTOUR

NOTES

1. ALL LOCATIONS ARE TO BE CONSIDERED APPROXIMATE.
2. PLAN NOT TO BE USED FOR DESIGN.
3. ECOLOGICAL SEDIMENT ANALYSES INCLUDE PCB, HOMOLOGUES, GRAIN SIZE, PAHs, METALS AND TOC.

TETRA TECH NUS, INC.

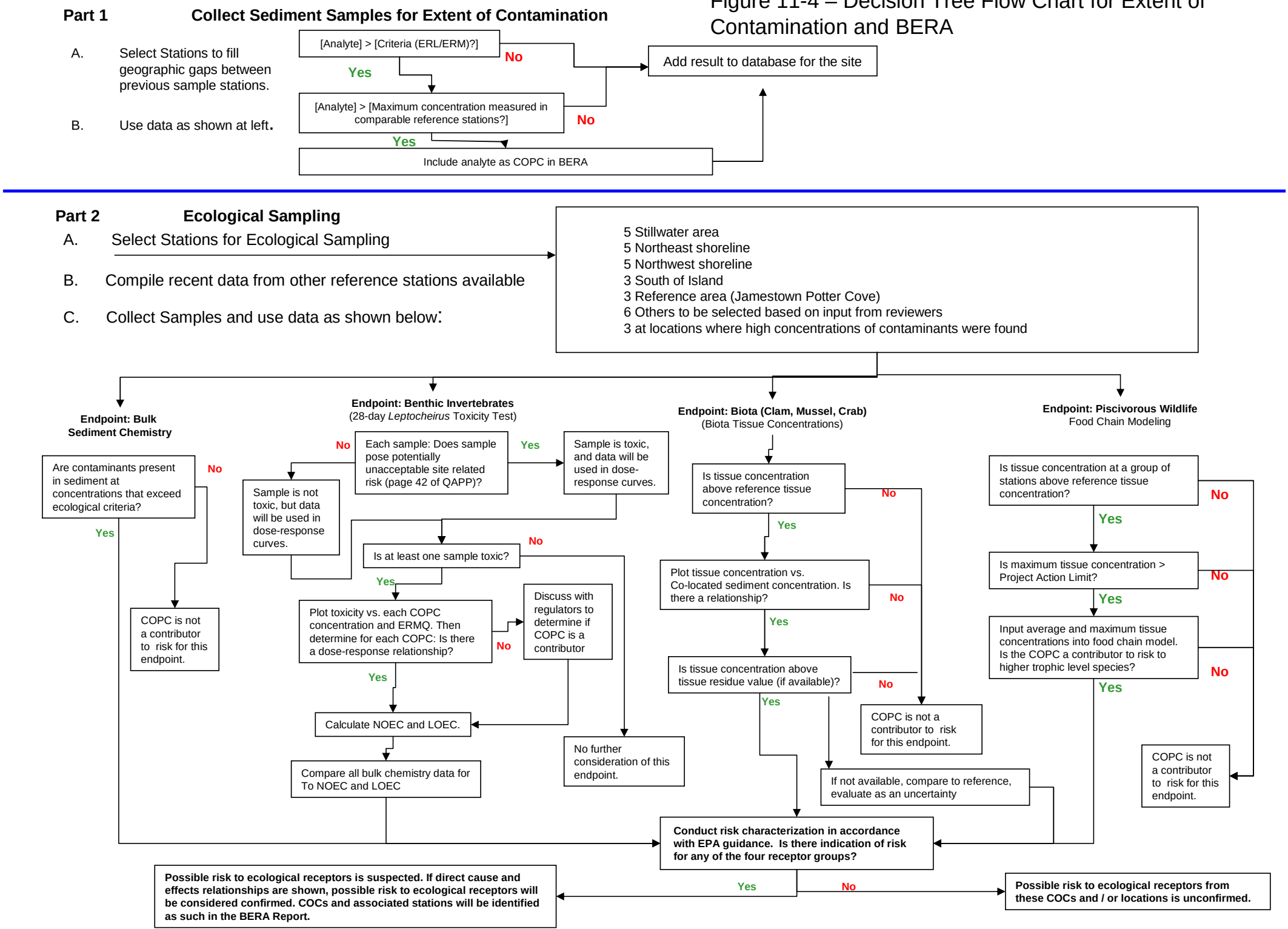
PROPOSED ECOLOGICAL SAMPLE LOCATIONS

SITE 17: BUILDING 32, GOULD ISLAND

NAVSTA NEWPORT, RHODE ISLAND

FILE	SCALE
\..\PROP_ECO.DWG	AS NOTED
FIGURE NUMBER 11-3	REV 0 / DATE 09/24/09

Figure 11-4 – Decision Tree Flow Chart for Extent of Contamination and BERA



REFERENCES

REFERENCES

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APPENDIX A

CONCEPTUAL SITE MODEL

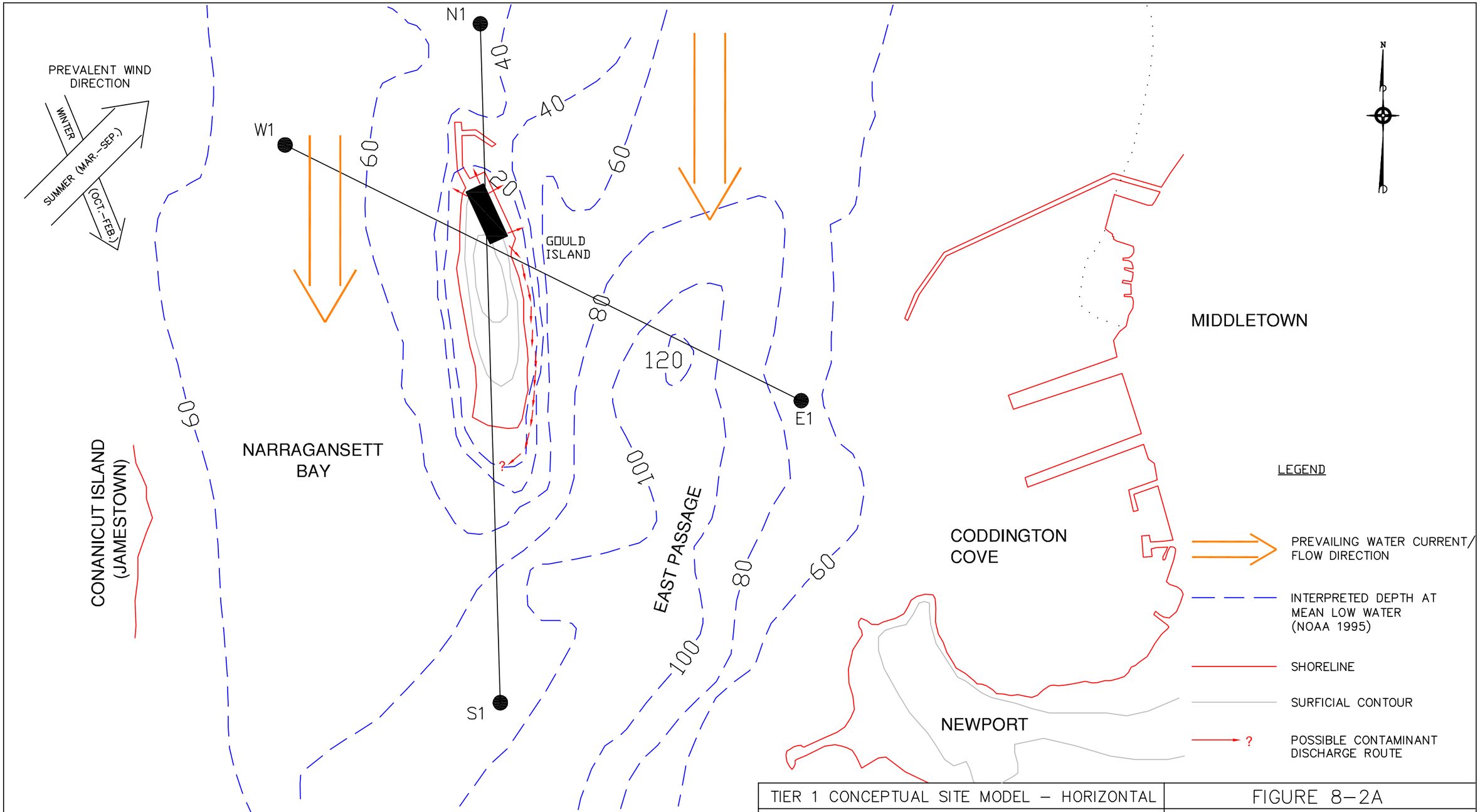
CONCEPTUAL SITE MODEL

Using the information provided in this report, a conceptual site model has been developed to better summarize the understandings of the site conditions and the risks that the site poses.

The conceptual model for this site was developed in the work plan around three two-dimensional views of the site and its surrounding landforms. These figures, provided as Figures 1 and 2, formulate the first tier conceptual site model. Figure 1 presents a plan view of the island and its surrounding influences, and Figure 2 presents cross-sectional views. These views show scale height and subtidal slopes of the island. These figures illustrate, on the simplest level, contaminant inputs to the environment at the site, based on data collected.

The second-tier conceptual site model diagram is presented on Figure 3. This is a schematic diagram that illustrates the general movement of contaminants based on the mechanical systems and physical properties found at the site. Specific sources, flow paths, and discharge areas are identified within the diagram for reference.

Pertinent information specific to the chemical constituents found and the predicted risks they pose to the different receptors is presented on Tables 1 through 5. These tables provide the third tier (fate and transport of contaminants) and fourth tier (predicted risks) conceptual site model. Predicted human health risks and risk drivers are presented for each media on Table 6, to complete the portion of the fourth tier conceptual site model for the human receptors. Similar detail for ecological risk is not currently available and will be incorporated into the conceptual site model after the baseline ecological risk assessment is completed.



NOTES:

1. ALL LOCATIONS ARE TO BE CONSIDERED APPROXIMATE.
2. PLAN NOT TO BE USED FOR DESIGN.
3. BOTTOM CONTOUR DATA INTERPRETED FROM NOAA CHART 13221, APRIL 15, 1995.
4. WIND ROSE INFORMATION FROM NORTHEAST REGIONAL CLIMATE CENTER, CORNELL UNIVERSITY, AUGUST 2000.

GRAPHIC SCALE

0′ 1200′ 2400′

TIER 1 CONCEPTUAL SITE MODEL – HORIZONTAL			FIGURE 8–2A
SITE 17: BUILDING 32, GOULD ISLAND			TETRA TECH NUS, INC.
NAVSTA NEWPORT, RHODE ISLAND			55 Jonspin Road Wilmington, MA 01887 (978)658–7899
DRAWN BY: D.W. MACDOUGALL	REV.: 0		
CHECKED BY: L. SEYDEWITZ	DATE: MARCH 24, 2006		
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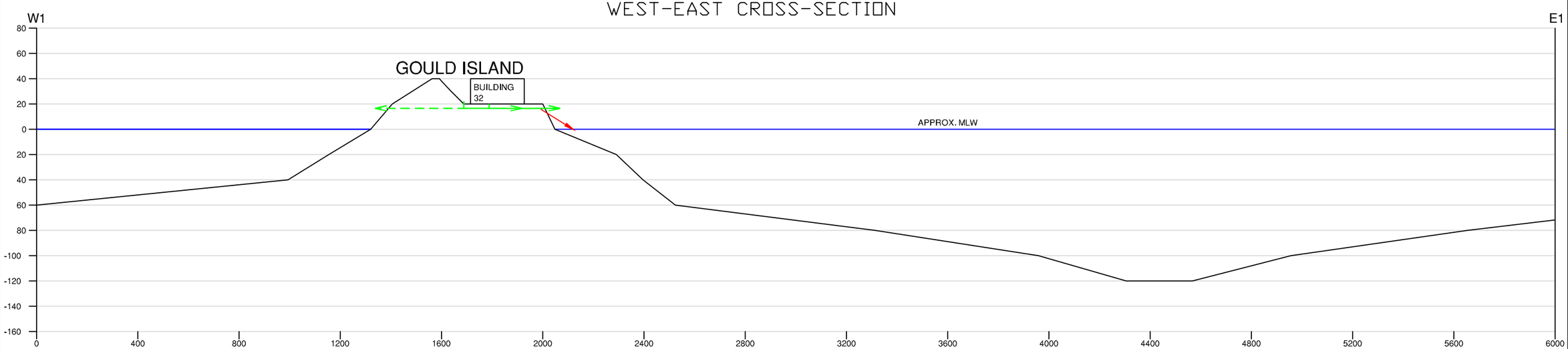
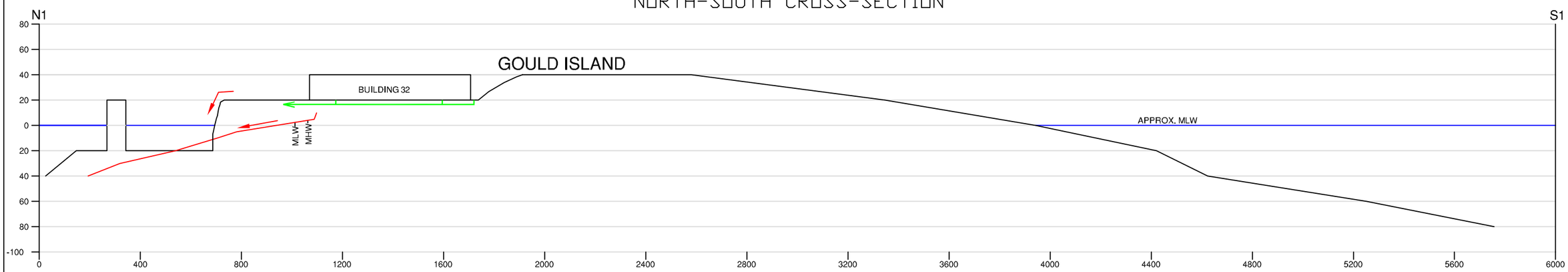
W5206382D

CT035

WEST-EAST CROSS-SECTION



NORTH-SOUTH CROSS-SECTION



L E G E N D

CONTAMINANT DISCHARGE ROUTE

PRE-CONSTRUCTION ELEVATION TAKEN FROM 'GOULD ISLAND FACILITIES GENERAL PLAN', DATED DECEMBER 29, 1941

PIPE

MHW - MEAN HIGH WATER
MLW - MEAN LOW WATER

NOTES:

1. ALL LOCATIONS ARE TO BE CONSIDERED APPROXIMATE.
2. PLAN NOT TO BE USED FOR DESIGN.
3. BOTTOM CONTOUR DATA INTERPRETED FROM NOAA CHART 13221, APRIL 15, 1995.

VERTICAL GRAPHIC SCALE

0′ 80′ 160′

HORIZONTAL GRAPHIC SCALE

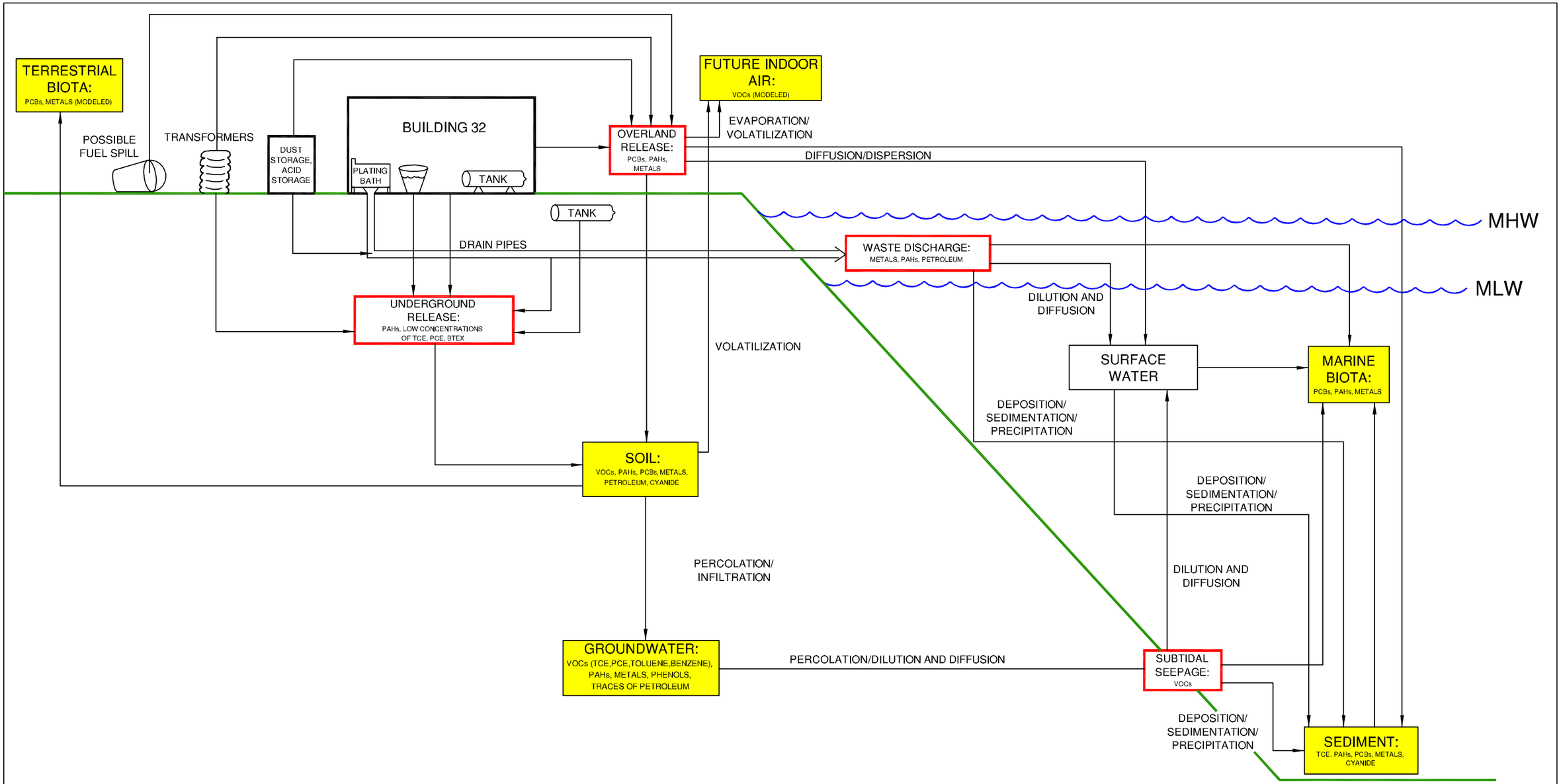
0′ 400′ 800′

TIER 1 CONCEPTUAL SITE MODEL – VERTICAL			FIGURE 8–2B
SITE 17: BUILDING 32, GOULD ISLAND			
NAVSTA NEWPORT, RHODE ISLAND			
DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	L. SEYDEWITZ	DATE:	MARCH 24, 2006
SCALE:	AS SHOWN	FILE NO.:	DWG\00065\0410\FIG_8–2B.DWG

TETRA TECH NUS, INC.
55 Jonspin Road Wilmington, MA 01887
(978)658–7899

W5206382D

CT035



RED BOXES SIGNIFY CONFIRMED CONTAMINANT TRANSPORT ROUTES

YELLOW BOXES SIGNIFY MEDIA IMPACTED BY CONTAMINANTS FOUND (OR PREDICTED BY MODELING)

MHW - MEAN HIGH WATER
MLW - MEAN LOW WATER

TIER 2 CONCEPTUAL SITE MODEL			FIGURE 8-3
SITE 17: BUILDING 32, GOULD ISLAND			
NAVSTA NEWPORT, RHODE ISLAND			TETRA TECH NUS, INC.
DRAWN BY:	D.W. MACDOUGALL	REV.: 0	55 Jonspin Road Wilmington, MA 01887 (978)658-7899
CHECKED BY:	L. SEYDEWITZ	DATE: MARCH 24, 2006	
SCALE:	NOT TO SCALE	FILE NO.: DWG\00065\0410\FIG_8-3.DWG	

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CT035

TABLE 1

THIRD AND FOURTH TIER CONCEPTUAL SITE MODEL – CONTAMINANTS IN SURFACE SOIL
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

SURFACE SOIL: 0-2 FEET

Contaminant	Fate and Transport	Receptors Affected	Predicted Risk
PCBs	Adhere to soil particles, possible erosion and physical transport Hydrophobic, lipophilic Do not decompose Bioaccumulate in animal tissues Do not move with groundwater	None	No Actionable Risk
VOCs	Degrade to breakdown products Chlorinated solvents sink, potentially to bedrock	None	No Actionable Risk
	Lighter solvents dissolve, move with groundwater Volatilize from ground slowly	None	No Actionable Risk
PAHs	Dispersion, dilution Adhere to soil particles, possible erosion and physical transport Hydrophobic and resist dissolution in water Degrade or decompose over time Volatilize (lighter fractions)	Terrestrial Plants, Invertebrates	**Possible Mortality**
		Current and Future Industrial Workers	**Cancer Risk**
Petroleum	Volatilize (lighter fractions) Some fractions may dissolve and move with groundwater	None	No Actionable Risk
Cyanide	Dissolve/Disperse Dissipate in water Remnants may remain	None	No Actionable Risk
Metals	Adsorption to soil Possible erosion and physical transport Precipitation under certain conditions Transport to shoreline through pipeline flow	Terrestrial Plants, Invertebrates	**Possible Mortality**
		Insectiverous Birds	**Uptake of Zinc**
		Current and Future Industrial Workers	**Cancer Risk**

TABLE 2

THIRD AND FOURTH TIER CONCEPTUAL SITE MODEL – CONTAMINANTS IN SUBSURFACE SOIL
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

SUBSURFACE SOIL 0-10 FEET

Contaminant	Fate and Transport	Receptors	Predicted Risk
PCBs	Adhere to soil particles, possible erosion and physical transport Hydrophobic, lipophilic Do not decompose Bioaccumulate in animal tissues Do not move with groundwater	Future Industrial Worker	**Non-Cancer Risk**
VOCs	Degrade to breakdown products Chlorinated solvents sink, potentially to bedrock	None	No Actionable Risk
	Lighter solvents dissolve, move with groundwater Volatilize from ground slowly	None	No Actionable Risk
PAHs	Dispersion, dilution Hydrophobic Degrade or decompose over time Volatilize (lighter fractions) Adhere to soil particles, possible erosion and physical transport	Future Industrial Worker	**Cancer Risk**
Petroleum	Volatilize (lighter fractions) to soil gas Dissolved fraction will move with groundwater Residual petroleum trapped in soil will degrade over time	None	No Actionable Risk
Cyanide	Dissolve/Disperse Dissipate in water Remnants may remain	None	No Actionable Risk
Metals	Adsorption to soil Precipitation under certain conditions Transport to shoreline through pipeline flow Erosion and physical transport of dust particles	Future Industrial Worker Future Construction Worker	**Cancer Risk, Non-Cancer Risk**

TABLE 3

THIRD AND FOURTH TIER CONCEPTUAL SITE MODEL – CONTAMINANTS IN GROUNDWATER
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

GROUNDWATER IN WELLS

Contaminants	Fate and Transport	Receptors	Predicted Risk
VOCs (TCE, PCE, toluene, benzene)	Dissolve/disperse Volatilize Will not bioaccumulate	None	No Actionable Risk
PAHs, Phenols	Soluble fraction will be transported with groundwater, disperse when released to surface water bodies.	None	No Actionable Risk

WATER (AND MODELED AIR EXPOSURES) IN TEST PITS

Contaminants	Fate and Transport	Receptors	Predicted Risk
VOCs	Dissolve/disperse Volatilize Will not bioaccumulate	Future Construction Worker	**Cancer Risk**
PCBs	Adhere to soil particles, possible erosion and physical transport Hydrophobic, lipophilic Do not decompose Bioaccumulate in animal tissues Do not move with groundwater	Future Construction Worker	**Cancer, Non-Cancer Risk**
PAHs	Soluble fraction will be transported with groundwater, disperse when released to surface water bodies.	Future Construction Worker	**Cancer, Non-Cancer Risks**
Metals	Adsorption to soil Precipitation under certain conditions Transport to shoreline through pipeline flow Erosion and physical transport of disturbed and soluble metals	Future Construction Worker	**Non-Cancer Risk**

TABLE 4

THIRD AND FOURTH TIER CONCEPTUAL SITE MODEL – CONTAMINANTS IN SEDIMENT
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

Intertidal Sediment

Contaminants	Fate and Transport	Receptors	Predicted Risk
Solvents, VOCs	Dissolve, Disperse, Volatilize	None	No Actionable Risk
PCBs	Strong adhesion to fine sediments, movement with sediment	Marine Organisms	**Toxicity**
Metals	Disperse Some bioaccumulate	Marine Organisms Future Child Recreational Visitor	**Toxicity** **Non-Cancer Risk**
Cyanide	Dissolve, disperse or precipitate Some adsorption to sediments	None	No Actionable Risk
PAHs	Adhesion to fine sediments, movement with sediment, biodegradation over time	Marine Organisms	**Toxicity**

Subtidal Sediment

Contaminants	Fate and Transport	Receptors	Predicted Risk
Solvents, VOCs	Dissolve, Disperse, Volatilize	None	No Actionable Risk
PCBs	Strong adhesion to fine sediments, movement with sediment	Marine Organisms	**Toxicity**
Metals	Disperse Some bioaccumulate	Marine Organisms	**Toxicity**
Cyanide	Dissolve, disperse or precipitate Some adsorption to sediments	None	No Actionable Risk
PAHs	Adhesion to fine sediments, movement with sediment, biodegradation over time	Marine Organisms	**Toxicity**

TABLE 5

THIRD AND FOURTH TIER CONCEPTUAL SITE MODEL – CONTAMINANTS IN BIOTA
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

Biota

Contaminant	Fate and Transport	Receptors	Predicted Risk
Polychlorinated Biphenyls (PCBs)	Bioaccumulate in animal tissues, will be passed up through the food chain PCBs do not metabolize	Humans Ingesting Mussels or Clams	**Cancer Risk, Non-Cancer Risk**
PAHs	Adhesion to fine sediments, movement with sediment, biodegradation over time, metabolized by many marine organisms	Marine Organisms Humans Ingesting Mussels	**Toxicity** **Cancer Risk**
Metals	Dissolve, disperse or precipitate Some adsorption to sediments, some bioaccumulate	Humans Ingesting Mussels or Clams	**Cancer Risk, Non-Cancer Risk**

TABLE 6

FOURTH TIER CONCEPTUAL SITE MODEL - HUMAN HEALTH RISKS
SITE 17: BUILDING 32, GOULD ISLAND
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

Surface Soil 0-2 feet						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Recreational Visitor	Adult	RME	1.70E-06	NA	<0.01	NA
	Adult	CTE	1.30E-07	NA	<0.01	NA
	Child	RME	3.40E-06	NA	0.03	NA
	Child	CTE	2.40E-07	NA	<0.01	NA
Current Trespasser	Adolescent	RME	2.30E-06	NA	<0.01	NA
	Adolescent	CTE	1.40E-07	NA	<0.01	NA
Current And Future Industrial Worker	Adult	RME	1.10E-05	PAHs, As	0.02	NA
	Adult	CTE	1.10E-06	NA	<0.01	NA

Soil 0-10 Feet						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Industrial Worker	Adult	RME	1.06E-05	PAHs, As	1.4	PCBs, As, Cd, Cr
	Adult	CTE	1.40E-06	PAHs, As	0.5	NA
Future Construction Worker	Adult	RME	5.10E-06	NA	1.4	Cd
	Adult	CTE	7.08E-08	NA	0.3	NA

Subsurface Soil Dust						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Industrial Worker	Adult	RME	2.80E-07	NA	<0.01	NA
	Adult	CTE	3.40E-08	NA	<0.01	NA
Future Construction Worker	Adult	RME	1.18E-03	Cd, Cr	14.50	Cr
	Adult	CTE	1.77E-04	Cd, Cr	2.18	Cr

Shallow Groundwater (From Test Pits)						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Construction Worker	Adult	RME	1.80E-03	PAHs, Pcp	1.40	NA (1)
	Adult	CTE	7.10E-04	PAHs, Pcp	0.23	NA

Pcp = Pentachlorophenol

Trench Air (Modeled from Shallow Groundwater)(2)						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Construction Worker	Adult	RME	4.00E+00	PCBs, PAHs	626.00	PCBs, PAHs
	Adult	CTE	1.20E+00	PCBs, PAHs	19.90	PCBs, PAHs

Intertidal Sediment						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Future Recreational Visitor	Adult	RME	1.20E-06	NA	0.28	NA
	Adult	CTE	8.70E-08	NA	0.01	NA
	Child	RME	2.30E-06	NA	2.36	Chromuim
	Child	CTE	1.64E-07	NA	0.10	NA
Current Trespasser	Adolescent	RME	6.76E-06	NA	0.50	NA
	Adolescent	CTE	1.62E-07	NA	0.02	NA

Clams and Mussels (3)						
Receptor		Type	Cancer Risk and Drivers		Non Cancer HI and Drivers	
Current/Future Clam Ingestion	Adult	RME	1.40E-03	PCBs, As	12.50	PCBs, As
	Adult	CTE	6.50E-05	PCBs, As	1.50	As
	Child	RME	5.10E-04	PCBs, As	18.20	PCBs, As, Th
	Child	CTE	1.90E-05	As	2.00	As
Current/Future Mussel Ingestion	Adult	RME	1.40E-03	PCBs, PAHs, As	23.10	PCBs, As
	Adult	CTE	6.40E-05	PCBs, PAHs, As	2.90	PCBs, As
	Child	RME	5.10E-04	PCBs, PAHs, As	33.80	PCBs, As
	Child	CTE	1.90E-05	As	3.80	PCBs, As

Notes:

(1) No organ specific HI or chemical specific HQ > 1.0 was determined for this receptor
(2) Trench air risk is based on trench dimensions and is biased high
(3) For shellfish ingestion, RME=Subsistence level exposure, and CTE=Recreational exposure
NA - Not Applicable, calculated risk is below acceptable levels.

Yellow shading indicates exceedance of RIDEM acceptable risk (Cancer risk >/= 1E-5).
Red shading indicates exceedance of RIDEM and USEPA acceptable risk
(Cancer risk >/= 1E-4 and Non-Cancer HI >/= 1.0)

APPENDIX B

NAVY ECOLOGICAL RISK ASSESSMENT TIERED APPROACH

NAVY ECOLOGICAL RISK ASSESSMENT TIERED APPROACH
SITE 17: BUILDING 32, GOULD ISLAND
NAVAL STATION NEWPORT, NEWPORT, RHODE ISLAND

RPM Input and Risk Management Consideration[3]

Step 8: Risk Management

Tier 1. Screening Risk Assessment (SRA): Identify pathways and compare exposure point concentrations to benchmarks.

Step 1: Site visit; Pathway Identification/Problem Formulation; Toxicity Evaluation

Step 2: Exposure Estimate; Risk Calculation (SMDP)[1]

Proceed to Exit Criteria for SRA

Exit Criteria for the Screening Risk Assessment (SRA): Decision for exiting or continuing the ecological risk assessment.

1) Site passes SRA. A determination is made that the site poses acceptable risk and shall be closed out for ecological concerns.
2) Site fails SRA: The site must have both complete pathway and unacceptable risk. As a result the site will either have an interim cleanup or moves to the Tier 2.

Tier 2. Baseline Ecological Risk Assessment (BERA): Detailed assessment of exposure and hazard to "assessment endpoints" (ecological qualities to be protected). Develop site-specific values that are protective of the environment.

Step 3a: Refinement of Conservative Exposure Assumptions[2] (SRA)----**Proceed to Exit Criteria for Step 3a**

Step 3b: Problem Formulation - Toxicity Evaluation; Assessment Endpoints; Conceptual Model; Risk Hypothesis (SMDP)

Step 4: Study Design/DQO - Line of Evidence; Measurement Endpoints; Work Plan and Sampling & Analysis Plan (SMDP)

Step 5: Verification of Field Sampling Design (SMDP)

Step 6: Site Investigation and Data Analysis (SMDP)

Step 7: Risk Characterization

Proceed to Exit Criteria for BERA

Exit Criteria Step 3a Refinement

1) If re-evaluation of the conservative exposure assumptions (SRA) supports an acceptable risk determination, then the site exits the ecological risk assessment process.
2) If re-evaluation of the conservative exposure assumptions (SRA) does not support an acceptable risk determination, then the site continues in the BERA process. **Proceed to Step 3b.**

Exit Criteria Baseline Risk Assessment

1) If the site poses acceptable risk, then no further evaluation and no remediation from an ecological perspective is warranted.
2) If the site poses unacceptable ecological risk and additional evaluation in the form of remedy development and evaluation is appropriate, proceed to Tier 3.

Tier 3. Evaluation of Remedial Alternative (RAGS C)

A. Develop site-specific, risk-based cleanup values.

B. Qualitatively evaluate risk posed to the environment by implementation of each alternative (short-term) impacts and estimate risk reduction provided by each (long-term) impacts; provide quantitative evaluation where appropriate. Weigh alternative using the remaining CERCLA 9 Evaluation Criteria. Plan for monitoring and site closeout.

Notes:
1 See U.S. EPA's 8 Steps ERA Process for requirements for each Scientific Management Decision Point (SMDP).
2 Refinement Includes but is not limited to background, bioavailability, detection frequency, etc.
3 Risk management is incorporated throughout the tiered approach.

EXPOSURE PARAMETERS FOR THE TERRESTRIAL WILDLIFE MODEL
SITE 17: BUILDING 32, GOULD ISLAND
REMEDIAL INVESTIGATION REPORT
NAVSTA NEWPORT, NEWPORT, RHODE ISLAND

Species/Exposure Inputs	Conservative Inputs		Average Inputs	
	Values	Units	Values	Units
Raccoon				
Body Weight = BW	3.670E+00	kg	5.636E+00	kg
Food Ingestion Rate = If	2.370E-01	kg/day	1.840E-01	kg/day
Sediment Ingestion Rate = Is	2.228E-02	kg/day	1.730E-02	kg/day
Home Range = HR	Assume 100% on site		1.558E+03	acres
Herring Gull				
Body Weight = BW	9.510E-01	kg	1.092E+00	kg
Food Ingestion Rate = If	3.438E-02	kg/day	3.356E-02	kg/day
Sediment Ingestion Rate = Is	2.819E-03	kg/day	2.752E-03	kg/day
Home Range = HR	Assume 100% on site		1.000E+01	km-radius

Notes:
The sediment ingestion rates were calculated by multiplying the food ingestion rates by the following incidental sediment ingestion rates:

Species	Conservative	Average	Source
Raccoon	9.4%	9.4%	1
Herring Gull	8.2%	8.2%	1, 2

1 - Beyer, N., E. Connor, and S. Gerould. 1994. Estimates of Soil Ingestion by Wildlife. Journal of Wildlife Management 58(2) pp. 375-382.

2 - Based on Canada goose.

DERIVATION OF BODY WEIGHT, FOOD INTAKE (DRY WEIGHT), AND WATER INTAKE FACTORS FOR TERRESTRIAL FOOD CHAIN MODELS

	Data from EPA (1993)			Derivation of Factors for Modeling			
Species/Factor	**Age/Sex/ Cond./Seas.**	**Value**	**Study Average**	**Calculation of Values**			**Notes**
Raccoon							
Body Weight (kg)	A M	7.6	6.7	Minimum Value	3.67	kg	
	A F parous	6.4		Maximum Value	7.6	kg	
	A F nulliparous	6		Overall Study Average	5.636	kg	
	A M	6.76	6.25				
	A F	5.74					
	A M	4.31	3.99				
	A F	3.67					
Food Ingestion Rate (g/g-day)			No Value	Conservative value:	0.237	kg/day	Used maximum body weight in below equation
				Average value	0.184	kg/day	Used average body weight in below equation
							Food ingestion rates were calculated from Nagy et al., (1999) for carnivores as follows: $FI = ((2.23)*BW(g)^{0.85})/1000$
Herring Gull							
Body Weight (kg)	A F spring	951	1068	Minimum Value	0.951	kg	
	A M winter	1184		Maximum Value	1.232	kg	
				Overall Study Average	1.0915	kg	
	A F spring	999	1115.5				
	A M winter	1232					
Food Ingestion Rate (g/g-day)	A M breeding	0.2	0.205	Conservative value:	0.03438	kg/day	Maximum ingestion rate * Average Body weight * 0.15 [(1)]
	A MF breeding	0.21		Average value	0.03356	kg/day	Average ingestion rate * Average Body weight * 0.15 [(1)]
							[(1)] - 0.15 = percent solids in mussels to convert to a dry weight ingestion rate

Notes:
A = Adult
F = Female, M = Male, B = Both
BW = Body Weight

APPENDIX B

SOURCES AND ENDPOINTS FOR NOAELS AND LOAELS FOR WETLAND WILDLIFE
STE 17: BUILDING 32, GOULD ISLAND
REMEDIAL INVESTIGATION REPORT
NAVSTA NEWPORT
NEWPORT RHODE ISLAND

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Primary Reference	Source of Reference
PCBs							
Aroclor-1242	0.685	LOAEL	reproduction	chronic	mink	Bleavins et al., 1980	Sample et.al., 1996
Aroclor-1254	1.8	LOAEL	reproductive	chronic	pheasant	Dahlgren et al., 1972	Sample et.al., 1996
Metals							
Aluminum	109.7	NOAEL	reproductive	chronic	ringed dove	Carriere et al., 1986	Sample et.al., 1996
Aluminum	19.3	LOAEL	reproductive	chronic	mouse	Ondreicka et al., 1966	Sample et.al., 1996
Antimony	0.059	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Antimony	2.76	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2006	
Arsenic	2.24	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Arsenic	4.51	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Arsenic	1.04	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Arsenic	4.55	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Barium	51.8	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Barium	82.7	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Barium	208.26	NOAEL	Survival	subchronic	chicks	Johnson et al., 1960	Sample et.al., 1996
Barium	416.53	LOAEL	Survival	subchronic	chicks	Johnson et al., 1960	Sample et.al., 1996
Beryllium	0.532	NOAEL	Survival	chronic	rat	USEPA, 2005	
Beryllium	0.673	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Cadmium	1.47	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Cadmium	6.35	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Cadmium	0.77	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Cadmium	6.9	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Chromium(III)	2.66	NOAEL	reproduction & growth	chronic	birds	USEPA, 2008	
Chromium(III)	15.63	LOAEL	reproduction & growth	chronic	birds	USEPA, 2008	
Chromium(III)	2.4	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2008	
Chromium(III)	58.17	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2008	
Chromium(VI)	5.66	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2008	
Chromium(VI)	38.37	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2008	
Cobalt	7.61	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Cobalt	18.34	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Cobalt	7.33	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Cobalt	18.9	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Copper	4.05	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Copper	34.76	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Copper	5.82	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Copper	81.42	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	

APPENDIX B

SOURCES AND ENDPOINTS FOR NOAELS AND LOAELS FOR WETLAND WILDLIFE
STE 17: BUILDING 32, GOULD ISLAND
REMEDIAL INVESTIGATION REPORT
NAVSTA NEWPORT
NEWPORT RHODE ISLAND

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Primary Reference	Source of Reference
Iron	500	LOAEL	unknown	chronic	rabbit	NAS, 1980	
Iron	1000	LOAEL	unknown	chronic	poultry	NAS, 1980	
Lead	1.63	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Lead	44.6	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Lead	4.7	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Lead	186.4	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Manganese	179	NOAEL	growth	chronic	birds	USEPA, 2007	
Manganese	376.6	LOAEL	reproduction & growth	chronic	birds	USEPA, 2007	
Manganese	51.5	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Manganese	145.67	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Mercury	0.064	LOAEL	reproductive	chronic	mallard duck	Heinz, 1979	Sample et.al., 1996
Mercury	0.032	NOAEL	reproductive	chronic	rat	Verschuuren et al., 1976	Sample et.al., 1996
Mercury	0.16	LOAEL	reproductive	chronic	rat	Verschuuren et al., 1976	Sample et.al., 1996
Nickel	6.71	NOAEL	reproduction & growth	chronic	birds	USEPA, 2007	
Nickel	18.57	LOAEL	reproduction & growth	chronic	birds	USEPA, 2007	
Nickel	1.7	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Nickel	14.77	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Selenium	0.4	NOAEL	reproductive	chronic	mallard duck	Heinz et al., 1989	Sample et.al., 1996
Selenium	0.8	LOAEL	reproductive	chronic	mallard duck	Heinz et al., 1989	Sample et.al., 1996
Selenium	0.2	NOAEL	reproductive	chronic	rat	Rosenfeld and Beath, 1954	Sample et.al., 1996
Selenium	0.33	LOAEL	reproductive	chronic	rat	Rosenfeld and Beath, 1954	Sample et.al., 1996
Silver	2.02	NOAEL	reproduction & growth	chronic	birds	USEPA, 2006	
Silver	60.47	LOAEL	reproduction & growth	chronic	birds	USEPA, 2006	
Silver	6.02	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2006	
Silver	118.62	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2006	
Thallium	0.74	LOAEL	reproductive	subchronic	rat	Formigli et al., 1986	Sample et.al., 1996
Vanadium	0.344	NOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Vanadium	1.686	LOAEL	reproduction & growth	chronic	birds	USEPA, 2005	
Vanadium	4.16	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Vanadium	7.743	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2005	
Zinc	75.4	NOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Zinc	297.58	LOAEL	reproduction & growth	chronic	mammals	USEPA, 2007	
Zinc	66.1	NOAEL	reproduction & growth	chronic	birds	USEPA, 2007	
Zinc	171.44	LOAEL	reproduction & growth	chronic	birds	USEPA, 2007	

NOAEL = No Observed Adverse Effects Level
LOAEL = Lowest Observed Adverse Effects Level
The LOAELs used for several metals were calculated as the geometric mean of growth and reproduction data from the Ecological Soil Screening Levels (U.S. EPA, 2005, 2006, 2007, 2008).
The TRVs for Aroclor-1254 were used as surrogates for Aroclor-1260; except for the mammal LOAEL, which is based on Aroclor-1242.

APPENDIX B

TRV REFERENCES
SITE 17: BUILDING 32, GOULD ISLAND
REMEDIAL INVESTIGATION
NAVSTA NEWPORT
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APPENDIX C

SELECTED TTNUS STANDARD OPERATING PROCEDURES

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1.0 PURPOSE

The objective of this Standard Operating Procedure is to specify the proper approach and methodologies to identify and quantify airborne chemical contamination levels through the use of direct reading instrumentation and air sample collection. The results of these activities provide vital information for site characterization and risk assessment considerations.

2.0 SCOPE

Applies to all Tetra Tech NUS site activities where the potential for personnel exposures to respiratory health hazards exists.

3.0 GLOSSARY

Direct Reading Instruments (DRIs) - Instrumentation operating on various detection principles such as flame ionization or photoionization providing real time readings of ambient contaminants in air.

Personal/Area Air Sampling - Personal/area air sampling is conducted utilizing an air sampling pump and a specific collection media to quantify airborne contaminants.

Meteorological Considerations - Meteorological information must be collected on site to properly determine air sampling results, as well as aid in the characterization of contaminant potential plume migration and intensity. This information will also be used to support the selection of sampling locations and determine which samples should be analyzed. The meteorological information will be used to estimate downwind concentration levels based on short-term field levels encountered at the source.

4.0 RESPONSIBILITIES

Project Manager (PM) - Responsible for all aspects of project implementation and direction. The project manager is responsible for providing the necessary resources in support of all air monitoring and sampling applications.

Field Operations Leader (FOL) - Responsible for implementing the air monitoring program as detailed in approved project plans for the specific site. Air monitoring requirements will be included in both the Field Sampling and Analysis Plan (FSAP) and the site-specific Health and Safety Plan (HASP).

Health and Safety Officer (HSO) - The health and safety officer provides technical assistance to the FOL concerning air monitoring and sampling applications, collection methodologies, data interpretations, and establishes action items based on results. This information is further used to assess atmospheric migration of airborne chemical contaminants.

5.0 PROCEDURES

5.1 Introduction

Air monitoring is used to help establish criteria for worker safety, document potential exposures, and determine protective measures for the site personnel and the surrounding public. To accomplish this, it is necessary for an effective air surveillance program to be tailored to meet the conditions found at each work site.

During site operations, data are collected concerning air contaminants representative for site operations. Monitoring for vapors, gases, and particulates is performed using DRIs, air sampling systems, and

meteorological considerations. DRIs can be used to detect many organics as well as a few inorganics and can provide approximate total concentrations through applications of relative response ratios of contaminants to reference standards. If specific chemicals (organics and inorganics) have been identified, then properly calibrated DRIs can be used for more accurate onsite assessments.

The most accurate method for evaluating any air contaminant is to collect samples and analyze them at a qualified laboratory. Although accurate, this method presents two disadvantages: (1) cost and (2) the time required to obtain results. Analyzing large numbers of laboratory samples can be expensive, especially if results are needed quickly. Onsite laboratories tend to reduce the turnaround time, but unless they can analyze other types of samples, they may also be costly. In emergencies, time is often not available for laboratory analysis of samples either on site or off site.

To obtain air monitoring data rapidly at the site, DRI utilizing flame ionization detectors (FIDs), photoionization detectors (PIDs), and other detection methodology can be used. Some of these may be used as survey instruments or operated as gas chromatographs. As gas chromatographs, these instruments can provide real-time, qualitative/quantitative data when calibrated with standards of known air contaminants. Combined with selective laboratory analysis of samples, they provide a tool for evaluating airborne organic hazards on a real-time basis and at a lower cost than analyzing samples in a laboratory.

5.2 Air Sampling

For more complete information about air contaminants, measurements obtained with DRIs can be supplemented by collecting and analyzing air samples. To assess air contaminants more thoroughly, air sampling devices equipped with appropriate collection media may be placed at various locations throughout the area and on persons with at-risk occupations. These samples provide air quality information for the period of time they are taken, and can indicate contaminant types and concentrations over the sampling period. As a result, careful selection of sampling types, numbers, and locations, by a qualified health and safety professional is essential to obtain representative information. As data is obtained (from the analysis of samples, DRIs, knowledge about materials involved, site operations, and the potential for airborne toxic hazards), adjustments can be in the types of samples, number of samples collected, frequency of sampling, and analysis required. In addition to air samplers, area monitoring stations may also include DRIs equipped with data logging capabilities and operated as continuous air monitors.

Area air sampling locations may be located in various places as required by project and site needs. Area air sampling stations may include, but are not limited to:

- Upwind - Industrial operations, vehicle traffic, spills and other contributing sources may cause or otherwise result in the generation of air pollutants. Upwind samples establish background levels

- Support Zone (SZ) - Samples may be taken near the command post or other support facilities to ensure that they are, in fact, located in an unaffected area, and that the area remains clean throughout operations at the site.

- Contamination Reduction Zone (CRZ) - Air samples may be collected along the decontamination line to ensure that decontamination workers are properly protected and that onsite workers are not removing their respiratory protective gear in a contaminated area.

- Exclusion Zone (EZ) - The Exclusion Zone presents the greatest risk of release/generation of contaminants and requires the highest concern for air sampling. The location of sampling stations shall be based upon factors such as hot-spots detected by DRIs, types of substances present, and potential for airborne contaminants. The data from these stations, in conjunction with intermittent

walk-around surveys with DRIs, are used to verify the selection of proper levels of worker protection and EZ boundaries as well as to provide a continual record of air contaminants.

- Downwind - One or more sampling stations may be located downwind from the site to indicate if any air contaminants are leaving the site. If there are indications of airborne hazards in populated areas, appropriate response action must be taken and additional samplers should be placed downwind. Downwind locations are further determined based on meteorological considerations concerning generation, air plume migration, and intensity.

5.3 Media for Collecting Air Samples

Hazardous material incidents and abandoned waste sites can involve thousands of potentially dangerous substances, such as gases, vapors, and particulates that could become airborne. A variety of media are used to collect these substances. Sampling systems typically include a calibrated air sampling pump, which draws air into selected collection media. It is essential that appropriate, approved air sampling methodologies (such as those published by NIOSH, OSHA, and EPA) be followed for the collection of each specific analyte. Some of the most common types of samples and the collection media used for them are described in the following information:

One of the most common types of collection media is activated carbon which is an excellent adsorbent for most organic vapors. However, other solid adsorbents (such as Tenax, silica gel, and Florisil) are routinely used to sample specific organic compounds or classes of compounds that do not adsorb or desorb well onto activated carbon. To avoid stocking a large number of sorbents for collecting samples for various chemicals, a smaller number is generally chosen for collecting the widest range of materials or for chemicals known to be present. The vapors are collected using an industrial hygiene personal sampling pump with either one sampling port or a manifold capable of simultaneously collecting samples on several sorbent tubes (provided that sampling parameters such as flow rates and sample volumes are satisfied). For example, in a manifold with four sorbent tubes (or on individual pumps with varying flow rates), the tubes might contain:

- Activated carbon to collect vapors of materials with a boiling point above zero degrees Centigrade. Common materials collected on activated carbon include organic vapors such as solvents, BTEX, and ketones.

- A porous polymer, such as Tenax or Chromosorb, to collect substances (such as high-molecular-weight hydrocarbons, organophosphorus compounds, and the vapors of certain pesticides) that adsorb poorly onto activated carbon. Some of these porous polymers also absorb organic materials at low ambient temperatures more efficiently than carbon.

- A polar sorbent, such as silica gel, to collect organic vapors (aromatic amines, for example) that exhibit a relatively high dipole moment.

- Another specialty absorbent selected for the specific site. For example, a Florisil tube could be used if polychlorinated biphenyls are expected.

- Liquid impingers - aldehydes, ketones, phosgene, phenols.

- Glass fiber filters, membrane filters, Teflon filters - Inorganics and other semivolatile compounds.

- Airborne particulates can be either solid or liquid. Examples of common particulate analytes include some metals, fibers such as asbestos, and condensed particulates such as welding fumes. Dusts, fumes, smoke, and fibers are dispersed solids; mists and fogs are dispersed liquids. For air sampling, most particulates are collected using glass fiber, mixed cellulose ester, or polyvinyl chloride filters,

depending on the filter's ability to collect the subject material and its suitability for laboratory analysis. A cyclone is used to collect particles of respirable size. Atomic Absorption Spectrophotometry, Emission Spectroscopy, Phase Contrast Microscopy, and other techniques are used to analyze various types of particulates. Direct-reading monitors are also used to quantify particulate concentrations, and are usually based on the light-scattering properties of the particulate matter.

5.3.1 Other Methods

Colorimetric detector tubes can also be used with a sampling pump when monitoring for some specific compounds. A detector tube is a vial that contains a chemical preparation that reacts with the measured substance by changing color. Most detector tubes are scale tubes that permit a comparison of the length of the stain to an indicated concentration. Passive organic vapor monitors can be substituted for the active monitoring if they are available for the types of materials suspected to be present at a given site.

5.3.2 NIOSH Methods

The National Institute for Occupational Safety and Health's (NIOSH) Manual of Analytical Methods, 4th ed., contains acceptable methods for collecting and analyzing air samples for a variety of chemical substances. Consult these volumes for specific procedures.

5.4 Collection and Analysis

Collection and analysis of air samples is a multi-faceted task, and is part of the overall air surveillance program. The program is structured to cover the following air pathway analyses:

5.4.1 Selecting Monitoring Constituents

Applications within this program are accomplished using two considerations:

- Air surveillance for specific constituents is based on quantity of the pollutant and the likelihood for vapor release or generation.

- Controlling toxicity - These substances, even when represented in limited quantities, present the greatest threat to the public or worker safety, and influence environmental impact.

5.4.2 Specifying Meteorological Considerations

The following factors will influence sample collection:

- Wind direction and speed
- Sigma theta (atmospheric stability)
- Temperature
- Barometric pressure
- Humidity

These factors will provide information essential to properly arrive at accurate air sampling concentration results. This information is also used to identify how airborne chemical contaminants will react for modeling and for monitoring purposes. The results will provide indicators of plume movement, intensity, and dilution.

5.4.3 Design of Monitoring Network

The air surveillance network is structured to consider:

- Source characteristics (physical state; vapor release and/or generation; emission rates; and disturbance of the source impacting these aspects)
- Receptor sites (receptor sites are monitored and tracked based on priority)
- Meteorological consideration
- Air modeling input
- Data quality objectives

5.4.4 Air Monitoring Documentation/Data Reduction

5.4.4.1 Air Monitoring Documentation

Elements of the air surveillance program are used to provide documentation valuable to safely performing/containing site activities.

Air monitoring results from DRIs must be recorded, such as on instrument results reporting forms, or in the field logbook. This information, where applicable, will be correlated to air sampling information if/when collected.

Air sampling results for personnel and area measurement efforts must be validated, prior to notifying affected individuals. Personal air sampling results notification is accomplished through verbal or written communications.

Results of air monitoring/sampling activities can be identified on site maps. This information is used to structure operational zones and identify levels of protection.

5.4.4.2 Data Reduction

Data reduction combines and correlates the DRI results, air sampling results, and meteorological information to determine area and source airborne contaminant levels and movement.

All air sampling surveillance efforts must incorporate appropriate and approved NIOSH, OSHA, or EPA analytical methods. These procedures identify specific sample collection media, sampling methodologies, and analytical procedures. Sample analysis for health and safety considerations must be further supported by using American Industrial Hygiene Association accredited laboratories.

5.5 Personnel Monitoring

In addition to area atmospheric sampling, personnel monitoring -- both active and passive -- can be used to sample for air contaminants. Representative workers must be identified, and equipped with appropriate personal sampling systems to determine contaminants at specific locations or for specific work being performed. When sampling devices are placed on workers (generally within 1 foot of the mouth and nose) the results are used to indicate worker exposures.

5.6 Calibration

As a rule, the entire air sampling system shall be calibrated. Proper pre-and post-calibration activities are essential for correct operation and for accurate data. In some instances, additional calibration during the sampling period may be required. The overall frequency of calibration will depend upon the particular sampling event, including the general handling and use of a given sampling system. Pump mechanisms shall be calibrated after repair, when newly purchased, and following suspected abuse. All DRIs will be calibrated according to manufacturers instructions. All calibration activities for both air monitoring and sampling equipment must be properly documented, such as through the use of a calibration form. This form will be kept on site throughout the life of the project. The calibration log will be submitted as documentation that instrument calibration was performed on a regular basis.

5.7 Meteorological Considerations

Meteorological information is an integral part of an air surveillance program. Data concerning wind speed and direction, temperature, barometric pressure, and humidity (singularly or in combination) are needed for:

- Selecting air sampling locations
- Calculating accurate air sampling results
- Calculating air dispersion
- Calibrating instruments
- Determining population at risk or environmental exposure from airborne contaminants

Knowledge of wind speed and direction is necessary to effectively place air samplers. In source-oriented ambient air sampling, samplers need to be located downwind (at different distances) of the source and others need to be placed to collect background samples. Shifts in wind direction must be known. Consequently, the samplers must be relocated or corrections made for these shifts. In addition, atmospheric simulation models for predicting contaminant dispersion and concentration need windspeed and direction as inputs for predictive calculations. Information may be needed concerning the frequency and intensity that winds blow from certain directions (windrose data). Consequently, the wind direction must be continually monitored when use of this type of data is contemplated.

Air sampling systems need to be calibrated before use. This must include corrections in the calibration curves for actual temperatures and pressures during the sampling event. After sampling, collected air volumes are also mathematically corrected for temperature and pressure conditions.

Air sampling is sometimes designed to assess population exposure (and frequently potential worker exposure). Air samplers are generally located in population centers, irrespective of wind direction. Even in these instances, however, meteorological data is needed for air dispersion modeling. Models are then used to predict or verify population-oriented sampling results.

Proper data is collected by having meteorological stations on site or by obtaining the information from one or more of several government or private organizations, which routinely collect this data. The choice of how information is obtained depends on the availability of reliable data at the location desired, resources needed to obtain meteorological equipment, accuracy of information needed, and use of information.

The collection, handling, and analysis of air samples is an intricate, involved process. Appropriate methodologies, media, and equipment must be used to collect accurate data. Furthermore, selection of appropriate numbers, types, and locations of samples is essential if the data collected are to be used for personnel exposure criteria. For these reasons, air sampling activities must be coordinated and conducted by properly qualified and experienced industrial hygiene professionals. Air monitoring activities also need to be established and monitored carefully. However, as the proper use of these instruments is

not as complicated as air sampling, it is commonly acceptable to cross-train capable environmental professionals to use DRIs, with adequate technical support provided by health and safety professionals.

6.0 REFERENCES

Standard Operating Safety Guides, EPA, November 1984.
NIOSH Manual of Analytical Methods, 4th Edition.

7.0 ATTACHMENTS

None.

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

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

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Drilling Area Background (ppm): ____

Remarks: _____

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

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size				
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES
GRAVELS (50% or more of coarse fraction retained on No. 4 sieve)	CLEAN GRAVELS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.
	GRAVELS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.
SANDS (50% or more of coarse fraction passes No. 4 sieve)	CLEAN SANDS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.
	SANDS WITH FINES (More than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.

FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size						
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)					GROUP SYMBOL	TYPICAL NAMES
Identification Procedures on Fraction Smaller than No. 40 Sieve Size						
	DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)			
SILTS AND CLAYS Liquid Limit < 50	None to Slight	Quick to Slow	None		ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
	Medium to High	None to Very Slow	Medium		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Slight to Medium	Slow	Slight		OL	Organic silts and organic silt-clays of low plasticity.
SILTS AND CLAYS Liquid Limit > 50	Slight to Medium	Slow to None	Slight to Medium		MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
	High to Very High	None	High		CH	Inorganic clays of high plasticity, fat clays.
	Medium to High	None to Very Slow	Slight to Medium		OH	Organic clays of medium to high plasticity.
HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.				Pt	Peat and other organic soils

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS	
DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS			
CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)		
Descriptive Terms	Scratcher or Knife Effects	Hammer Effects
Soft	Easily Gouged	Crushes when pressed with hammer
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges
Medium Hard	Can be scratched	Breaks (one blow); sharp edges
Hard	Cannot be scratched	Breaks considerably (several blows); sharp edges

ROCK BROKENNESS		
Descriptive Terms	Abbreviation	Spacing
Very Broken	(V.Br.)	< 2"
Broken	(Br.)	2"-6"
Blocky	(Bl.)	6"-30"
Massive	(M.)	>30"

LEGEND :

SOIL SAMPLES - TYPES	ROCK SAMPLES - TYPES	WATER LEVELS	
5-2" Split Barrel Sample	X-NX (Conventional) Core (2-1/8" O.D.)	12/94	
ST(3" O.D. Undisturbed Sample	O-NQ (Wireline) Core (2-1/8" O.D.)		▼ 12.5' Initial Level w/Date & Depth
O - Other Samples, Specify in Remarks	Z - Other Core Sizes, Specify in Remarks	12/94	
			▼ 12.5' Stabilized Level w/Date & Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
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 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 shall be divided into 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 shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges 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 shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall 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." Since 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 shall 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" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 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 noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. 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 Figure 2.

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

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer 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. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised 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

FIGURE 2

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

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.

5.2.5 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 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 drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall 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
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).

- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm) Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR: TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810	6.0	5/7	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
	•	9/8		7' ▽									
	8.0			8.0									
S-3 @ 0820	10.0	6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER: 7'±	0	0	0	0
	12.0	17/16		12.0									
S-4 @ 0830	14.0	7/6 5/8	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
	15.0			15.0					AUGER REF @ 15'				
9/5	①		4.0/5.0	16	M HARD	BRN	SILTSTONE	VBR	WEATHERED LO ✱ JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H2O @ 17'± SET TEMP 6" CAS TO 15.5				
4.0/5.0	②		5.0/5.0						SET 2"Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z 1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min, RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: _____ Request Date: _____
CTO: _____ Date Data Available for Production: _____
Project Manager: _____ Request in Support of: _____
Requestor: _____ Database Lead: _____
Program/Client: _____ GIS Lead: _____
State/EPA Region: _____ Statistics Lead: _____
Risk Lead: _____
Site Name(s) (Area, OU, etc.): _____
Sampling Date(s): _____
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other: _____

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions: _____

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other: _____
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other: _____
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions: _____

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - Is a solution selected/identified within the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

Potable Water - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

Solvent - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Failure to meet these objectives are sufficient to restrict equipment from entering the site/exiting the site/ or moving to a new location on the site until the objectives are successfully completed.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide the following characteristics

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

  - Liner – Depending on the types of equipment and the decontamination method the liner should be of sufficient thickness to provide a puncture resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. Achieving the desired thickness maybe achieved through layering lighter constructed materials. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner a light coating of sand maybe applied to provide traction as necessary.

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.

2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable*.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

---

* - The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

1) Wash with soap and water
2) Rinse with tap water
3) Rinse with deionized water

Note: In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer's instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.

- Cleanliness – As per protocol only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or associated with noticeable odors it should be decontaminated prior to use.

  1) Wash with soap and water
  2) Rinse with tap water
  3) Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole Drilling Equipment

This includes any portion of the drill rig that is over the borehole including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

During the course of field investigations disposable/single use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- Rinsate Blanks – It is recommended that Rinsate samples be collected to

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling 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 and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (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. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

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

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

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

Photo Ionization 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.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 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.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

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 Project Manager in accordance with the project-specific plan.

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

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

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

- Attach the metal trough from the Geoprobe® Sampling Kit 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 that accompanies the Geoprobe® Sampling Kit. 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, and observe/examine the sample (according to SOP GH-1.3). 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 organics 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 cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

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.

6.2 Sampling Equipment

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

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

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ($\pm$10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____
TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required
Full face APR ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern | Action Level(s) | Response Measures

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

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

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

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

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a 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 transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which 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 used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B

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

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR ___	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (~~y~~261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (~~y~~261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11], [12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11], [14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (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 in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. 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.

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

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

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

001 - Monitoring Well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

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

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to 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.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

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 routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site specific planning documents.

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A SITE RECONNAISSANCE CHECKLIST 6

1.0 PURPOSE

The purpose of a site reconnaissance is to collect both general and technical information which will support the scoping, scheduling, implementing project activities, and writing reports for an environmental investigation. This procedure is not intended as a guide for Phase I investigations or for Environmental Baseline Survey activities.

2.0 SCOPE

This procedure is applicable to the performance of a site reconnaissance for initial site characterization. The steps necessary to develop and carry out a site reconnaissance are presented here. These steps include a list of equipment and items which may be needed, areas of special interest during field observations, and methods by which the field observation team can ensure that necessary and appropriate observations have been made.

3.0 GLOSSARY

Site reconnaissance. An onsite inspection program used to identify site-specific conditions that control scheduling, manpower, and affect costs. A site reconnaissance usually consists of visual observations and, often, the use of field monitoring instruments to identify potential health and safety threats and potential sampling locations for site evaluation during subsequent field investigations.

4.0 RESPONSIBILITIES

Field Operations Leader (FOL) is responsible for ensuring that the survey is carried out in sufficient detail. To accomplish this, the FOL must assign the proper personnel and equipment to characterize the site adequately, in accordance with the requirements defined in this procedure and best engineering practices. Other disciplines which may be applicable include (but are not limited to): Geology/Hydrogeology; Health and Safety; Ecological Specialists; and/or Engineering. In addition, the FOL is responsible for supervising equipment preparation, including necessary calibrations, and supervising field data collection and documentation in accordance with the methods described in all referenced standard operation procedures.

Project Manager is responsible for the following:

- Supervising the retrieval and examination of available, applicable information regarding the site.
- Obtaining appropriate program approvals and ensuring the preparation of a site Health and Safety plan for the site reconnaissance.
- Coordinating the field activities with the client and regulatory agencies, as applicable.

Field Personnel are primarily responsible for observing and documenting, either through written documentation or photographic evidence, the site reconnaissance. Field personnel will take direction from the FOL.

5.0 PROCEDURES

5.1 Equipment Items/Needed

Below is a list of items that may be useful when conducting a site reconnaissance. All, or a portion of these items may be required, depending upon the objective of the site reconnaissance.

- Health and Safety equipment and information as required by the Site Safety Officer.
- Maps (U.S.G.S. quadrangle, geologic maps, street and highway maps, and client facility maps).
- Geologic tools (compass, tape measure, hand level, camera, etc.).
- Physical monitoring equipment, if applicable (PID, Immunoassay Test Kits, etc.)
- Regional publications (U.S.G.S reports, water well surveys, U.S.D.A. soil conservation surveys, etc.).
- Site-specific publications by previous investigators (EPA aerial photographic analyses, remedial investigation reports, data on waste disposal practices, boring logs, etc.).
- Marking items (ink markers, surveyor's flagging, spray paint, pin flags, wooden stakes).
- Field notebooks.
- Local telephone book with yellow pages (for obtaining utilities, site trailer, living accommodations, etc.).

Sufficient time will be required in order to obtain some of the aforementioned material. In general, most publications can be obtained in time to be used in the site reconnaissance if ordered approximately 2 weeks before the actual site visit takes place.

5.2 Observations

A site reconnaissance usually requires one to two days, however, additional time may be needed depending upon the objective, site size, etc. The following observations, when applicable, should be documented either on a site map, field notebook, or photographed.

- General Site Access. It should be noted whether site roads provide access to all proposed work locations, or if it will be necessary to prepare access roads with either a backhoe, dozer, chain saws, etc., in order to get drill rigs, excavators, or other work vehicles to specific locations. If temporary driveways must be constructed from existing public roads, regulatory permits may be required. Military facilities may have specific security requirements which require detailed clearance procedures.
- Location of the Command Post or Site Trailer and Sanitary Facilities. The ideal location for the site trailer and sanitary facilities is a level area, within an uncontaminated zone, and centralized in order to provide easy access to work areas on the site. However, certain utility companies may require that the site trailer be placed within a specified radius (usually 100 feet), of the nearest utility pole. Contact the necessary utility companies and inquire about the requirements regarding service before conducting the site reconnaissance. Information that may be required by the utility companies is: type of electric service needed (inquire with trailer vendor for this information); and utility pole number of interest (pole numbers are usually stamped on a brass plate on the pole).

- Potable Water Sources. Local fire departments may allow access to fire hydrants. Private water delivery companies may also be available in the area.

- Sources of Possible Contamination. Drums, tanks, sludge areas, areas of stressed vegetation, fill areas, and leachate seeps may indicate where sources of contamination exist. Filler pipes protruding from the ground surface may indicate the presence of underground storage tanks. Areas where the original ground surface has been reworked may be contaminated fill areas that have since been buried and covered with natural material. Previous environmental investigations may also identify source areas.

- Location of Decon Areas and Storage/Disposal Areas for Equipment and Wastes Generated by Field Activities.

- Locations of Surface Water Bodies. The locations of surface water bodies, both man-made and natural, and their relation to topographic highs may give an indication of the groundwater flow direction in the area (groundwater flow typically follows topography with the topographic highs serving as groundwater recharge areas, and the surface waters at topographic lows serve as groundwater discharge areas). Visible signs of contamination, the existence of aquatic life, flow rates, and approximate levels should also be observed and noted. Check if the surface water bodies could potentially be impacted by field activities. If so, appropriate sedimentation and erosion controls will be required.

- Existing Wells. Existing monitoring wells, or domestic wells within the site and off site, should be noted on a map, and access checked to see if the wells can be used for data collection.

- Outcrops. Outcrops can be useful in providing hydrogeologic data (lithologic description, strike and dip information, fracture and joint system analysis, identification of moist zones, etc.) Outcrops may occur naturally or be a part of a man-made feature such as a road-cut.

- Lineaments. A lineament is a straight lengthy feature on the earth's surface which is expressed topographically as a line of depression. Stream beds, vegetation patterns or soil characteristics may be aligned or controlled by this feature. Lineaments are due in some cases to the presence of intense jointing or faults beneath the ground surface. Groundwater in the bedrock may follow lineaments. Lineaments should be noted on site maps and described in the field notebooks.

- Bench or Property Markers. Benchmarks or property markers should be marked with paint or surveyor's flagging if encountered during a site reconnaissance. Surveyors may need to use these markers as a reference point when surveying. Benchmarks are typically a brass plate secured in concrete in the ground with numbering on the top. Property markers can range from a stake driven into the ground to a rock protruding from the ground surface. Facility contacts may also be aware of local benchmarks used during the course of other environmental or public work projects.

- Metal Cultural Effects. Overhead power lines, railroad tracks, junk automobiles, fences, etc. will greatly affect certain geophysical surveys. These features should be noted while conducting a site reconnaissance.

6.0 RECORDS

The data collected during a site reconnaissance may have to be compiled into a trip report when returning from the field. This trip report can then be distributed to the project team. A site reconnaissance checklist is located in Attachment A which can be copied and used while conducting the site reconnaissance.

ATTACHMENT A

SITE RECONNAISSANCE CHECKLIST

SITE SKETCH

Include the following as appropriate:

- Site Name
- Site location
- Site Boundaries
- Entrance locations
- Access Roads and Security Requirements
- Disposal locations
- Storage areas
- Office areas
- Well locations
- Treatment facility locations
- Surface drainage, outcrops, general topography descriptions
- Cultural interferences

CHEMICAL STORAGE FACILITIES DESCRIPTION

- Storage tanks - numbers, volumes, condition, contents, etc.
- Drums - number, conditions, labeling, etc.
- Lagoons and surface pits - number, size, use of liner, contents, etc.

TREATMENT SYSTEMS

Note the presence of any treatment systems. These can be difficult to evaluate visually. One should appraise general appearance, maintenance and visual integrity; ask operators for any monitoring records; note presence of odors; and visually characterize any effluents or residues. Describe type of wastes and volumes treated.

- Incinerators
- Flocculation/filtration
- Chemical/physical treatment
- Biological treatment
- Volume reduction
- Waste recycling
- Compositing
- Other

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE TWO

DISPOSAL FACILITIES

Note the presence and use of any of the following operations. Include a description of the size, use of liners, soil type, and the presence of leachate. Provide a description of management practices. Interview site workers if possible. Describe waste types.

- Landfills
- Land forms
- Open dump
- Surface impoundment
- Underground injection
- Incineration

Also, records for disposal of concentrated/containerized waste should be reviewed.

HAZARDOUS SUBSTANCE CHARACTERISTICS

Ask facility contacts for manifests, inventories, or monitoring reports. Note markings on containers.

- Chemical identities
- Quantities
- Hazard characteristics (toxic, explosive, flammable, etc.)
- Container markings
- Monitoring data, other analytical data
- Physical state (liquid, solid, gas, sludge)

CHEMICAL PROCESS INFORMATION

- Manufacturing processes and chemicals
- Off-specification or by-product disposal processes
- Housekeeping practices
- Locations of Plant Operations

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE THREE

HYDROGEOLOGIC ASSESSMENT

Look for situations that promote hazardous substance migration, i.e., porous soils, fractured bedrock formations, shallow water table and karst features.

- Soil type
- Surface water features
- Surface drainage pattern
- Outcrop studies
- Water wells (use, water depth, and construction details)
- Erosion potential
- Flooding potential
- Climatology

IDENTIFICATION OF SENSITIVE RECEPTORS

- Number and locations of private homes
- Public buildings including tenant usage
- Areas of dead or dying vegetation or animals
- Presence of sensitive ecosystems (wetlands, tidal marshes, etc.)
- Other public use areas (roads, parks, etc.)
- Natural areas

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1.0 PURPOSE

This procedure discusses the methods used to collect surface, near surface, and subsurface soil samples. Additionally, it describes the method for sampling of test pits and trenches to determine subsurface soil and rock conditions, and recover small-volume or bulk samples.

2.0 SCOPE

This procedure is applicable to the collection of surface, near surface and subsurface soils for laboratory testing, which are exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites.

3.0 GLOSSARY

Composite Sample - A composite sample exists as a combination of more than one sample at various locations and/or depths and times, which is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples are not to be collected for volatile organics analysis.

Grab Sample - One sample collected at one location and at one specific time.

Non-Volatile Sample - A non-volatile sample includes all other chemical parameters (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Hand Auger - A sampling device used to extract soil from the ground in a relatively undisturbed form.

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2-inch OD to 3-1/2 inch OD. The larger sizes are commonly used when a larger volume of sample material is required.

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine the shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher excavator, or bulldozer).

Confined Space - As stipulated in 29 CFR 1910.146, a confined space means a space that: 1) is large enough and so configured that an employee can bodily enter and perform assigned work; 2) has limited or restricted means for entry or exit (for example tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and 3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for determining sampling objectives, as well as, the field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches, and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring and excavation activities, and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - The FOL is responsible for finalizing the location of surface, near surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits and trenches, and for adherence to OSHA regulations during these operations.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of soil samples and the completion of all required paperwork (i.e., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms).

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions which are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

5.0 PROCEDURES

5.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they have migrated into the water table, and can establish the amount of contamination sorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during the sampling operations, particularly noting the location, depth, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. As a result, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. Often this information on soil properties can be obtained from published soil surveys available through the U.S. Geological Surveys and other government or farm agencies. It is the

intent of this procedure to present the most commonly employed soil sampling methods used at hazardous waste sites.

5.2 Soil Sample Collection

5.2.1 Procedure for Collecting Soil Samples for Volatile Organic Compounds

The above described traditional sampling techniques, used for the collection of soil samples for volatile organic analysis, have recently been evaluated by the scientific community and determined to be ineffective in producing accurate results (biased low) due to the loss of volatile organics in the sampling stages and microbial degradation of aromatic volatiles. One of the newly adopted sampling procedures for collecting soil samples includes the field preservation of samples with methanol or sodium bisulfate to minimize volatilization and biodegradation. These preservation methods may be performed either in the field or laboratory, depending on the sampling methodology employed.

Soil samples to be preserved by the laboratory are currently being performed using method SW-846, 5035. Laboratories are currently performing low level analyses (sodium bisulfate preservation) and high level analyses (methanol preservation) depending on the end users needs.

It should be noted that a major disadvantage of the methanol preservation method is that the laboratory reporting limits will be higher than conventional testing. The reporting levels using the new method for most analytes are 0.5 µg/g for GC/MS and 0.05 µg/g for GC methods.

The alternative preservation method for collecting soil samples is with sodium bisulfate. This method is more complex to perform in the field and therefore is not preferred for field crews. It should also be noted that currently, not all laboratories have the capabilities to perform this analysis. The advantage to this method is that the reporting limits (0.001 µg/g for GC/PID or GC/ELCD, or 0.010 for GC/MS) are lower than those described above.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

5.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organics that are to be preserved at the laboratory will be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample will be obtained using a reusable sampling handle provided with the EnCore™ sampler. The sample is collected by pushing the EnCore™ sampler directly into the soil, ensuring that the sampler is packed tight with soil, leaving zero headspace. Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler shipment by the manufacturer.

Once the sample is collected, it should be placed on ice immediately and shipped to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

If the lower detection limits are necessary, an option would be to collect several EnCore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.

5.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) method and medium-level (methanol preservation) method.

Methanol Preservation (Medium Level):

Soil samples to be preserved in the field with methanol will utilize 40-60 mL glass vials with septum lids. Each sample bottle will be filled with 25 mL of demonstrated analyte-free purge and trap grade methanol. Bottles may be prespiked with methanol in the laboratory or prepared in the field.

Soil will be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol preserved sample bottle. Calibration of the scale should be performed prior to use and intermittently throughout the day according to the manufacturers requirements.

The sample should be collected by pulling the plunger back and inserting the syringe into the soil to be sampled. The top several inches of soil should be removed before collecting the sample. Approximately 10 grams $\pm$2g (8-12 grams) of soil should be collected. The sample should be weighed and adjusted until obtaining the required amount of sample. The sample weight should be recorded to the nearest 0.01 gram in the field logbook and/or sample log sheet. The soil should then be extruded into the methanol preserved sample bottle taking care not to contact the sample container with the syringe. The threads of the bottle and cap must be free of soil particles.

After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

Samples to be preserved using the sodium bisulfate method are to be prepared as follows:

Add 1 gram of sodium bisulfate to 5 mL of laboratory grade deionized water in a 40-60 mL glass vial with septum lid. Bottles may be prespiked in the laboratory or prepared in the field. The soil sample should be collected in a manner as described above and added to the sample container. The sample should be weighed to the nearest 0.01 gram as described above and recorded in the field logbook or sample log sheet.

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soils containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode.

When preparing samples using the sodium bisulfate preservation method, duplicate samples must be collected using the methanol preservation method on a one for one sample basis. The reason for this is because it is necessary for the laboratory to perform both the low level and medium level analyses. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

If the lower detection limits are necessary, an option to field preserving with sodium bisulfate would be to collect 3 EnCore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.

5.2.2 Procedure for Collecting Non-Volatile Soil Samples

Non-volatile soil samples may be collected as either grab or composite samples. The non-volatile soil sample is thoroughly mixed in a stainless steel or disposable, inert plastic tray, using a stainless steel trowel or other approved tool, then transferred into the appropriate sample container(s). Head space is permitted in a non-volatile soil sample container to allow for sample expansion.

5.2.3 Procedure for Collecting Undisturbed Soil Samples (ASTM D1587-83)

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) will be employed. The following method will be used:

1. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and clean out the borehole to the sampling depth, being careful to minimize the chance for disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

2. The use of bottom discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Use of any side-discharge bits is permitted.

3. A stationary piston-type sampler may be required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal and to maintain a suction within the tube to help retain the sample.

4. To minimize chemical reaction between the sample and the sampling tube, brass tubes may be required, especially if the tube is stored for an extended time prior to testing. While steel tubes coated with shellac are less expensive than brass, they're more reactive, and shall only be used when the sample will be tested within a few days after sampling or if chemical reaction is not anticipated. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil by a continuous and rapid motion, without impacting or twisting. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

5. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated. Remove disturbed material in the upper end of the tube and measure the length of sample again. After removing at least an inch of soil from the lower end and after inserting an impervious disk, seal both ends of the tube with at least a 1/2-inch thickness of wax applied in a way that will prevent the wax from entering the sample. Clean filler must be placed in voids at either end of the tube prior to sealing with wax. Place plastic caps on the ends of the sample tube, tape the caps in place, and dip the ends in wax.

6. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label. Mark the "up" direction on the side of the tube with indelible ink, and mark the end of the sample. Complete Chain-of-Custody (see SOP SA-6.3) and other required forms (including Attachment A of this SOP). Do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

Thin-walled undisturbed tube samplers are restricted in their usage by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soils with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soils. Using these devices normally increases sampling costs, and therefore their use shall be weighed against the need for acquiring an undisturbed sample.

5.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples (most commonly collected to a depth of 6 inches) for subsequent analysis is by use of a stainless steel trowel. Surface soils are considered 0-12 inches bgs.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Real-time air monitoring instrument (e.g., PID, FID, etc.).
- Latex gloves.
- Required Personal Protective Equipment (PPE).
- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP).
- Required decontamination equipment.
- Required sample container(s).
- Wooden stakes or pin flags.
- Sealable polyethylene bags (i.e., Ziploc® baggies).
- Heavy duty cooler.
- Ice.
- Chain-of-custody records and custody seals.

When acquiring surface soil samples, the following procedure shall be used:

1. Carefully remove vegetation, roots, twigs, litter, etc., to expose an adequate soil surface area to accommodate sample volume requirements.

2. Using a decontaminated stainless steel trowel, follow the procedure cited in Section 5.2.1 for collecting a volatile soil sample. Surface soil samples for volatile organic analysis should be collected from 6-12 inches bgs only.

3. Thoroughly mix (in-situ) a sufficient amount of soil to fill the remaining sample containers and transfer the sample into those containers utilizing the same stainless steel trowel employed above. Cap and securely tighten all sample containers.

4. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.

5. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

5.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6-18 inches) can be accomplished with tools such as shovels and stainless steel or pre-cleaned disposable trowels.

The following equipment is necessary to collect near surface soil samples:

- Clean shovel.
- The equipment listed under Section 5.3 of this procedure.
- Hand auger.

To obtain near-surface soil samples, the following protocol shall be observed:

1. With a clean shovel, make a series of vertical cuts to the depth required in the soil to form a square approximately 1 foot by 1 foot.

2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 2 through 5 listed under Section 5.3 of this procedure.

5.5 Subsurface Soil Sampling With a Hand Auger

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), and a cross handle. A larger diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then withdrawn. In turn, the larger diameter bit is replaced with a smaller diameter bit, lowered down the hole, and slowly turned into the soil at the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

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.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes).
- Stainless steel mixing bowls.
- The equipment listed under Section 5.3 of this procedure.

To obtain soil samples using a hand auger, the following procedure shall be followed:

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

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

3. Begin augering (periodically removing accumulated soils from the bucket bit) and add additional rod extensions as necessary. Also, note (in a field notebook, boring log, and/or on standardized data sheets) any changes in the color, texture or odor of the soil.

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

5. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is commonly smaller in diameter than the bucket bit employed to initiate the borehole.

6. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

7. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

8. Discard the top of the core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the sample material.

9. Fill volatile sample container(s), using a properly decontaminated stainless steel trowel, with sample material directly from the bucket bit. Refer to Section 5.2.1 of this procedure.

10. Utilizing the above trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl and thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

11. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.6 Subsurface Soil Sampling With a Split-Barrel Sampler (ASTM D1586-84)

Split-barrel (split-spoon) samplers consist of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-lb. or larger casing driver.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (O.D. 2 inches, I.D. 1-3/8 inches, either 20 inches or 26 inches long); Larger O.D. samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-lb. weight, driving head and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed under Section 5.3 of this procedure.

The following steps shall be followed to obtain split-barrel samples:

1. Remove the drive head and nosepiece, and open the sampler to reveal the soil sample. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.). Carefully separate the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

2. Collect the volatile sample from the center of the core where elevated readings occurred. If no elevated readings where encountered the sample material should still be collected from the core's

center (this area represents the least disturbed area with minimal atmospheric contact). Refer to Section 5.2.1 of this procedure.

3. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl. Thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

4. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.7 Subsurface Sol Sampling Using Direct Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using direct push technology (DPT). DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

5.8 Excavation and Sampling of Test Pits and Trenches

5.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise which control the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is still required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden or steel support structures. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments. Any entry may constitute a Confined Space and must be done in conformance with all applicable regulations. In these cases, substantial air monitoring is required before entry, and appropriate respiratory gear and protective clothing is mandatory. There must be at least two persons present at the immediate site before entry by one of the investigators. The reader shall refer to OSHA regulations 29 CFR 1926, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146.

Excavations are generally not practical where a depth of more than about 15 feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If data on soils at depths greater than 15 feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

5.8.2 Test Pit and Trench Excavation

These procedures describe the methods for excavating and logging test pits and trenches excavated to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or by power equipment to permit detailed description of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration.
- The space required for efficient excavation.
- The chemicals of concern.
- The economics and efficiency of available equipment.

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table, which is based on equipment efficiencies, gives a rough guide for design consideration:

Equipment	Typical Widths, in Feet
Trenching machine	2
Backhoe	2-6
Track dozer	10
Track loader	10
Excavator	10
Scraper	20

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous waste materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, field conditions may necessitate revisions to the initial plans. The final depth and construction method shall be determined by the field geologist. The actual layout of each test pit, temporary staging area, and spoils pile will be predicated based on site conditions and wind direction at the time the test pit is made. Prior to excavation, the area can be surveyed by magnetometer or metal detector to identify the presence of underground utilities or drums.

As mentioned previously, no personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is still required, Occupational Safety and Health Administration (OSHA) requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be in the hole at all times, and a temporary guardrail must be placed along the surface of the hole before entry). It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example, samples of leachate, groundwater, or sidewall soils can be taken with telescoping poles, etc.

Dewatering may be required to assure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation dry. This is an important consideration for excavations in cohesionless material below the groundwater table. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

5.8.3 Sampling in Test Pits and Trenches

5.8.3.1 General

Test pits and trenches are usually logged as they are excavated. Records of each test pit/trench will be made as presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable Health and Safety and OSHA requirements have been met.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information would include soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples, which can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

5.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottleware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps and right angle adapter for conduit (see Attachment D).

5.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 5.8.3.4.

- Excavate trench or pit in several depth increments. After each increment, the operator will wait while the sampler inspects the test pit from grade level to decide if conditions are appropriate for sampling. (Monitoring of volatiles by the SSO will also be used to evaluate the need for sampling.) Practical depth increments range from 2 to 4 feet.

- The backhoe operator, who will have the best view of the test pit, will immediately cease digging if:

  - Any fluid phase or groundwater seepage is encountered in the test pit.
  - Any drums, other potential waste containers, obstructions or utility lines are encountered.
  - Distinct changes of material are encountered.

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending upon the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Remove loose material to the greatest extent possible with backhoe.

- Secure walls of pit if necessary. (There is seldom any need to enter a pit or trench which would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material once it has been deposited on the ground. The sampler or Field Operations Leader directs the backhoe operator to remove material from the selected depth or location within the test pit/trench. The bucket is brought to the surface and moved away from the pit. The sampler and/or SSO then approaches the bucket and monitors its contents with a photoionization or flame ionization detector. The sample is collected from the center of the bucket or pile and placed in sample containers using a decontaminated stainless steel trowel or disposable spatula.

- If a composite sample is desired, several depths or locations within the pit/trench are selected and a bucket is filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the side wall or bottom of the pit. The face of the pit/trench shall first be scraped (using a long-handled shovel or hoe) to remove the smeared zone that has contacted the backhoe bucket. The sample shall then be collected directly into the sample jar, by scraping with the jar edge, eliminating the need to utilize samplers and minimizing the likelihood of cross-contamination. The sample jar is then capped, removed from the assembly, and packaged for shipment.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

5.8.3.4 <u>In-Pit Sampling</u>

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soils or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There is no practical alternative means of obtaining such data.

- The Site Safety Officer and Competent Person determines that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of volatile organics, explosive gases and available oxygen).

- A Company-designated Competent Person determines that the pit/trench is stable or is made stable (by grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements must be strictly observed.

If these conditions are satisfied, one person will enter the pit/trench. On potentially hazardous waste sites, this individual will be dressed in safety gear as required by the conditions in the pit. He/she will be affixed to a safety rope and continuously monitored while in the pit.

A second individual will be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations. The individual entering the pit will remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon. As an added precaution, it is advisable to keep the backhoe bucket in the test pit when personnel are working below grade. Such personnel can either stand in or near the bucket while performing sample operations. In the event of a cave-in they can either be lifted clear in the bucket, or at least climb up on the backhoe arm to reach safety.

5.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 5.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., open tube samplers), which can be pushed or driven into the floor of the test pit.

- Suitable driving (i.e., a sledge hammer) or pushing (i.e., the backhoe bucket) equipment which is used to advance the sampler into the soil.

- Knives, spatulas, and other suitable devices for trimming hand-carved samples.

- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.

- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soils in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification, while larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soils using open tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe,

rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the sampler when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 5.8.3.4 of this procedure must be followed. The open tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate, because the sample will not have the correct orientation.

A sledge hammer or the backhoe may be used to drive or push the sampler or tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry of the test pit, the requirements in Section 5.8.3.4 of this procedure must be followed. Prepare, label, pack and transport the sample in the required manner, as described in SOP SA-6.3 and SA-6.1.

5.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew shall photograph all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL.

If a low permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

5.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. In addition, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job.
- Date of boring and excavation.

- Approximate surface elevation.
- Total depth of boring and excavation.
- Dimensions of pit.
- Method of sample acquisition.
- Type and size of samples.
- Soil and rock descriptions.
- Photographs.
- Groundwater levels.
- Organic gas or methane levels.
- Other pertinent information, such as waste material encountered.

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American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

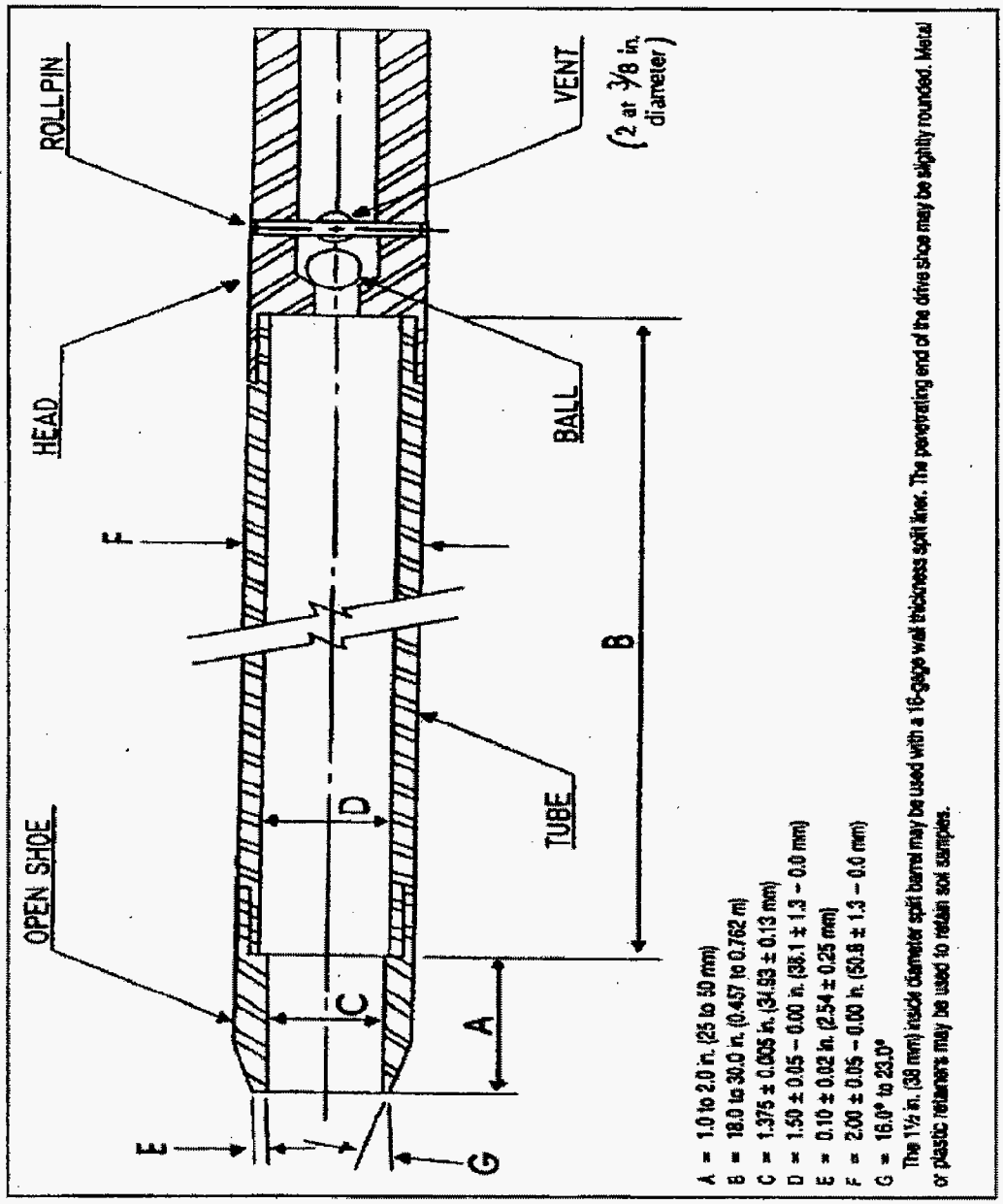
MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 16.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

Tetra Tech NUS, Inc.

TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

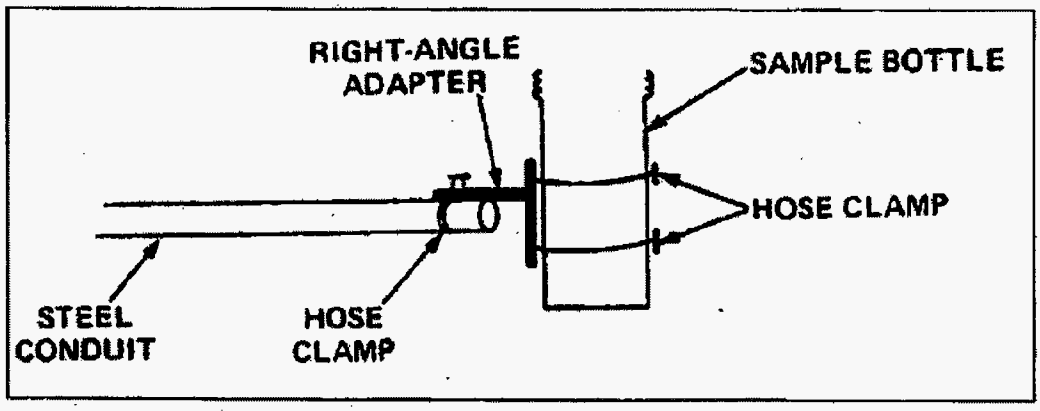
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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1.0 PURPOSE

This procedure describes methods and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing, or for subsequent laboratory analysis.

2.0 SCOPE

The information presented in this guideline is generally applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions and equipment different from those described herein.

3.0 GLOSSARY

Environmental Sample - a sample containing (or suspected to contain) low-level concentrations of contaminants, which does not require special handling or transport considerations as detailed in SOP SA-6.1.

Hazardous Waste Sample - a sample containing (or suspected to contain) higher concentrations of contaminants thus requiring special handling and/or transport considerations per SOP SA-6.1.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel.

Field Operations Leader - The Field Operations Leader (FOL) is responsible for the supervision of onsite water quality analyses, ensuring proper sample collection, handling, and the completion and accuracy of all field documentation, and making sure that custody of all samples obtained is maintained according to proper procedures.

5.0 PROCEDURES

5.1 Introduction

Collecting a representative sample from surface water or sediments is difficult because of water movement, stratification, or patchiness. To collect representative samples, one must standardize sampling bias related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification.

Representativeness is a qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been taken. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples.

5.2 Defining the Sampling Program

Many factors must be considered in developing a sampling program for surface water or sediments including study objectives, accessibility, site topography, physical characteristics of the water body (such as flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on the vertical and lateral mixing within the body of water. For sediments, dispersion depends on bottom current or flow characteristics, sediment characteristics (density, size) and geochemical properties (which affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes, but also must understand the role of fluvial-sediment transport, deposition, and chemical sorption.

5.2.1 Sampling Program Objectives

The objective of surface water sampling is to determine the surface water quality entering, leaving or remaining within the site. The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., spills). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the waterbody, direct waste disposal (solid or liquid) into the water body; and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) which encompass the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the location of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc., shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation are: (1) move the site far enough downstream to allow for adequate mixing, or (2) collect integrated samples in a cross section. Also, nonhomogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and the heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

5.2.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled

by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream, because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes and reservoirs, as well as those on larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Sampling in marshes or tidal areas may require the use of an all-terrain vehicle (ATV). The same precautions mentioned above with regard to sediment disturbance apply.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed in order to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of streamflow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Streamflow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining streamflow data by direct or indirect methods.

5.2.3 Frequency of Sampling

The sampling frequency and the objectives of the sampling event will be defined by the project plan documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of the contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples shall be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly, and during droughts and floods). Samples of bottom material shall be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water-quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

5.3 Surface Water Sample Collection

5.3.1 Streams, Rivers, Outfalls and Drainage Features (Ditches, Culverts)

Methods for sampling streams, rivers, outfalls, and drainage features at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode, shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited.

Generally, the number and type of samples to be taken depend on the river's width, depth, discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of DO, pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project plan documents.

5.3.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained.

The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, the measurement of DO, pH, temperature, etc., is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally-formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample. These verticals are often taken along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline which is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer which is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several verticals with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality since it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in-situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, dissolved oxygen, some cations and anions, and light penetration.

5.3.3 Estuaries

Estuarine areas are by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Estuaries are generally categorized into three types dependent upon freshwater inflow and mixing properties. Knowledge of the estuary type is necessary to determine sampling locations. Each type of estuarine area is described below:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically this type of estuary is shallow and is found in major freshwater sheetflow areas. Being well mixed, the sampling locations are not critical in this type of estuary.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally, back and forth, with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small with the preponderance of the fresh-saline water mixing occurring near, or at, the shore line.

Sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical dissolved oxygen and temperature profiles.

5.3.4 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. The most frequently used samplers are:

- Open tube.
- Dip sampler.
- Weighted bottle.
- Hand pump.
- Kemmerer.
- Depth-Integrating Sampler.

The dip sampler and the weighted bottle sampler are used most often, and detailed discussions for these devices only (and the Kemmerer sampler) are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.
4. Nonreactive/noncontaminating properties - Teflon-coated, glass, stainless-steel or PVC sample chambers are preferred (in that order).

As specified above, each sample (grab or each aliquot collected for compositing) shall be measured for but not limited to:

- Specific conductance.
- Temperature.
- pH.
- Dissolved oxygen (optional).

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field

notebook or on sample logsheets (see Attachment A). These analyses will provide information on water mixing/stratification and potential contamination.

Dip Sampling

Water is often sampled by filling a container either attached to a pole or held directly, from just beneath the surface of the water (a dip or grab sample). Constituents measured in grab samples are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration that is distributed throughout the water column and in the cross section. Therefore, whenever possible, it is recommended to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

Weighted Bottle Sampling

A grab sample can also be taken using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of a stopped glass or plastic bottle, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The procedure for sampling with this device is:

- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
- Pull out the stopper with a sharp jerk of the stopper line.
- Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
- Raise the sampler and cap the bottle.
- Decontaminate the outside of the bottle. This bottle can be used as the sample container as long as the bottle is an approved container type.

Kemmerer

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless-steel or acrylic cylinder, with rubber stoppers that leave the ends open while being lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles.

5.3.5 Surface Water Sampling Techniques

Most samples taken during site investigations are grab samples. Typically, surface water sampling involves immersing the sample container in the body of water; however, the following suggestions are made to help ensure that the samples obtained are representative of site conditions:

- The most representative samples are obtained from mid-channel at a 0.6 foot stream depth in a well-mixed stream.
- Even though the containers used to obtain the samples are previously laboratory cleaned, it is suggested that the sample container be rinsed at least once with the water to be sampled before the sample is taken. This is not applicable when sample containers are provided "pre-preserved."
- For sampling moving water, it is suggested that the farthest downstream sample be obtained first, and that subsequent samples be taken as one works upstream. In general, work from zones suspected of low contamination to zones of high contamination.
- To sample a pond or other standing body of water, the surface area may be divided into grids. A series of samples taken from each grid node is combined into one sample, or several grid nodes are selected at random.
- Care should be taken to avoid excessive agitation of the water, as loss of volatile constituents could result.
- When obtaining samples in 40 mL septum vials for volatile organics analysis, it is important to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. The vial can be turned upside down to check for air bubbles.
- Do not sample at the surface, unless sampling specifically for a known constituent which is immiscible and on top of the water. Instead, the sample container should be inverted, lowered to the approximate depth, and held at about a 45-degree angle with the mouth of the bottle facing upstream. When sample containers are provided "pre-preserved," use a dedicated, clean, un-preserved bottle for sampling and transfer to an appropriately-preserved container.

5.4 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

5.5 Sediment Sampling

5.5.1 General

Sediment samples are usually collected at the same verticals at which water samples were collected. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body.

Generally, the coarser grained sediments are deposited near the headwaters of the reservoir. Bed sediments near the center of a water body will be composed of fine-grained materials which may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathometry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled while areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials, shall be avoided.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if

water column concentrations are below detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B.

5.5.2 Sampling Equipment and Techniques

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

The following health and safety provisions apply when working on/over/near water.

Size of Work Team:

1) Never less than 2 persons [who are wearing USCG approved Personal Flotation Devices (PFDs)]

2) A minimum of 3 persons if *any* of the following conditions are anticipated or observed:

- Depth is greater than 3 feet
- Involves a waterway that is turbulent *or* swift
- The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples would include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.)
- Waterway is tidal, and conditions such as those listed above could change

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered PFD (life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect bottom materials:

- Scoop sampler.
- Dredge samplers.

Each type of sampler is discussed subsequently.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if it can be waded, the easiest and best way to collect a sediment sample is to use a scoop sampler. This reduces the potential for cross-contamination. This method is accomplished by reaching over or wading into the water body and, while facing upstream (into the current), scooping the sampler along the bottom in an upstream direction. It is very difficult not to disturb fine-grained materials of the sediment-water interface when using this method.

Dredges

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., coarse-grained or partially-cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger". Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. There are three major types of dredges: Peterson, Eckman and Ponar dredges.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave". The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

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ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA

	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	NA
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

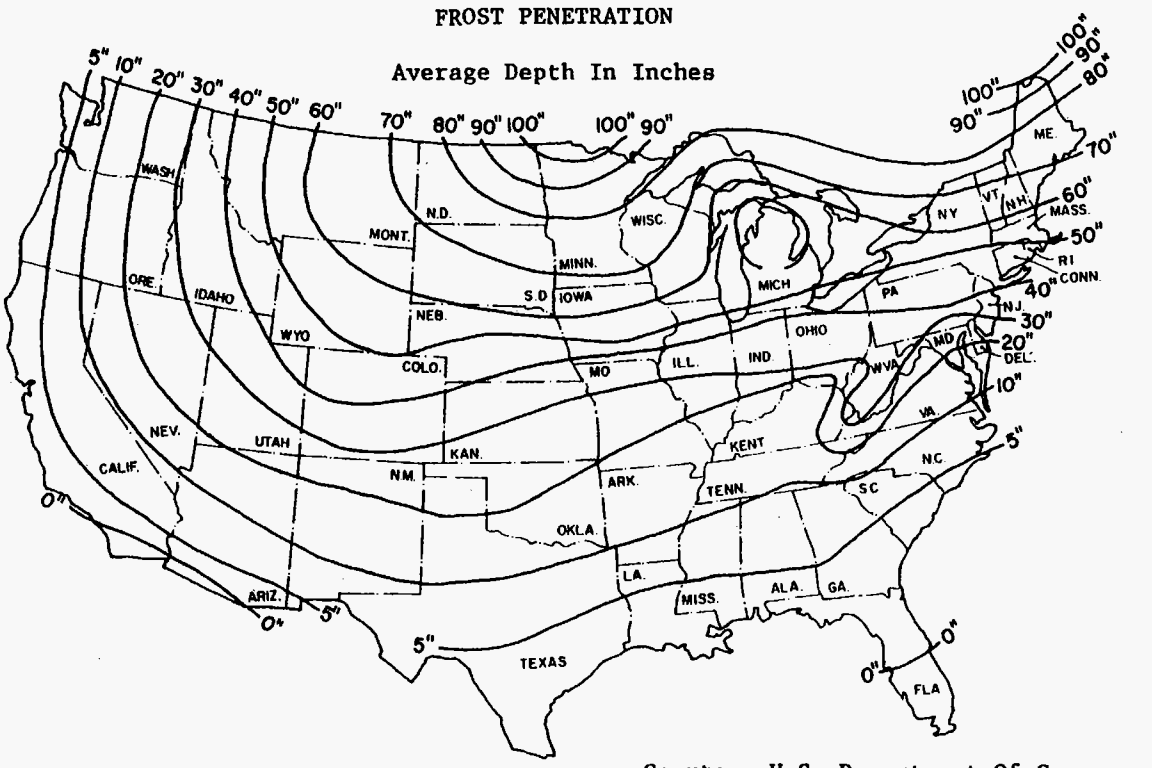
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes	no	N/A
b)	Interview local personnel?	yes	no	N/A
c)	Site visit and inspection?	yes	no	N/A
d)	Excavation areas marked in the field?	yes	no	N/A
e)	Utilities located in the field?	yes	no	N/A
f)	Located utilities marked/added to site maps?	yes	no	N/A
g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes	no	N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes	no	N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes	no	N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes	no	N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes	no	N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes	no	N/A
b)	Marked on site maps	yes	no	N/A
c)	Necessary to lockout/insulate/re-route	yes	no	N/A
d)	Document procedures used to lockout/insulate/re-route	yes	no	N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _____			

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

APPENDIX D

FIELD DOCUMENTATION FORMS

TETRA TECH NUS, INC.

ANALYTICAL SERVICE
Packing List/Chain-of-Custody

Case No. _____ Page _____ of _____

Subcontract No. _____

Project No.	Laboratory Name:	Container Type	Container Type	Container Type	Container Type	Container Type
Sampler Signatures	Date Shipped Carrier Airbill No. No. of Coolers	Analysis	Analysis	Analysis	Analysis	Analysis

Sample Number	Matrix	Date/Time	Sample Location	Tag Number(s)	QC	Preservative	Preservative	Preservative	Preservative	Preservative

Relinquished By: (Signature)	Date/Time	Received By: (Signature)	Shipment for Case Complete? YES NO	Remarks
Relinquished By: (Signature)	Date/Time	Received for Laboratory By:	Date/Time	

TtNUS Form 0022

Tetra Tech NUS, Inc.

BORING LOG

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

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

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): _____

Remarks: _____

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

Tetra Tech NUS, Inc.

PROJECT: _____ **JOB #:** _____
LOCATION: _____ **DATE:** _____
PROJECT MANAGER: _____ **FOL:** _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1 _____			
2 _____			
3 _____			
4 _____			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

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

TETRA TECH NUS, INC.

FIELD INSTRUMENT CALIBRATION LOG

INSTRUMENT NAME: _____ MODEL No.: _____

SERIAL No.: _____ DECAL No.: _____ TETRA TECH NUS CHARGE No. _____

CALIBRATION DATE	INITIAL READING	PROCEDURE	FINAL READING	SIGNATURE	COMMENTS

TtNUS Form 0007

TETRA TECH NUS, INC.

FIELD MODIFICATION RECORD

Site Name: _____ Location: _____

Project Number: _____ Task Assignment: _____

To: _____ Location: _____ Date: _____

Description:_____

_____

_____

_____

Reason for Change: _____

_____

_____

Recommended Action: _____

_____

_____

Field Operations Leader (Signature): _____ Date: _____

Disposition/Action:_____

_____

_____

Project Manager (Signature): _____ Date: _____

Distribution:

		Others as Required:
Program Manager:	_____	
Project Manager:	_____	_____
Quality Assurance Officer:	_____	_____
Field Operations Leader:	_____	_____
Project File:	_____	_____

TtNUS Form 0003

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS, INC.

SAMPLE COLLECTION SUMMARY RECORD

PROJECT NAME: _____ TETRA TECH NUS CHARGE NUMBER: _____

SAMPLING EVENT: _____ CASE NO.: _____ DAS NO.: _____

DATE	TIME	SAMPLE LOCATION	FIELD QC							COMMENTS

TtNUS Form 0012

TETRA TECH NUS, INC.

SITE ENTRY LOG

Site Name: _____ Date: _____

Location: _____ Project Number: _____

NAME	REPRESENTING	TIME IN (HOURS)	TIME OUT (HOURS)	INITIALS

TtNUS Form 0002

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX E

MITKEM LABORATORIES
SELECTED STANDARD OPERATING PROCEDURES AND
QUALITY ASSURANCE PROGRAM MANUAL

SELECTED MITKEM LABORATORIES
STANDARD OPERATING PROCEDURES CONTENTS

SOP NUMBER	SUBJECT
20.0003	Logging Workorders and Samples into Omega ME
30.0003	Sample Receipt, Storage, Tracking and Disposal
30.0024	Sample and Waste Disposal
50.0052	Organic Preparation of Soil Samples by Sonication (Method 3550B)
50.0053	Organic Preparation of Soil Samples by Soxhlet (Method 3540C)
50.0054	Organic Extract Filtration and Concentration Techniques
50.0100	Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3770)
60.0003	Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector (Analysis by SW846 Method 8082)
60.0050	Total Petroleum Hydrocarbons by GC-FID Using EPA SW-846 Method 8015B/State Methods
70.0033	Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270
90.0012	Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C
90.0038	Gasoline Range Organics in Aqueous and Soil Samples by Gas Chromatography-Flame Ionization Detector (GC-FID) using Methods SW-846 8015 and Maine 4.2.17
100.0110	Determination of Metals in Water and Soils by SW-846 Method 6020A Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)
100.0012	Mercury Analysis in Aqueous and Soil Samples by Flow Injection Mercury System (FIMS) for Atomic Absorption by SW846 Method 7470A/7471B
100.0104	Preparation of Soil Samples by Acid Digestion for ICP Analysis by SW846 Method 3050B
100.0111	Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C
110.0026	Handling Evidentiary Materials
110.0034	Sample Data Control for Inorganic CLP (ILM/ISM)
110.0035	Sample Data Control for CLP (SOM01.2)
110.0038	Percent Solids Determination As Required for Various SW846 and EPA Methods

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

Determination of Metals in Water and Soils by SW-846 Method 6020A Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

SOP No.100.0110
Rev. 0

	Signature	Date
QA Director:		3/27/09
Lab Director:		3/27/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

For

Determination of Metals in Water and Soils by SW-846 Method 6020A

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

SOP No.100.0110
Rev. 0

1. Scope and Application

This SOP describes the procedures applicable to the analysis of elements listed on page 1 of SW-846 Method 6020A. ICP technology was built upon the same principles used in atomic emission spectrometry. Samples are decomposed to neutral elements in high temperature argon plasma and analyzed based on their mass to charge ratios. An ICP-MS can be thought of as having four main processes, including sample introduction and aerosol generation, ionization by an argon plasma source, mass discrimination, and the detection system.

All matrices, including surface and ground waters, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments and other solid wastes, wipes and air filters are applicable to this analysis. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Total recoverable metals require acid digestion prior to analysis. This SOP pertains to the analysis of digestates and standards.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. Supervisors/Managers are responsible for ensuring that SOPs are accurate and up to date, and that they are implemented appropriately. Supervisors/Managers review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, total (acid-soluble) metals samples must be digested using appropriate sample preparation methods. Dissolved metals do not require digestion. (see **Section 8.1**)

3.2 An ICP-MS combines a high-temperature ICP (Inductively Coupled Plasma) source with a mass spectrometer (MS). The ICP source converts the atoms of the elements in the sample to ions. These ions are then separated and detected by the mass spectrometer. For a complete description of ICP-MS see the primer at http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm, which gives a concise breakdown of how an ICP/MS works.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the client. For metals analysis, water samples are collected in 1liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Sample holding time for metals analysis is 180 days from VTSR for both water and soil for all metals other than mercury.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICP-MS

5.1 Isobaric elemental interferences are caused by isotopes of different elements, which form singly or doubly charged ions of the same nominal mass-to-charge ratio.

5.2 Abundance sensitivity is affected by ion energy and quadruple operating pressure. Wing overlap interferences may result when a small m/z peak is being measured adjacent to a large one (also called wing overlap).

5.3 Isobaric Polyatomic Ion Interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer in use.

5.4 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 salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element.

Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.5 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.6 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, or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.7 Contamination by samples: Significant laboratory or instrument contamination may result when untreated effluents, in-process waters, landfill leachates, and other samples containing high concentrations of inorganic substances are processed and analyzed.

5.8 Contamination by indirect contact: It is imperative that every piece of the apparatus that is directly or indirectly used in the collection, processing, and analysis of ambient water samples be cleaned.

5.9 Contamination by airborne particulate matter: Samples may be contaminated by airborne dust, dirt, particles, or vapors from unfiltered air supplies; nearby corroded or rusted pipes, wires, or other fixtures; or metal-containing paint.

5.10 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. Mitkem performs the following analyses in each analytical sequence.

a) Serial Dilution Test: One sample per matrix per batch; A 5x dilution must agree to within 10% of the original determination if the analyte concentration is sufficiently high (minimally a factor of 10 times the PQL of the sample). If not, a chemical or physical interference effect is suspected. Samples identified as field blanks or PEs should not be used for serial dilutions.

b) Post Digestion Spike Addition: If the spike (pre-digestion) recovery of an analyte falls outside of the control limits, a portion of the same sample (digestate) is spiked with a post-digestion spike. The PDS should be recovered to within 80-120% of its true value. The spike addition should produce a minimum level of 10 x and a maximum of 100x the PQL If both the Matrix spike and the PDS fail, matrix effects are confirmed.

6. Equipment and Apparatus

6.1 Glassware

6.1.1 Class A volumetric flasks:

- 100 mL and 50 mL

6.1.2 Class A volumetric pipettes:

- 10-100µL adjustable Eppendorf.
- 100-1000µL adjustable Eppendorf.
- 1-5mL adjustable Fisher Scientific.

6.1.3 15mL Evergreen Polystyrene ICP tubes.

6.1.4 100µL and 1mL fixed Wheaton.

6.2 ICP-MS- Thermo Electron Xseries2

The built-in radio-frequency generator is FCC compliant.

The high purity grade (99.9%) argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.2.1 Operating conditions: The analyst should follow the instructions provided by the instrument manufacturer. Method detection limits, precision and interference effects must be investigated and established for each analyte. All measurements must be in the instrument operational range where spectral interference correction factors are valid. 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.

6.2.2 Instrument Maintenance: See manufacturer's operation manual for additional information.

6.2.2.1 Peristaltic pump tubing should be replaced after approximately 20 hours of instrument run time. Relative standard deviations within samples containing measurable analytes that are above 3% indicate excessive tubing wear.

6.2.2.2 Torch and cones should be inspected daily for obvious buildup. Degraded performance will be clear when the daily performance check is run and fails or barely passes. Problems may be rectified by adjusting the instrument settings in a tune, and this should be attempted before cleaning the system. Light elements (Be) will be the first to be impacted by a dirty sample introduction system or cones.

6.2.2.3 The daily performance check will also indicate the need for mass calibration if responses are acceptable and mass verification differs by more than 0.1 from the true value

6.2.3 Troubleshooting: See manufacturer's operation manual.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, Accustandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. ***Please note that standards and reagents from other vendors than listed below could be used as long as the standards are of high purity (>96%) and traceable to reference materials.*** All standards are labeled as instructed in Mitkem SOP# 80.0001 Standard Preparation, Equivalency and Traceability. Reagents are labeled as instructed in Mitkem SOP# 80.0013 Reagent Purchasing and Tracking.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.

7.2 Nitric acid (conc.), HNO_3, Trace Grade, Fisher Scientific.

7.3 Reagent water. Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. It is also referred to as DI Water.

7.4 Primary Standards: Mixed calibration standards and individual analyte standards; obtained from High Purity Standards, come in several stock solutions.

7.4.1 Intermediate (Secondary) Standards: Dilutions of the above primary standards in acidified reagent water as appropriate.

7.4.2 Working Calibration standards and QC solutions are made up on a daily basis. All standards and solutions are documented in the lab's standard logbooks. Each laboratory area has Primary, Intermediate and the Working Standard Logbooks. Concentrations of working standards and the associated QC limits can be found in LIMS within the individual testcode SPECs, as identified by Standard or QC Sample name (i.e.: ICAL1) or refer to Table 1.

7.5 Tuning Solution and Mass Calibration: Prepare a dilution of mixed calibration standards with Beryllium, Magnesium, Cobalt, Indium and Lead at 10 ug/L. The solution should be in 1%HNO_3.

7.6 Initial Calibration Verification Standard (ICV): An independent source standard prepared at a concentration near the midpoint of the calibration curve. This standard may also be purchased. See Table 1.

7.7 Low Level Initial Calibration Verification Standard (LLICV): A same source standard at the lowest concentration used in the calibration.

7.8 Interference Check Standard (ICS): QC Standards ICSA and ICSAB stock solutions are obtained from Accustandard. Prepare ICSA and ICSAB fresh daily (ICSAB may require preparation more frequently due to silver precipitation). See Table 1.

7.9 Matrix Spike and Laboratory Control Sample: See preparatory methods for vendor information. LIMS Omega will have final concentrations expected in the digestates in the LIMS testcode SPECs. See Table 1.

8. Procedure

8.1 The methods used for sample digestion or preparation are as follows:

8.1.1 For water samples to be analyzed for total acid soluble/recoverable metals determination: a 50mL aliquot of the unfiltered (well-shaken) sample is digested with nitric and hydrochloric acids using Mitkem **SOP #100.0003,** Methods SW-846 3005/3010.

8.1.2 For water samples to be analyzed for dissolved metals determination: a 50mL aliquot of the filtered (0.45µm filter) sample is nitric acid preserved using Mitkem **SOP #100.0003,** Methods SW-846 3005/3010. No digestion is required.

8.1.3 For soil samples to be analyzed for total metals determination: a representative sample (between 1.0 and 2.0 gram) is digested with nitric acid using Mitkem **SOP #100.0104,** Method SW-846 3050.

8.2 Precalibration routine

8.2.1 Prior to calibration, the instrument must be allowed to become stable. This may take up to a half-hour. Conduct any necessary mass calibration and resolution routines to bring peak width within the manufacturer's specifications and adjust mass calibration to within 0.1u over the range of 6 to 210u. The resolution must be verified to be less than 0.9amu full width at 10% peak height.

8.2.2 Tuning Solution: Demonstrate instrument stability and precision by analyzing the tuning solution as a single analysis with at least four integrations. The %RSD of the absolute signals for all of the multiple integrations in the tuning solution (as calculated by the instrument) must be ≤ 5.0% for each analyte.

8.2.3 Internal Standardization: For full range mass scans, a minimum of 5 internal standards (IS) must be used. The IS chosen must be consistent throughout the entire run sequence. IS shall be present in all samples, standards and blanks at the same levels. No IS are used in the tuning solution. If dilutions are performed on the digested samples, then the IS must be added after the dilution. IS are added by the instrument. The IS used at Mitkem are listed in Table 3.

8.3 Calibration: Instrument calibration is required each time the instrument is set up for a sequence or after a continuing calibration failure. Extensive instrument maintenance or significant analytical changes would require re-calibration as well.

8.3.1 Calibrate the instrument with one blank standard and at least 3 non-zero standards. See Table 2. One standard must be at, or below, the PQL. With a multi-point curve, the upper quantitation limit (UQL) may exceed the highest concentration calibration point and can be defined as the "linear dynamic" range. Alternately, the calibration curve could be prepared daily with a minimum of a calibration blank and a single standard at the appropriate concentration to effectively outline the desired quantitation range. The multi-point curve is the preferred method of calibration at Mitkem.

8.3.2 A minimum of 3 replicate integrations are required for data acquisition. Use the average of the integrations for instrument calibration and data reporting.

8.3.3 The calibration curve shall be calculated using linear regression by plotting the concentration of the standard in ug/L on the X-axis versus the corrected instrument response on the Y-axis. The correlation coefficient, r, for the calibration curve must be ≥ 0.998.

DoD requires a correlation coefficient r ≥0.995

8.3.3.1 If the recommended linear response cannot be attained using a minimum of three non-zero calibration standards, consideration should be given to adding more standards, particularly at the lower concentrations, in order to better define the linear range and the lower limit of quantitation. Conversely, the extreme upper and lower calibration points may be removed from the multi-point curve as long as three non-zero points remain such that the linear range is narrowed and the non-linear upper and/or lower portions are removed.

8.4 Calibration Verification

8.4.1 Initial Calibration Verification (ICV): After successful initial calibration, the accuracy of the curve is verified for every analyte by the analysis of the second or independent source ICV for each mass used to report final data results. The concentration of the ICV is at or near the mid-point of the calibration range. Recovery should be within 90-110% recovery to verify the curve.

8.4.2 Low Level Initial Calibration Verification (LLICV): After successful ICV analysis, it is recommended that the accuracy of the low-end of the calibration range be verified by the analysis of a LLICV for each mass used to report final data results. The standard is prepared from the same source as that used for the instrument calibration. The recovery should be within ±30%. Alternatively, the low standard (S1) of a multi-level curve could be plugged back into the curve for verification. The LLICV may be analyzed again at the end of the sequence to verify continued sensitivity (then called LLCCV).

DoD requires the LLICV meet 80-120 % recovery criteria when performing a one point calibration.

8.4.3 Continuing Calibration Verification (CCV): To ensure calibration accuracy during each analytical sequence, a CCV shall be analyzed and reported for each mass used for reporting final results for each element, at a frequency not to exceed every 10 samples during an analytical sequence. The CCV must be analyzed at the beginning of the run and after the last analytical sample. The recovery should be within 90-110% recovery to accept ongoing calibration accuracy. The analytical sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCVs meet the previously mentioned criteria.

8.4.3.1 CCV standards are prepared from the same source as the calibration and are at or near the mid-level concentration used during initial calibration.

8.4.3.2 The same CCV is used throughout the entire analytical sequence.

8.4.3.3 The CCV is prepared in the same acid matrix as the calibration standards.

8.5 Initial and Continuing Calibration Blanks (ICB/CCB): The ICB and CCB are identical in composition to the calibration blank (S0) used in the initial calibration. The ICB/CCB are analyzed immediately after the ICV/CCV to monitor for potential carryover of analytes. If the absolute value of the ICB/CCB result exceeds the PQL, the analysis is stopped and the problem corrected. The instrument must then be recalibrated.

8.6 Sample Analysis

8.6.1 Interference Check Sample (ICS): To verify corrections for elemental and polyatomic isobaric interferences and to monitor for all interferents, an ICS is analyzed and reported for all elements on the Target Analyte List (TAL) immediately after the initial calibration but not before the ICV/ICB pair. The analysis of the ICS is immediately followed by the analysis of the CCV/CCB pair. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents and Solution AB consists of the analytes mixed with the interferents. The ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

8.6.2 Matrix Spike Sample (MS): Spiked samples are analyzed to provide information about the effect of sample matrix on the digestion and/or measurement methodology. The spike is added before the digestion reagents. At least one spike sample is performed on each batch of samples of a similar sample matrix type (water, soil...). Samples identified as field blanks or PEs should not be used for matrix spike samples. In some cases the sample requested for use as a matrix spike will be identified. The MS is spiked with the same solution as is used for the LCS, at the same concentration. Percent recovery is calculated for each analyte added.

8.6.3 Duplicate Sample (DUP): Duplicate samples are analyzed to provide information about the precision of the methodology for each element. Samples identified as field blanks or PE should not be used as duplicates. In some cases the sample requested for use as a duplicate will be identified. Relative Percent Difference is calculated for each analyte detected in the duplicate samples.

8.6.4 Laboratory Control Sample (LCS): The LCS is prepared by spiking an aliquot of reagent water or digesting an aliquot of an SRM. One LCS is prepared per preparation batch of water or soil samples in a batch. Percent Recovery is calculated for each analyte in the LCS. The LCS must include all elements of interest.

8.6.5 After completion of the initial requirements of this method, field samples should be analyzed in the same operational manner used in the calibration routine. A rinse blank is run between all sample solutions, quality control samples, method blanks, and check solutions. Samples that exceed the linear dynamic range must be diluted and reanalyzed. Dilution factors should be appropriate to bring the readings within the upper 75% limit of the linear range.

Analytical Sequence:

1. Performance Check
2. Standard S0 (Blank std)

3. Standard S1 (Low std)
4. Standard S2
5. Standard S3
6. Standard S4
7. Standard S5 (Mid-range std =CCV)
8. Standard S6 (High std)
9. ICV
10. ICB
11. LLICV
12. ICSA
13. ICSAB
14. CCV
15. CCB
16. Samples
17. CCV (not to exceed 10 samples since last CCV)
18. CCB
19. Samples
20. LLCCV(if end of sequence)
21. CCV(not to exceed 10 samples since last CCV)
22. CCB

Note: If the last CCV, CCB meet the QC criteria the analytical run can be continued with using the same sequence protocol outlined above.

All analyses are documented in the ICP/MS Instrument Run Log.

9. Data Reduction and Calculations

9.1 After completion of the analyses, the data files are exported to the server (\CLP Files directory).

9.2 Soil samples are reported on a dry weight basis.

9.2.1 Percent Solids are calculated using the following formula:

$$\% \text{ solids} = \frac{DW \times 100}{WW}$$

DW = Sample weight (g) dried overnight
WW = Sample weight (g) before drying

10. Quality Assurance/Quality Control

10.1 Instrument detection limits (IDLs) are a useful tool to evaluate the instrument noise

level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the PQL, nor should they be used in establishing this limit. IDLs are established initially when the instrument is set up and then on a quarterly basis. IDL values are uploaded to the LIMS system for each instrument.

IDLs in µg/L can be estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). It may be helpful to compare the calculated IDLs to the testcode PQLs. It should be understood that the IDL must be less than the PQL. The PQL needs to be verified initially by analysis of a lower limit of quantitation check (LLQC) sample and on an as needed basis to verify the lab's ability to detect elements at the reporting level. See **Section 10.4**.

10.2 The linear dynamic range (LDR) is established when the system is first setup, or whenever significant instrument components have been replaced or repaired, and on an as needed basis only after the system has been successfully calibrated using either the single or multi-point standard calibration approach. The upper limit of the linear dynamic range needs to be established for each wavelength utilized by determining the signal responses from a minimum of three, preferably five, different concentration standards across the range. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The data, calculations and rationale for the choice of range made should be documented and kept on file.

A standard at the upper limit should be prepared, analyzed and quantitated against the normal calibration curve. The calculated value should be within ±10% of the true value. New upper range limits should be determined whenever there is a significant change in instrument response. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply a spectral correction, the correction might not be valid and those analytes where the spectral correction has been applied may be inaccurately reported.

DoD requires LDR or upper limit check standard every six months, at a minimum.

10.3 Method detection limits (MDLs) are established annually however there is *no requirement* in the current SW-846 6020A method.

DoD requires an MDL study when the instrument is initially set up and annually thereafter.

10.4 The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits (PQL) and on an as needed basis to demonstrate the desired detection capability. This check sample and the low-level calibration verification standard will be prepared at the same concentrations with the only difference being the LLQC sample is

carried through the entire preparation and analytical procedure. PQLs are verified when all analytes in the LLQC sample are detected within ± 30% of their true value.

10.5 Initial Precision and Accuracy (IPANDA studies): Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. The precision and accuracy studies are done as a combination of a preparative method and analysis. If an autosampler is used to perform sample dilutions, before using the autosampler to dilute samples, the laboratory should satisfy itself that those dilutions are of equivalent or better accuracy than is achieved by an experienced analyst performing manual dilutions. The laboratory must also repeat the demonstration of proficiency whenever new staff members are trained or significant changes in instrumentation are made.

10.6 Tune: If the mass calibration is not within the 0.1u over the range of 6 to 210 u, or the %RSD of all the integrations of the absolute signals of the analytes exceeds 5.0%, the analysis is terminated and the problem corrected. The instrument would then be re-tuned before any further calibration.

10.7 Initial Calibration Verification (ICV): The measurements should be within 90-110% recovery to verify the curve. If the ICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated before reanalyzing the ICV.

10.8 Low Level Initial Calibration Verification (LLICV): The measurements should be within 70-130% recovery to verify detection at the low end of the curve. If the LLICV does not meet criteria the analysis shall be stopped, the problem corrected, and the instrument recalibrated.

10.9 Continuing Calibration Verification (CCV): If the deviation of the CCV is greater than the control limits of 90 - 110% recovery, the analysis shall be stopped, the problem corrected, the instrument recalibrated, the calibration verified, and re-analysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the elements affected.

10.10 Blanks: There are three types of blanks required by this method. The calibration blank (S0) is used in establishing the initial calibration. This same blank is used for the initial and continuing blanks (ICB/CCB) to monitor for carryover. The Method blank, or Preparation blank, is used to monitor contamination from preparation and analysis. A Rinse blank is used to flush the system between standards and samples.

10.10.1 ICB/CCB: If the absolute value of an analyte result exceeds the PQL the analysis is terminated and the problem corrected. The instrument would then require recalibration and reanalyis of all samples that were analyzed since the last compliant calibration blank. Calibration Blanks consist of the same concentrations of the same acids used to prepare the final dilution of the calibrating solutions. Usually this is 1% (v:v) Nitric acid in reagent water, however the use of HCl for high antimony or silver

may be needed. Therefore we generally use 1% (v:v) Nitric acid and 0.5% (v:v) HCl in reagent water. See section 7.2 of SW-846 6020A.

10.10.2 Method/Preparation Blanks (MB): The MB contains all the reagents in the same volumes as the samples it is prepared with. The MB is carried through the full procedure and has the same acid concentration in the final digestate as the associated samples. Analyze the MB in the same manner as a sample. Each preparation batch of 20 or less field samples has a minimum of one MB. For positive MB concentrations (the MB may be rerun once to verify results):

10.10.2.1 If the absolute value of the concentration is less than or equal to the PQL in the MB, no corrective action is needed.

10.10.2.2 If the analyte concentration is above the PQL in the MB, the lowest concentration of that analyte in the associated batch samples (not field blanks) is reportable if it is greater than or equal to 10 times the blank concentration. Otherwise all associated batch samples, with the analyte concentration less than 10 times the blank concentration and above the PQL, will be redigested and reanalyzed with new QC for that analyte. No blank correction is performed.

DoD requires no analyte in the MB detected > ½PQL or greater than 1/10 the amount measured in any associated sample. Common lab contaminants should be <PQL.

10.10.2.3 If the analyte concentration in the sample(s) is below the PQL, the samples may be reported despite the method blank contamination. This should be noted in the narrative.

10.10.3 Rinse Blanks consist of 2% HNO_3 and 0.5% HCl in reagent water.

10.11 Interference Check Sample (ICS):

10.11.1 Analytical results of Solution A (ICSA) shall fall within the control limits of ± 2 times the PQL or ±20% of the analyte's true value (the true value shall be zero unless otherwise stated or determined by multiple analyses as directed in section 9.7 of method 6020A) in the ICSA, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

DoD requires the absolute value of concentration for all non-spiked elements <2xMDL, unless they are a verified trace impurity from one of the spiked elements.

10.11.2 Analytical results of Solution AB (ICSAB) shall fall within the control limits of ± 2 times the PQL or ±20% of the analyte's true value for the analytes included in the

ICSAB, whichever is greater. If not, the analysis shall be terminated and the problem corrected before recalibration.

10.12 Matrix Spike analysis is acceptable if recovery is within ±25%. If recovery is outside of this limit for any analyte then the associated samples are flagged. The only exception to this criteria is when the sample concentration exceeds the concentration of the spike added by a factor of four or more. In this case no flagging is applied. When recoveries fail to meet criteria, a post digestion spike (PDS) is required.

DoD recovery limits for MS analyses are the same as LCS limits. DoD QSM does not give a set of limits for method SW6020, therefore, method SW6010 LCS limits or 80-120% will be used (silver in soil is 75-120%).

10.13 Post digestion spike (PDS): When matrix spike recovery fails, a post digestion spike is performed on the same sample as the spike was done originally. Spike the unspiked aliquot of the undiluted digestate to produce a minimum level of 10 x PQL and a maximum of 100x PQL. PDS recoveries outside of ±20% of the true value confirm a matrix interference and the associated samples are flagged.

DoD allows PDS recoveries up to ±25%.

10.14 Duplicate: A control limit of ±20% for RPD is used for original and duplicate samples. Corrective action required as a result of the duplicate analyses would be to analyze a serial dilution on the sample.

10.15 Serial Dilution (SD): If the analyte concentration is sufficiently high (minimally, a factor of 10 times above the PQL), a 5 times dilution should agree to within ±10% of the original sample analyte determination. If not, a chemical or physical interference effect is suspected and the associated samples are flagged.

10.16 Laboratory Control Sample (LCS): Recovery is calculated for all analytes and are to be within the control limits of ±20%. In the event that the LCS does not meet criteria it may be reanalyzed once. If still unacceptable, associated samples must be redigested. Solid Reference Material (SRM) may be used as an LCS for soil sample batches. The manufacturer's established acceptance criteria should be used instead of the ±20% criteria.

10.17 Internal Standards (IS): Internal standard response is monitored throughout the analytical sequence. Ratios of the raw uncorrected IS responses between isotopes should also be monitored routinely. This information can be useful for correcting problems caused by mass dependent drift, errors incurred in the IS addition, or background contributions from samples that cause high bias. If the intensity of any internal standard in a sample falls below 70% of the intensity of that internal standard in the initial calibration standard S0, a significant matrix effect must be suspected. Under these conditions, the established PQL has degraded and the

correction ability of the internal standard technique becomes questionable. Make sure the instrument has not drifted by observing the internal standard intensities in the nearest clean matrix. If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample must be diluted fivefold and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit.

DoD Internal standard intensity limits are 30-120% of the IS in the initial calibration.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory supervisor or another senior chemist reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 The reporting group generates reports. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated in the event of an out of control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. These sheets are stored in the bookcase adjacent to the Organic Analysis lab. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Health and Safety Manual. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Method 6020A, Inductively Coupled Plasma- Mass Spectrometry. Revision 1, February 2007, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

Operation manual(s) for Thermo Electron Xseries2

http://www.cee.vt.edu/ewr/environmental/teach/smprimer/icpms/icpms.htm, internet reference for how an ICP/MS works.

Attachments:

Table 1:QC/ Standard Concentrations
Table 2:Calibration Standard Concentrations
Table 3: Internal Standards

Table 1:QC/ Standard Concentrations

ELEMENT	MASS	PPB 6020 LLICV	PPB 6020 ICV	PPB 6020 CCV	PPB 6020 ICSA	PPB 6020 ICSAB	PPB 6020 LCS	PPB 6020 MS
Be	**9**	**1**	**100**	**100**	**X**	**X**	**100**	**100**
B *	**10**	**100**	**1000**	**1000**	**X**	**X**	**1000**	**1000**
Na	**23**	**250**	**25000**	**25000**	**250000**	**250000**	**10000**	**10000**
Mg	24	250	25000	25000	100000	100000	10000	10000
Mg	**25**	**250**	**25000**	**25000**	**100000**	**100000**	**10000**	**10000**
Mg	26	250	25000	25000	100000	100000	10000	10000
Al	**27**	**20**	**2000**	**2000**	**100000**	**100000**	**4000**	**4000**
K	**39**	**250**	**25000**	**25000**	**100000**	**100000**	**10000**	**10000**
Ca	**44**	**250**	**25000**	**25000**	**300000**	**300000**	**10000**	**10000**
V	**51**	**2**	**100**	**100**		**200**	**1000**	**1000**
Cr	**52**	**2**	**100**	**100**		**200**	**400**	**400**
Cr	53	2	100	100		200	400	400
Fe	54	100	10000	10000	250000	250000	2000	2000
Mn	**55**	**1**	**100**	**100**		**200**	**1000**	**1000**
Fe	56	100	10000	10000	250000	250000	2000	2000
Fe	**57**	**100**	**10000**	**10000**	**250000**	**250000**	**2000**	**2000**
Co	**59**	**1**	**100**	**100**		**200**	**1000**	**1000**
Ni	**60**	**1**	**100**	**100**		**200**	**1000**	**1000**
Ni	61	1	100	100		200	1000	1000
Cu	**63**	**2**	**100**	**100**		**200**	**500**	**500**
Cu	65	2	100	100		200	500	500
Zn	**66**	**2**	**100**	**100**		**100**	**1000**	**1000**
Zn	67	2	100	100		100	1000	1000
Zn	68	2	100	100		100	1000	1000
As	**75**	**1**	**100**	**100**		**100**	**200**	**200**
Se	78	5	100	100		100	200	200
Se	**82**	**5**	**100**	**100**		**100**	**200**	**200**
Mo *	**95**	**2**	**100**	**100**	**2000**	**2000**	**500**	**500**
Mo *	97	2	100	100	2000	2000	500	500
Ag	**107**	**1**	**100**	**100**		**50**	**500**	**500**
Ag	109	1	100	100		50	500	500
Cd	**111**	**1**	**100**	**100**		**100**	**100**	**100**
Cd	114	1	100	100		100	100	100
Sb	**121**	**2**	**100**	**100**	**X**	**X**	**500**	**500**
Sb	123	2	100	100	X	X	500	500
Ba	135	10	1000	1000	X	X	4000	4000
Ba	**137**	**10**	**1000**	**1000**	**X**	**X**	**4000**	**4000**

Tl	203	1	100	100	X	X	200	200
Tl	**205**	**1**	**100**	**100**	**X**	**X**	**200**	**200**
Pb	**206-208**	**1**	**100**	**100**	**X**	**X**	**200**	**200**

Bold are Reported Mass
X= no requirement for 6020
*B and Mo not listed in 6020

Table 2:Calibration Standard Concentrations

		ppb	ppb	ppb	ppb	ppb	ppb
ELEMENT	MASS	S1	S2	S3	S4	S5	S6
Be	**9**	1	2	5	20	100	200
B	**10**			50	200	1000	2000
Na	**23**	250	500	1250	5000	25000	50000
Mg	24	250	500	1250	5000	25000	50000
Mg	**25**	250	500	1250	5000	25000	50000
Mg	26	250	500	1250	5000	25000	50000
Al	**27**	20	40	100	400	2000	4000
K	**39**	250	500	1250	5000	25000	50000
Ca	**44**	250	500	1250	5000	25000	50000
V	**51**		2	5	20	100	200
Cr	**52**		2	5	20	100	200
Cr	53		2	5	20	100	200
Fe	54	100	200	500	2000	10000	20000
Mn	**55**	1	2	5	20	100	200
Fe	56	100	200	500	2000	10000	20000
Fe	**57**	100	200	500	2000	10000	20000
Co	**59**	1	2	5	20	100	200
Ni	**60**	1	2	5	20	100	200
Ni	61	1	2	5	20	100	200
Cu	**63**		2	5	20	100	200
Cu	65		2	5	20	100	200
Zn	**66**		2	5	20	100	200
Zn	67		2	5	20	100	200
Zn	68		2	5	20	100	200
As	**75**	1	2	5	20	100	200
Se	78			5	20	100	200
Se	**82**			5	20	100	200
Mo	**95**	1	2	5	20	100	200
Mo	97	1	2	5	20	100	200
Ag	**107**	1	2	5	20	100	200
Ag	109	1	2	5	20	100	200
Cd	**111**	1	2	5	20	100	200
Cd	114	1	2	5	20	100	200
Sb	**121**		2	5	20	100	200
Sb	123		2	5	20	100	200
Ba	135	10	20	50	200	1000	2000
Ba	**137**	10	20	50	200	1000	2000
Tl	203	1	2	5	20	100	200
Tl	**205**	1	2	5	20	100	200
Pb	**206-208**	1	2	5	20	100	200

Table 3: Internal Standards

Concentrations chosen to yield approximately 700,000 raw counts

element	ppb
Li6	100
Sc	50
Y	15
Rh	20
In	15
Tb	15
Ho	15
Lu	10
Bi	15

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURES

for

Mercury Analysis in Aqueous and Soil Samples by Flow Injection Mercury System (FIMS) for Atomic Absorption

by

SW846 Method 7470A/7471B

Rev. 9

	Signature	Date
QA Director:		11/6/08
Lab Director:		11/6/08

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Mercury Analysis in Aqueous and Soil Samples
by Flow Injection Analysis System
for Atomic Analysis

by

SW846 Method 7470Aand 7471B

Rev. 9

1. Scope and Application

This SOP describes the procedures applicable to the preparation and analysis of mercury in aqueous and soil samples. Matrices include ground waters, aqueous samples, soils, sediments, sludges, and TCLP. All samples require digestion prior to analysis.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 The aqueous samples are digested with concentrated HNO_3, concentrated H_2SO_4, potassium permanganate and potassium persulfate at 95°C. The procedure converts various organically bound compounds and inorganic forms

of mercury into mercuric ions, which can be analyzed with a Flow Injection Analysis System (FIAS) for atomic spectroscopy. The soil/sediment samples are digested using aqua regia and potassium permanganate at 95±3°C and analyzed same as the aqueous samples.

3.2 The mercury ions formed during the digestion step are reduced to the elemental state and aerated into an absorption cell. Absorbance is measured at 253.7nm and is a function of mercury concentration.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For mercury analysis by SW 846 method 7470A water samples are collected in one-liter plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 All samples are stored at room temperature until analyzed.

4.3 Sample hold time for mercury analysis by SW 846 methods 7470A and 7471B is 28 days from date of sample collection.

5. Interferences and Potential Problems

5.1 Sulfides at levels above 20 mg/L or 20mg/Kg interfere. Potassium permanganate is added to the samples to eliminate possible interference of sulfides.

5.2 Copper at levels above 10 mg/L or 10mg/Kg interfere.

5.3 Seawaters, brines and industrial effluents high in chlorides interfere and require additional potassium permanganate for conversion to free chlorine. Free chlorine also absorbs radiation at 253.7nm. Therefore, the free chlorine is removed by addition of hydroxylamine sulfate reagent. In addition, the dead air space in the BOD bottle must be purged before adding the stannous sulfate.

5.4 Some volatile organic materials absorb at 253.7nm and may interfere.

6. Equipment and Apparatus

Equipment and instrumentation used in this analysis method include:

6.1 Equipment:

6.1.1 Perkin-Elmer FIMS 100.

6.1.2 Printer.

6.1.3 Wheaton BOD bottles.

6.1.4 Top loading balance - capable of accurate measurement to 0.01gram.

6.1.5 Hot Plate with graphite block digestor

6.2 Preventative Maintenance:

6.2.1 Pump tubing is replaced every 48 hours of instrument run time.

6.2.2 The windows of the optical cell are cleaned whenever the cell is replaced.

6.2.3 The inside of the optical cell is cleaned once every 48 hours of instrument run time.

6.3 Troubleshooting - Refer to the FIMS Analysis manual.

6.4 Glassware

6.4.1 100mL Class "A" volumetric flasks.

6.4.2 Class "A" volumetric pipettes ranging from 10µL to 1.0mL.

6.4.3 100 and 250mL Class "A" graduated cylinder or equivalent.

7. Reagents

7.1 Sulfuric Acid: Ultra Trace Grade

7.2 Nitric Acid: Ultra Trace Grade

7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Stannous chloride solution:
15 g $SnCl_2$ to 1000mLs of 3% HCl solution.

7.5 Sodium chloride-hydroxylamine sulfate solution:
12 g NaCl and 12g hydroxylamine sulfate to 100mLs of reagent water.

7.6 5% Potassium permanganate solution:
Dissolve 50g of $KMnO_4$ in 1000mLs of DI H_2O.

7.7 5% Potassium persulfate solution:
Dissolve 50g of $K_2S_2O_8$ in 1000mL of DI H_2O.

7.8 Stock mercury solution(Primary):
1000 mg/L purchased commercially.

7.9 Stock mercury solution(Independent Source):
1000 mg/L purchased commercially.

7.10 Mercury standards are stored in Wet Chem cupboards. Expiration dates for the standards are either the date one year from date of receipt or that designated by the manufacturer whichever comes first. Reagents and intermediate standards have a 28-day hold time.

7.11 3% HCl: 30mL concentrated HCl diluted to 1L with DI H_2O.

7.12 HCl: conc., Ultra Trace grade.

7.13 Aqua Regia: prepare immediately before use by carefully adding three volumes of conc. HCl to one volume HNO_3.

8. Procedure

8.1 Preparation

8.1.1 Aqueous Samples: Transfer 100mL of sample or an aliquot diluted to 100mL to a reagent water-rinsed BOD bottle with a graduated cylinder. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool samples and add 6mL sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved.

Record all volumes and reagents in the Mercury Digestion logbook , **Attachment 1**. Immediately prepare for analysis.

8.1.2 Soil Samples: Weigh a 0.50 – 0.60g portion of a well homogenized untreated sample and place in the bottom of a BOD bottle. See SOP 110.0039 for sub-sampling techniques. Add 5mL of reagent water and 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50mL reagent water and 15mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool and add 6mL of the sodium chloride – hydroxylamine sulfate solution to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved. Add reagent water to a final volume of 100mL. Pour an aliquot into a polyethylene tube for analysis. Be careful to avoid pouring the sediment into the tube. Record all volumes and reagents in the Mercury Digestion logbook, **Attachment 1**.

8.2. Calibration Standards

8.2.1 Working Calibration standards are prepared from a mercury intermediate standard at 500µg/L. The intermediate is prepared by pipetting 50µL of 1000mg/L Primary stock standard into a 100mL volumetric flask. Bring up to volume with 3% HCl.

Label standard as IIyymmdd#, where:

I I = Inorganic Intermediate
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

All standard preparation information is documented in the Metals Standard Receipt/Preparation Logbooks (Primary, Intermediate and Working).

Into BOD bottles containing approximately 10mL DI H_2O, pipet the following volumes of intermediate standard in order to achieve the corresponding final working standard concentrations:

Volume	Concentration (µg/L Hg)	Standard ID
40µL	0.2	S1
200µL	1.0	S2
400µL	2.0	S3

1mL	5.0	S4
2mL	10.0	S5
0mL	0.0	S0

8.2.2 Depending on the matrix of samples being prepared, follow the digestion as follows:

8.2.2.1 Soil Calibration standards, add 5mL of aqua regia. Heat 2 minutes on hot plate with graphite holders at 95±3°C. Cool; then add 50 mL reagent water and 15 mL of the potassium permanganate solution to each sample bottle. Mix thoroughly and place on hot plate with graphite holders for 30 minutes at 95±3°C. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. **CAUTION**: do this addition under the hood, Cl_2 could be evolved. Calibration working standards are prepped with each batch of samples and undergo a digestion of at least 30 minutes. Soil standards are brought up to 100mL final volume with DI H_2O prior to analysis.

8.2.2.2 Aqueous Calibration standards, add approximately 90 mL DI H_2O to each BOD bottle. Add 5mL concentrated H_2SO_4 and 2.5mL concentrated HNO_3, mix. Add 15mL of potassium permanganate solution. Additional permanganate may be required until the purple color persists for at least 15 minutes. Add 8mL potassium persulfate solution; mix. Heat for 2 hours at 95°C on hot plate with graphite holders. Cool, transfer to the Mercury Analysis Lab where 6mLs of the sodium chloride – hydroxylamine sulfate solution will be added to reduce the excess permanganate. Do not volumize to 100 mL with DI H_2O prior to analysis. **CAUTION**: do this addition under the hood, Cl_2 could be evolved

8.2.3 A Calibration Blank, ICB/CCB, is prepared as the S0 calibration standard.

Sample concentrations are not reported below the lowest calibration standard of 0.2ug/L (aqueous samples, or 0.033 mg/Kg for soils) without method modification. This effectively becomes the method reporting limit (MRL) also referred to as Project Quantitation Limits (PQL).

8.2.4 ICV/CCV Intermediate standards are prepared at 5.0µg/L from an **Independent Source** Hg Primary stock standard. The intermediate

Hg CCV standard is prepared by pipetting 50µL of 1000mg/L Hg into a 100mL volumetric flask. The standard is brought to volume with 3%HCl for a final concentration of 500ug/L.

8.2.5 The working ICV/CCV is prepared by pipetting 1mL of the above intermediate standard into a BOD bottle and digesting as in **section 8.2.2.** An ICV/CCV is prepped with each batch of samples. Final concentration is 5µg/L and the standard is labeled as a mercury working calibration standard:

Label standard as IWyymmdd#, where:

I W = Inorganic Working
yy = year of preparation
mm = month of preparation
dd = day of preparation and
# = Sequential letter of intermediate standards prepped on this day.

8.2.6 LCS/Matrix spike standard is also an intermediate concentration standard that is prepared by combining 45.5 uL of the **Independent Source** Hg Primary stock standard into a 100 mL volumetric flask and volumized to 100 mL with 3% HCl. The standard is labeled as for Hg working standards, as in **section 8.3**.

8.2.7 1mL of the above LCS/Matrix spike is added into 100 mL of DI H_2O and digested as an aqueous sample for the LCS-Water (LSCW) as in **section 8.1.1** . The true value of the LCS = 4.55µg/L at the instrument level.

8.2.8 1mL of the LCS/spike working spike is added to 0.6 g of acid-washed Teflon chips used to simulate a soil matrix for the LCS-Soil (LCSS). The true value of the LCS = 4.55µg/L at the instrument level.

8.2.9 1mL of the LCS/spike working spike is added to a chosen sample, and digested as per the sample matrix. This is the Matrix Spike. At the time of analysis, the spike = 4.55µg/L at the instrument level, times any sample dilution factor.

8.3 Instrumental Analysis

8.3.1 Adjust the argon pressure to 44psi for FIMS analysis. Turn on the FIMS system with the auto-sampler. Turn on the computer, printer. Allow the lamp to stabilize for 35-45 minutes.

8.3.2 Prepare the reductant solution, stannous chloride in **section 7.4**, and the carrier solution, 3% HCl in **section 7.11**.

8.3.3 Bring up the appropriate element file (Hg comm).

8.3.4 Set up the sample info file to coincide with the locations and sample identifications that will be analyzed in the run.

8.3.5 Fill out the Automated Control Window:

8.3.5.1 Type in a data file name.

8.3.5.2 Type in the name of the sample info file.

8.3.6 Load the auto-sampler tray.

8.3.7 Place the carrier tubing inlet into the carrier solution, and the reductant tubing inlet in the reductant solution. The reductant solution tubing has a red tab on it, and the carrier solution a yellow tab. Remove the cap from the gas chamber and start the pump to ensure it is working properly. Turn off the pump and replace the cap.

8.3.8 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the instrument run logbook.

8.3.9 On the AS-90 control window either click on "Run All" to run both standards and samples or "Calibrate" to run only the standards. If you choose only to calibrate at this time, you will need to click on "Reset Sampler" and "Run Samples" when calibration is complete. Analyze standards and samples. Samples that exceed the upper calibration range must be diluted and reanalyzed.

8.3.10 When analysis is complete place both carrier tubing inlet and the reductant tubing inlet with the auto-sampler probe in a beaker of DI water. Allow the water to pump through the system. Continue to flush all the water through until no more bubbles appear in the waste tubing. Turn off the pump.

8.3.11 Turn off the FIMS unit.

8.3.12 No rinse between samples is necessary per manufacturer's instructions.

8.3.13 The following Analytical Sequence should be used.

1. Calibration Blank(S0)
2. Standard #1 (S1)
3. Standard #2 (S2)
4. Standard #3 (S3)
5. Standard #4 (S4)
6. Standard #5 (S5)
7. ICV
8. ICB
9. Method blank
10. LCS
11. Samples (≤6)
12. CCV
13. CCB
14. MS
15. Sample Duplicate
16. Serial dilution
17. Samples (<5)
18. CCV
19. CCB

8.3.14 All information including the analytical sequence is documented in the FIMS 100 Run Logbook, **Attachment 2.**

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of ug/L for aqueous samples, and ug/Kg dry weight for soils/solids. Results are reported to two significant figures.

9.1.1 For aqueous results, report the data generated directly from the instrument with allowance for sample dilution. Upload the data from the FIMS directly into the Omega LIMS system for reporting.

9.1.2 For soil/solid samples, upload the data as for aqueous samples. The LIMS will calculate the final results in soil units, with allowance for dilution, sample weight and percent moisture.

9.2 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)

where: SSR = spiked sample result
SR = sample concentration

SA = spike added

9.3 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10 Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Linear correlation for the standard curve must be **≥0.995**. The Calibration curve must not be forced to go through the origin zero point.

10.3 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method Blanks must not contain Mercury at a concentration ≥ the MRL. If mercury is present in the Method Blank and the lowest sample concentration in the batch exceeds 10 times the blank concentration, no corrective action is needed. Otherwise, corrective action for method blank contamination involves determining the source of the contamination and re-prepping the entire batch.

DoD QSM - Method blank concentrations must be less than 1/2 RL.

10.4 Calibration verification – A second source ICV prepped with the associated samples is analyzed immediately after the curve and must be within 90-110% of its true value. The ICV concentration is at the mid-level of the calibration curve.

10.5 CCVs are analyzed at least every 10 samples with 80-120% recovery requirements.

10.6 An ICB/CCB is run immediately after the ICV/CCV set; the mercury value in the ICB/CCB is not to exceed the MRL.

DoD – Mercury is not to exceed 2 times MDL in the ICB/CCB.

10.7 A matrix spike and a matrix duplicate are prepped and analyzed with every batch not to exceed 20 samples. The RPD for duplicates is 20% and the Aqueous spike recovery control limit is 75-125%. The soil spike recovery control limit is 80-120%.

DoD – Spike recoveries must be within 80-120% for both water and soil

10.8 Laboratory Control Sample (LCS) -An LCS is prepped with a minimum of every 20 samples of the same matrix. Control limits are 80-120% of the true value for mercury. If the LCS is outside the acceptance limits, the corresponding samples are re-prepped and reanalyzed. Corrective action includes re-digestion/reanalysis for all samples and QC in the batch.

10.9 Dilution test (serial dilution) - A dilution test is performed once for each analytical run or if a unique sample matrix is encountered. If the analyte concentration is at least 25 times the MDL, an analysis of a 5 times dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected.

DoD- Serial Dilution is required once per preparation batch.

10.10 Sample concentrations that exceed the highest calibration are diluted and rerun so that their concentration fall within the calibration range.

10.11 The Inorganic Laboratory Supervisor/Manager authorizes any method deviations.

10.12 The intermediate standard is stable for 28 days. All standards made from a primary standard expire on or before the primary standard's expiration date.

10.13 Method detection limits (MDLs) are established once a year. The MDL is obtained by multiplying the standard deviation of seven or eight analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the expected MDL. An MDL verification check is performed immediately following MDL study, and quarterly each year in lieu of the annual MDL study if desired. An MDL verification check sample is spiked at approximately 2 times the current MDL.

10.14 Hg lamp intensity, measured as absorbance for the 10ug/L mercury standard, is recorded daily in the maintenance logbook. Trends are to be monitored to show the instrument is within the optimal absorbance range. The normal/optimal absorbance range is from 0.17-0.24.

11. Data Validation and Reporting

11.1 Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the Supervisor/Lab Manager. The Supervisor/Lab Manager reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory. After each review, the appropriate section of the Data Review Checklist is checked off, **Attachment 3**.

11.2 Reports are generated by the data reporting group. The data submitted for report preparation is dependent on project requirements.

11.3 Data is always reported to the MRL. If clients require reporting limits different from the MRL/PQL, the data will need to be reported using a special form set through the Omega LIMS system depending on project needs.

11.4 Electronic files (EDD) are generated by the Data Management Department and are stored in the server until downloaded to tapes by the server backup system.

12. Corrective Action Procedures

12.1 Corrective Action to be implemented in the event QC results are outside of the acceptance range are covered in **section 10**.

12.2 Corrective Action reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a corrective action report for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. These sheets are stored in the Health & Safety Officer's office. In addition, laboratory personnel

should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric, sulfuric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 The cell-heating compartment maintains a temperature of 100°C throughout the analysis. Care should be taken to avoid burns from the cell-heating compartment.

13.4 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency: Test Methods for Evaluating Solid Waste, Update IIB, SW-846 Method 7470A and Update IV SW-846 Method 7471B.
2. Department of Defense, Quality Manual for Environmental Laboratories. Final Version 3, January 2006.

Attachments:

Attachment 1: Mercury Digestion Logbook
Attachment 2: FIMS Instrument Logbook
Attachment 3: Data Review Checklist Form

Attachment 1
Mercury Digestion Logbook

MITKEM LABORATORIES

Mercury Digestion Logbook

						Reagents Added							
Date	Bottle No.	Sample ID		Client ID	Sample Vol (ml)/ Wt (g)	Conc. H_2SO_4 (ml)	Conc. HNO_3 (ml)	5% $KMnO_4$ (ml)	5% K_2SO_4 (ml)	Aqua-regia (ml)	Final Volume (ml)	Comments	Analyst

Waters
In: _____
Out: _____
Matrix: Aqueous

Soils
In: _____ Out: _____
In: _____ Out: _____
Matrix: Soil/Solid

LCSS ID _____
LCSS volume: _____
Spike ID _____
Spike volume: _____

Temp: _____ °C
H_2SO_4 Lot # _____
HNO_3 Lot # _____
HCl Lot # _____
KMnO4 ID # _____
K2S2O8 ID# _____
Method # _____

RELINQUISHED TO: _____

Reviewed by: _____

Logbook ID: 100.0128 - 10/08

Attachment 2
FIMS Instrument Logbook

MITKEM LABORATORIES: FIMS 100 RUN LOGBOOK

Date: **Analyst:**

Analysis:				Analysis:				Analysis:				Analysis:			
SEQ#	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**		**SEQ#**	**Bottle ID**	**LAB ID**	
1				26				51				76			
2				27				52				77			
3				28				53				78			
4				29				54				79			
5				30				55				80			
6				31				56				81			
7				32				57				82			
8				33				58				83			
9				34				59				84			
10				35				60				85			
11				36				61				86			
12				37				62				87			
13				38				63				88			
14				39				64				89			
15				40				65				90			
16				41				66				91			
17				42				67				92			
18				43				68				93			
19				44				69				94			
20				45				70				95			
21				46				71				96			
22				47				72				97			
23				48				73				98			
24				49				74				99			
25				50				75				100			

Calibration/ICV Prep Date:_____ SnCl2 _____ HCl: _____

Reviewed By: _____ NaCl: _____ Hydroxylamine Sulfate: _____

Logbook ID: 100.0164-05/08

Attachment 3
Data Review Checklist Form

Mitkem Laboratories,
A Division of Spectrum Analytical, Inc.
Batch Data Review Checklist - Inorganics

Analysis:_____

Level 1 (Analyst) Review:_____ Level 2 (Technical) Review:_____

Workorder:_____

	Yes	No --List/Explain any Unusual Occurrences or Nonconformances
Calibration Acceptable *ICAL / CCAL*		*List all non-conforming project analytes*
LCS Acceptable		*List all non-conforming project analytes*
Blank Acceptable		*List all non-conforming project analytes*
Spike Acceptable		*Reasonable recovery / Matrix effect / Spike to sample concentration ratio*
Dup Acceptable		*Reasonable precision / Sample non-homogeneity?*
Serial Dilution		*List all non-conforming analytes, Matrix effect?*
Post-Digestion Spike		*Matrix effect?*
Within Holding Time		*List runs/re-runs out of holding time; Explain*
Within Analysis Range		*Dilutions properly noted; Explain any "E" flag analytes or dilutions with no target hit*
Calc/Data Xfer Check		*Including proper significant figures, rounding*
Reasonableness Check:		*Compared to historic or on-going trends, or for this analysis in general?*

Notes:

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion for ICP Analysis

by

SW846 Method 3050B

SOP No. 100.0104

Rev. 7

	Signature	Date
QA Director:		3/17/09
Lab Director:		3/20/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Preparation of Soil Samples by Acid Digestion for ICP Analysis

by

SW846 Method 3050B

Rev. 7

1. Scope and Application

This Standard Operating Procedure (SOP) deals with the preparation of soil samples utilizing USEPA SW846 Method SW3050B for analysis by Methods SW6010 and 6020. Discussion includes sample digestion and sample concentration technique for the analysis of metals in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. Supervisors/lab managers are responsible for ensuring that this SOP is accurate and up to date and that it is implemented appropriately. Supervisors/lab managers review logbooks and data generated for this procedure and approve all reported results.

3. Summary of Procedure

A 1.00 to 2.00 gram soil/solid sample is digested with the addition of acids and hydrogen peroxide for metals analysis by Inductively Coupled Argon Plasma (ICAP). This method has been adapted to utilize polyethylene digestion tubes and a final 50mL digestate volume, rather than the 100mL volume noted in the original method. The reagent volumes have been reduced to accommodate the method as well.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Hold time for ICP analysis is 180 days from date collected.

4.2 Samples are stored at 4°C in amber glass jars with Teflon lined caps.

5. Interferences and Potential Problems

Possible sources of contamination:

5.1 Hood fall-out.

5.2 Acid bath for glassware.

5.3 Acid dispensers.

5.4 Sample matrix effects: Extreme organic samples.

5.5 Disodium Stannate preservative for peroxide: Do not use this peroxide when analyzing tin in soils. An alternative peroxide source must be used.

6. Equipment and Apparatus

Equipment used in this preparation method include:

6.1 10% HNO_3 acid bath.

6.2 50 mL polyethylene tubes.

6.3 Watch Glasses.

6.4 Hot plates.

6.5 Centrifuge.

7. Reagents

The following reagents are used during sample preparation:

7.1 Concentrated HNO_3, Fisher, ACS trace metals grade.

7.1.1 1:1 (v/v) HNO_3, Fisher, ACS trace metals grade.

7.2 Concentrated HCl, Fisher, ACS trace metals grade.

7.3 30% Hydrogen peroxide (H_2O_2), Fisher, ACS certified.

7.4 Spiking solutions, High Purity Standards.

7.5 Laboratory Control Sample Solutions, High Purity Standards.

7.6 Teflon chips, Chemware Ultra PTFE Boiling Stones.

8. Procedure

See SOP 110.0039 for sub-sampling procedures.

8.1 Digestion of soil samples for ICAP analysis:

8.1.1 Note in Soil/Solid Sample Preparation Logbook the presence of any artifacts (see **Figure 1**).

8.1.2 Mix the sample thoroughly so that a homogenous representative aliquot can be taken. Weigh an approximate1.00 to 2.00 gram (wet weight) or 1.00 gram (dry weight) aliquot of the sample to the nearest 0.01g and transfer it to a 50 mL digestion tube.

8.1.3 Add 5 mL of 1:1 (v/v) HNO_3, mix well. Cover the tube with a ribbed watch glass. Heat to 95°C ± 5 °C in graphite holders on hotplates in the hood, and reflux for 10 minutes without boiling.

8.1.4 Remove from hotplate and allow to cool.

8.1.5 Add 2.5mL of conc. HNO_3. Replace on hotplate and reflux for 30 minutes. If brown fumes are generated at this step repeat the addition of 2.5mL conc. HNO_3 with heating steps until no more brown fumes are given off. Do not allow the volume to fall below 5mL.

8.1.6 Allow the sample to cool and add 1mL of DI water and 1.5 mL of 30% H_20_2. Return the tube to hotplate to start peroxide reaction, making sure no losses occur due to excessive effervescence action. Heat until effervescence subsides and then allow tube to cool

8.1.7 Continue adding 30% H_2O_2 in 0.5mL aliquots with warming until minimal effervescence occurs, the appearance of the sample remains unchanged, or 5mL have been added. If the blank or a sample no longer have effervescence, set them aside, do not continue to add H_2O_2. When all are done proceed to next step.

Note: do not add more than a total of 5mL H_2O_2

8.1.8 ***For ICP-AES samples only:*** Add 5mL of concentrated HCl and return to the hotplate and reflux for an additional 15 minutes.

8.1.9 Take tube off the hotplate and cool to room temp.

8.1.10 Volumize to 50mL with DI Water.

8.1.11 Centrifuge sample for approximately 5 minutes at 3000 rpm to settle any insoluble material.

8.1.12 Label the tube properly. Transfer digestate to Inorganic Instrument Lab. All digestion information is documented in the Soil/Solid Sample Preparation Logbook **(figure 1)**.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are established to ensure generation of data of known quality. QA/QC procedures associated with the Inorganic prep lab include preparation of Method Blanks, Lab Control Samples, matrix spikes and sample duplicates.

10.1 Preparation Blanks:

A method blank must be run with every discrete batch of samples that is being prepped. A batch of samples cannot exceed 20 samples.

10.1.1 Use 1.0 ±.01 g acid washed Teflon chips as a "soil blank" and treat it as if it were a regular sample. Label appropriately.

10.2 Laboratory Control Samples:

A laboratory control sample containing all target analytes must be prepped and analyzed for each batch of samples. The number of samples per batch cannot exceed 20.

10.2.1 For ICP-AES soil samples measure 1.0 ±.01 g acid washed Teflon chips into a 50mL -digestion tube. Spike 455 uL of CV-1 and 45.5 uL each of CV-2 and CV-3 into the tube and digest as described in **Section 8**. Label appropriately.

10.2.2 For ICP-MS soil samples measure 1.0 ±.01 g acid washed Teflon chips into a 50mL -digestion tube. Spike 200uL of CV-1 and 20uL each of CV-2 and CV-3 into the tube and digest as described in **Section 8**. Label appropriately.

10.3 Matrix Spikes and Duplicate Samples:

Prepare 3 digestion tubes to be used for the same aqueous sample. Designate one tube as the sample, one as the duplicate of that sample, and the other as a spiked portion of the sample. Into each tube, measure 1.00-2.00 g (wet weight) or 1 gram (dry weight) of sample (the same sample).

10.3.1 Matrix Spikes:

- For ICP-AES soil samples: To one aliquot add three spiking standards containing all target analytes; High Purity Standard CLP-CV-1 at 455 uL; CLP-CV-2 and CLP-CV-3 at 45.5 uL each. Label this sample with an "MS" suffix, and digest as described in **Section 8**.

- For ICP-MS soil samples: To one aliquot add three spiking standards containing all target analytes Spike 200uL of CV-1 and 20uL each of CV-2 and CV-3 into the tube and digest as described in **Section 8**. Label this sample with an "MS" suffix, and digest as described in **Section 8**.

10.3.2 Duplicates: Label the tube designated for the sample duplicate with a "D" suffix and digest as described in **Section 8**.

10.4 Standard Preparation

All standards made from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

Data (logbook entries) generated in the inorganic preparation laboratory will be reviewed and signed by a peer, the supervisor or the department manager.

12. Corrective Action Procedures

All corrective action will stem from the analytical results in the Metals Laboratory. See the specific SOP for details on QC requirements.

12.1 If any method blank shows contamination, associated samples are scheduled for reprep by the department supervisor or manager.

12.2 If any spike recovery or duplicate RPD is outside of control limits, samples are scheduled for reprep by the department supervisor or manager.

12.3 If a Spike sample was not spiked, it will be re-prepped and re-analyzed with the appropriate sample and duplicate.

13. Health and Safety

Health and safety hazards in the Inorganic Preparation Laboratory (prep lab) include exposure to concentrated acids, their fumes and toxic metals standards. Labcoats, gloves and safety glasses must be worn in the prep lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

U. S. Environmental Protection Agency. Test Methods for Evaluating Solid Waste, SW-846, Update III, Revision 2, December 1996, Method 3050B.

Attachments:

Figure 1: Soil/Solid Sample Preparation Logbook

Figure 1

Soil/Solid Sample Preparation Logbook

MITKEM LABORATORIES: Soil/Solid Metals Prep Logbook

Date	Sample ID	Sample Wt. (g)	Sample Color Before	Sample Texture	Artifiacts (Y/N)	1:1 HNO_3 (ml)	Conc. HNO_3 (ml)	30% H_2O_2 (ml)	Conc. HCl (ml)	Sample Color After	Sample Clarity After	Final Volume (ml)	Comments

HCl Lot# _____ Analyst: _____ Digestion Temp: _____oC

HNO3 Lot# _____ Method: _____ LCS/Spike Lot No.: _____

H2O2 Lot# _____

RELINQUISHED TO: _____ Reviewed By: _____ Narrative Notes on Reverse Side

Yes / No

Logbook ID 100.0124-10/08

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil by Inductively Coupled Argon Plasma Atomic Emission Spectrometry using Method SW846 6010C

Rev. 12

	Signature	Date
QA Director:	Sharyn B Lauh	2/3/09
Lab Director:	Kin Chui	2/3/09

MITKEM LABORATORIES,
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Determination of Metals in Water and Soil
by Inductively Coupled Argon Plasma Atomic Emission Spectrometry
using Method SW846 6010C
Rev. 12

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the elements listed in **Attachment 1**. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. **Section 8.1** provides the Method references for sample digestion procedures. See **LIMS Test Information/ Test/Limits** for analytes and their associated MDL/PQLs.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve all reported results.

3. Summary of Procedure

3.1 Prior to analysis, samples must be digested using appropriate sample preparation methods.

3.2 The method measures element specific emitted light by optical spectrometry. The samples are nebulized and the resulting aerosol is transported to the plasma torch. The metals pass through the hot zone of the plasma, where they take up energy. Subsequently the metals pass through the cold zone (relatively) of the plasma where they give up the excess energy at element specific wavelength. The spectra are dispersed by a grating spectrometer, and the intensity of the emitted light is measured by a solid state photomultiplier. Background correction is required for trace element determination. 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 interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured.

4. Sample Preservation, Containers, Handling, and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For metals analysis by Method 6010C, water samples are collected in 500 ml plastic containers and preserved (acidified) with nitric acid to a pH of less than 2. Soils are collected in 8-ounce glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Soil samples are stored at 4°C ± 2°C until analyzed.

4.3 Sample holding time for metals analysis by method 6010C is 180 days from the date of sample collection for both water and soil.

5. Interferences and Potential Problems

Several types of interference effects may contribute to inaccuracies in the determination of an analyte by ICAP-AES.

5.1 Spectral interferences - Can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena. The first of these can be compensated for by utilizing a computer correction of raw data, requiring monitoring and measurement of the interfering element. The second effect may require selection of an alternative wavelength. In addition one could select an alternate wavelength where interference is minimal or absent. The 4300DV and the 3100XL used at Mitkem have many spectral lines from which to choose. The third effect can usually be compensated by a background correction adjacent to the analyte line.

5.2 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 salts or high acid concentrations. If physical interferences are present, they must be reduced by such means as a high-salts nebulizer, diluting the sample, using a peristaltic pump, or using an appropriate internal standard element. Another problem that can occur with high dissolved salts is a salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. This problem can be controlled by a high-salts nebulizer, wetting the argon prior to nebulization, using a tip washer, or by diluting the samples. A mass flow controller is used to control the argon gas flow rate.

5.3 Chemical interferences include molecular-compound formation, ionization effects, and solute-vaporization effects. These effects can be minimized by careful selection of

operating conditions, by buffering of the sample, by matrix matching, and by standard-addition procedures.

5.4 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, or from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with the rinse solution between samples. This method requires a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed using a longer rinse period.

5.5 Physical, chemical and spectral interferences are primarily attributed to the sample matrix. If interference caused by a particular sample matrix is known, in many cases it can be circumvented. However, when the nature of the sample is unknown, following tests can be used to ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.5.1 Dilution Test -If the analyte concentration is sufficiently high (minimally a factor of 10x above the method detection limit (MDL)), an analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, a chemical or physical interference effect could be suspected.

5.5.2 Post Digestion Spike Addition - If the matrix spike (pre-digestion) recovery falls outside of the control limits (75% - 125%), matrix interference is suggested. In this case an analyte spike is added to a portion of a prepared sample or its dilution at a level just below the mid-point of the calibration curve. Post digestion spikes should be recovered to within 80% - 120% of the known value. If not, a matrix effect should be suspected.

5.5.3 Comparison with alternative method analysis-when investigating a sample matrix, comparison tests may be performed with other analytical techniques, such as atomic absorption spectrometry, or ICP-mass spectrometry. This should only be done after consultation with the client.

5.5.4 Internal Standard Addition technique can be used (Refer to Section 4.4.2 of Method 6010C.)

6. Equipment and Apparatus

6.1 Inductively coupled argon plasma emission spectrophotometer (ICAP).

The ICAPs used at Mitkem are a Perkin-Elmer Model 4300DV and a 3100XL. The 4300DV is outfitted with an AS-93plus, 157-position autosampler and a high precision, three channel, peristaltic pump. The 3100XL is outfitted with an AS-91, 160-position

autosampler and a high precision, three channel peristaltic pump. Both ICPs have axial viewing capability, as compared to the more traditional radial viewing ICAP. Axial viewing provides greater sensitivity for all elements analyzed. The solid state detector is capable of analyzing at approximately 6000 wavelengths.

The built-in radio-frequency generator is FCC compliant.

The systems are computer controlled through a 32-Bit, Microsoft Windows NT operating system. This system allows for great flexibility in controlling the instrument.

The required high purity argon gas is piped in from a main storage tank that is located in the rear of the building. The gas is stored in liquid form, drawn-off as a gas and then piped into the building for distribution to the instruments. The liquid argon supply is monitored remotely by the supplier, and resupplied on an automatic-delivery basis.

6.1.1 Operating conditions - The analyst should follow the instructions provided by the instrument manufacturer. Instrument detection limits, linear dynamic ranges, and interference effects are established for each analyte line used. All measurements must be in the instrument linear range where spectral interference correction factors are valid. 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.

6.1.2 Daily emissions of the highest standard for arsenic, copper, lead and selenium are recorded as a means to ensure the plasma is stable before analysis and also to chart potential problems especially with the power tube, sample introduction system, or RF generator. The normal range of the four analytes above are (the unit is intensity generated by instruments):

Optima 2 (3100 XL)	Optima 3 (4300 DV)
As 2,000 - 3,000	As 2,000 - 2,500
Cu 600,000 - 900,000	Cu 400,000 - 600,000
Pb 12,000 - 20,000	Pb 4,000 - 8,000
Se 2,000 - 4,000	Se 1,800 - 2,400

6.1.3 For analysis of normal environmental samples use the following standard operating conditions:

Wattage: 1450 – 1500
Argon flow rate (L/min): 15
Nebulizer flow rate (L/min): 0.55
Sample flow rate (mL/min): 2
Rinse time (sec): 60
Read delay (sec): 60

6.1.4 For regular day-to-day ICAP measurements, the plasma need not be optimized prior to analysis. The plasma needs to be optimized each time a major change occurs in instrument configuration, such as replacement of the torch and/or maintenance, unless response is still within normal ranges. Responses and alignment are routinely checked after torch cleaning or replacement with the analysis of a 1 mg/L Mn solution for axial view calibration and 10 mg/L Mn solution for radial view calibration. "Optimization" is a function of a series of procedures; refer to the Mitkem SOP 100.0006 for additional information on how to perform general maintenance and troubleshooting procedures.

6.1.5 To optimize the plasma, follow the instructions in the Optima Software Guide.

6.2 ICAP Instrument Maintenance:

6.2.1 Preventative Maintenance:

- Peristaltic pump tubing will be replaced every 16 hours of instrument time or sooner if memory effects are manifested.
- The plasma torch is cleaned using concentrated HNO_3 when torch and sample injector buildup is noted.
- The spray chamber and nebulizer are cleaned approximately every month as needed. Replacement is done when needed.
- Air filters are cleaned once every two weeks or as needed upon visual inspection.
- The RF coil, window and cone are cleaned every 2-3 weeks.
- The instrument undergoes extensive maintenance by a manufacturer's service engineer as needed.

6.2.2 Troubleshooting:

6.2.2.1 Sudden drop in CCV concentration occurs in a run:

- Check to see that sample introduction system (tubing) has not become disconnected.
- Check to see if a clog has occurred in the sample introduction system.
- Check CCV Standard to ensure it is not empty.
- Check room temperature to see if a fluctuation has occurred.

- Recalibrate and rerun if necessary.

6.2.2.2 CCVs drift up and down:

- Remake the CCV standard.
- Check room temperature to see if a fluctuation has occurred.
- If problem persists, call Perkin-Elmer. The problem is usually indicative of a power tube failure.

6.2.2.3 Plasma goes out during analysis:

- Sample is not reaching spray chamber (tubing came apart or there is a clog)
- High levels of salt in the sample caused plasma temperature changes.
- The sample injector is coated with high salt material and needs to be changed.
- The power tube is going (after all others fail repeatedly) and Perkin-Elmer must be called for service.

6.3 Glassware:

6.3.1 Class A volumetric flasks: 100 mL and 50 mL.

6.3.2 Class A volumetric pipettes:

6.3.3 10-100µL adjustable Eppendorf.

6.3.4 100-1000 µL adjustable Eppendorf.

6.3.5 5mL adjustable Fisher Scientific.

6.3.6 100µL and 1mL fixed Wheaton.

6.3.7 10 mL Wheaton ICP tubes.

7. Reagents and Standards

All standard solutions (multi-, and single element), and second source QC solutions are purchased from outside vendors (Perkin-Elmer, Accustandard, Inorganic Ventures, ERA, and High Purity Standards, Inc.). All these solutions are traceable and meet with Mitkem's high purity requirements. ***Please note that standards and reagents from other vendors could be***

used as long as the standards are of high purity (>96%0 and traceable to reference materials. All standards are labeled as instructed in Mitkem SOP # 80.0001 Standard Preparation, Equivalency and Traceability.

7.1 Hydrochloric acid (conc.), HCl, Trace Grade, Fisher Scientific.

7.2 Nitric acid (conc.), HNO_3, Trace Grade, Fisher Scientific.

7.3 Reagent water (ASTM Type I water). Mitkem's water system consists of a Culligan high volume, 1 Megohm feed water system, combined with a Millipore Milli-Q, four bowl, high purity system. Reagent water is also referred to as DI water.

7.4 Mixed calibration standard solutions. Mixed calibration standards, obtained from High Purity Standards, come in three stock solutions. Calibration standards and QC solutions are made up on a daily basis.

7.4.1 CLP CAL 1 consists of two solutions:

- CLP CAL 1B contains silver only.
- CLP CAL 1A contains all other elements other than antimony, arsenic, cadmium, lead, thallium, and selenium.

7.4.2 CLP CAL 2 contains antimony only.

7.4.3 CLP CAL 3 contains arsenic, cadmium, lead, thallium, and selenium.

7.5 High standard (**ICP Standard 1**):

- Pipet 1mL HNO_3 (conc) and 1.5mL HCl (conc) into a 100mL volumetric flask.
- Pipet 1mL each of CLP CAL 1A & 1B stock solution and 0.1mL each of intermediate CLP CAL 2 and 3 into the flask.
- Pipet 0.1mL of Titanium single element standard into the flask.
- Bring to volume with DI water.

7.6 Second (middle) standard (**ICP Standard 2**):

- Pipet 5mL of a 1% HNO_3 and 1.5% HCl acid mixture into a 10 mL ICP tube.
- Add 5mL of the ICP Standard 1.

7.7 Third (low) standard (**ICP standard 3**):

- Pipet 10mL of 1% HNO_3 and 1.5% HCl solution into a 10 mL ICP tube.
- Withdraw 100µL.
- Add 100µL of ICP Standard 1 and mix well.

See **LIMS Test Information/ Test/SPEC** for concentrations of the Initial Calibration Standards.

7.8 **Second source** ICV/CCV standards (CCV100XCONC and Antimony (Sb) Stock solutions) are obtained from Accustandard.

- Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl (conc.) into a 100mL volumetric flask.
- Pipet 1.5mL (for ICV)of CCV100XCONC stock solution into the flask.
- Pipet 0.15mL of Antimony (Sb) Stock solution into the flask.
- Bring to volume with DI water.
- Transfer the solution to a 50mL plastic ICP tube.
- This standard is prepared as needed (in amounts consistent with the ratios above) usually every 1 – 2 days.

See **LIMS Test Information/ Test/SPEC** for analytes, concentrations and recovery limits for the ICV/CCV.

7.9 QC Standards ICSA and ICSB solutions are obtained from High Purity Standards.

ICSAB solution: In addition to the normal element composition of the ICSA solution, sodium, potassium, lead and selenium are added to the ICSAB solution. These stock solutions are also obtained from High Purity Standards.

7.9.1 Pipet 1mL of HNO_3 (conc.) and 1.5mL HCl into a 100mL volumetric flask.

7.9.2 Pipet 1mL of ICSB stock solution (ANALCS-R) into the flask.

7.9.3 Pipet 10mL of ICSA (CLP-INF-1) stock solution into the flask.

7.9.4 Pipet 0.5mL of the sodium/potassium stock solution (INFCS-5) into the flask.

7.9.5 Pipet 1mL of 45mg/L Pb intermediate standard solution into the flask.

7.9.5.1 The intermediate is prepared by adding 4.5mL of Pb at 1000mg/L (#100028-1) up to 100mL with 1%HNO_3 and 1.5% HCL acid solution in a volumetric flask.

7.9.6 Pipet 1mL of 45mg/L Se intermediate solution into the flask.

7.9.6.1 The Selenium intermediate standard is prepared by adding 4.5mL of Se at 1000mg/L (#031) up to 100mL with 1% HNO_3 and 1.5% HCl acid solution in a volumetric flask

7.9.7 Bring to volume with DI water.

7.9.8 Transfer the solution to a 10 mL ICP tube.

See **LIMS Test Information/ Test/SPEC** for concentrations of the ICSA and ICSAB.

7.10 Laboratory control samples (LCS) and Matrix spikes: The LCS standards for soils and waters are obtained from High Purity Standards.

- LCS/Spike standard 1 is prepared by adding 455uL of CLP-CV-1 to the digestion tube.
- LCS/Spike standard 2 and 3 are prepared by adding 45.5uL each of CLP-CV-2 and CLP-CV-3 to the digestion tube.

See **LIMS Test Information/ Test/SPEC** for final concentrations of the LCS/MS.

7.11 Linear Dynamic Range

The stock linear dynamic range (LDR) standards are purchased from Ultra Scientific, Inorganic Ventures and High Purity. The LDR is determined as the highest concentration of standard in which the determined value is within 10% of the true value.

- *LDR Standard I* is prepared by addition of 1 mL of Pb at 1000 mg/L, volumized to 10 ml with 1% HNO3 and 1.5% HCl. Final concentration 100mg/L.
- *LDR Standard II* is prepared by addition of 5 mL of mix QCS-19 and 0.5mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 50 mg/L.

- *LDR Standard III* is prepared by addition of 2.5 mL of mix QCS-19 and 0.25 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 25 mg/L.

- *LDR Standard IV* is prepared by addition of 1.0 mL of mix QCS-19 and 0.1 mL of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 10mg/L.

- *LDR Standard V* is prepared by addition of 0.5 mL of mix QCS-19 and 0.05ml of Ba at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 5mg/L.

- *LDR Standard VI* is prepared by addition of 0.5 mL of Al and 0.5ml of Mg at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard VII* is prepared by addition of 3.0 mL of Fe at 1000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 300mg/L.

- *LDR Standard VIII* is prepared by addition of 0.5 mL of Ca at 10,000 mg/L, volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 500mg/L.

- *LDR Standard IX* is prepared by addition of 0.5 mL of mix INFCS-5 (High Purity) and volumized to 10 mL with 1% HNO3 and 1.5% HCl. Final concentration 250mg/L.

8. Procedure

8.1 The methods in SW-846 for sample digestion or preparation are as follows:

8.1.1 Method 3005 , SOP No.100.0003, prepares ground water and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with hydrochloric and nitric acids prior to metal determination.

8.1.2 Method 3010 , SOP No.100.0003, prepares aqueous samples, mobility-procedure extracts and waste samples that contain suspended solids for total metal determination by ICP. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

8.1.3 Method 3050 , SOP No.100.0104, prepares solid waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrochloric acid.

8.2 The wavelengths and background correction locations in the reference method can be substituted if they can provide the needed sensitivity and are corrected for spectral interferences. The analyst should follow the instrument manufacturer's instructions, and if possible, approximate the recommended operating conditions.

For each analyte there are a number of possible wavelengths at which analyses could be made. The wavelengths used were selected based on consultations with the ICP specialists at Perkin-Elmer and our own experience.

8.3 Background correction factors are obtained by aspirating a concentrated solution, usually about 200-500ppm, of the interfering element, and measuring the resulting concentrations at the wavelength used. The background correction factors for that interfering element are obtained by dividing the measured concentrations by the actual concentration. For example: by aspirating a 500ppm solution of aluminum, one measures a cadmium concentration 0f 0.010ppm. The aluminum correction factor for cadmium is (0.010)/500 = 0.000020. This means that if the aluminum concentration in a sample is, say, 120ppm, one must subtract 120*0.000020 = 0.0024ppm from the measured cadmium concentration in that sample.

8.4 Interelement Correction (IEC) factors are established quarterly (or biannually at a minimum), for the major salts: aluminum, calcium, iron, magnesium, and also for chromium, copper, manganese, nickel, thallium, titanium and vanadium. Correction factors should not exceed 20% difference from the previous IEC values.

8.5 Linear Dynamic Range is performed quarterly (or biannually at a minimum). Following instrument calibration, solutions with varying concentrations (**section 7.5.13**) of each analyte (LDR standards) are analyzed. The highest concentration, within ±10 % of the true value, establishes the linear range.

8.6 Allow the plasma to become thermally stable before beginning the analyses. This usually requires at least 30 minutes. If the plasma is (for whatever reason) extinguished and needs to be re-lit, subsequent re-stabilization of the plasma takes only 15 minutes provided the plasma is re-lit immediately after it is extinguished.

8.7 The nebulizer flow rate used is constant for all aqueous sample extracts and needs not be reset prior to analysis.

8.8 The instrument operating conditions finally selected as being optimum should provide the lowest reliable instrument detection limits (IDL). (See **Section 10** for IDL calculation)

8.9 The calibration curve consists of a blank (S0) and at least 3 calibration standards. The low concentration calibration standard concentration is less than or equal to the method reporting limit (MRL). Sample concentrations less than the lowest standard can only be reported after method modifications have taken place, or with estimated results flagging.

The highest concentration must be within the instrument's linear dynamic range. The standards are run in a sequence from high concentration to low concentration.

8.10 The minimum correlation coefficient for the calibration curve is 0.998. If a correlation coefficient less than 0.998 is obtained, the calibration must be repeated.

8.11 A mid-range ICV (Initial Calibration Verification) Standard from an independent source than the calibration standards) precedes the analysis of the samples. Recovery limits for the ICV are ± 10% of the true value. If the ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. An ICV can be reanalyzed only once prior to corrective action.

8.11.1 A low level ICV (not required to be second source) is run to verify the lower level of calibration . Recovery limits for the low level ICV are + 30% of the true value. The low level ICV is the same concentration as the lowest standard in the initial calibration (S3), which is at the reporting limit. If the low level ICV recoveries do not meet acceptance criteria, corrective action must be taken and/or the calibration must be repeated. A low level ICV can be reanalyzed only once prior to corrective action.

8.12 A CCV (Continuing Calibration Verification Standard) is analyzed after at least every tenth sample and at the end of the sample run. Recovery limits for the CCV are ±10% and in the case of failure for a particular element, all samples following the last acceptable CCV must be reanalyzed for that element. A CCV can be reanalyzed only once prior to corrective action.

8.13 The ICB is analyzed after the ICV and is of the same source as the calibration blank. The CCB (Continuing Calibration Blank) is analyzed after each CCV. Concentrations of any analytes detected must not exceed the MRL or corrective action, such as a single reanalysis and evaluation, must be taken. If the ICB/CCB still fails, reanalysis of all samples since the last valid ICB/CCB for that element is required.

DoD QSM: The acceptable absolute value of the ICB and CCB must be < 2x MDL.

8.14 The ICS (ICSA and ICSAB) standards must be run at the beginning, every 8 hours and at the end of each analytical run.

8.14.1 The ICSA solution, containing the Al, Ca and Mg at 500mg/L, and Fe at 200mg/L, must be run at all wavelengths used for each analyte reported. The analytical results for those target analytes with MRLs ≤ 10ug/L shall fall within ± 2x MRL of the analyte's true value (the true value shall be zero unless otherwise stated). If the results for these analytes fall outside the ± 2x MRL window, check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. For analytes with a MRL > 10 ug/L, the ICSA results shall fall with ± one MRL of the analytes true value (0).

DoD QSM: the absolute value of all non-spiked analytes < 2x MDL

8.14.2 The ICSAB contains Al, Ca, Mg and Fe at the same concentrations as the ICSA as well as all other analytes of interest. Recovery limits for the ICSAB must be within ± 20% of the true value. If the ICSAB recoveries do not meet acceptance criteria, corrective action must be taken. Check that the background correction factors are appropriate, and readjust if necessary. Recalibration may be necessary. If the ICSAB at the end of an analytical sequence fails for a particular element, all samples must be reanalyzed for that element.

8.15 After completion of the initial requirements of this method, samples should be analyzed in the same operational manner used in the calibration routine. A 60 second rinse (rinse solution: 1% HNO_3 and 1.5% HCl) is conducted between all sample solutions, quality control samples, method blanks, and standards.

Analytical Sequence: The following QC protocol should be employed.

1. Standard S0
2. Standard S1
3. Standard S2
4. Standard S3
5. mid-range ICV(second source)
6. low-level ICV (either source)
7. ICB
8. ICSA
9. ICSAB
10. Sample 1
11. *
12. *
13. *
14. *
15. *
16. *
17. Sample 8
18. CCV
19. CCB
20. Sample 9
21. *
22. *
23. *
24. *
25. Sample 16
26. ICSA
27. ICSAB

28. CCV
29. CCB

Note: The ICSA and ICSAB sample count as analytical samples between CCV/CCBs.

Any deviations must be approved by the Inorganic Laboratory Manager or the Operations Manager before they can be implemented.

All analyses are documented in the Instrument Run Log (**Attachment 3**) .

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of mg/L for aqueous samples and mg/Kg dry weight for solid samples. Results are reported to two or three significant figures.

9.2 For dissolved aqueous analytes, report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the reporting limit (RL) unless specifically requested by the client.

9.3 Soil concentrations are calculated using the equation below:

$$\text{Sample Conc. (mg/Kg)} = \frac{C \times V \times Df}{W}$$

Where:
C = Concentration in extract (µg/L)
V = Volume of extract (L, 100mL = 0.1L)
Df = Dilution factor (undiluted =1)
W= Dry weight of sample aliquot extracted (g)

9.4 Recovery Calculations:

The recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)

Where:
SSR = spiked sample result
SR = sample concentration
SA = spike added

9.5 Relative Percent Difference Calculations:

The relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{D1 - D2}{(D1 + D2)/2} \times 100\%$$

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (replicate)

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks - A preparation blank is prepped and analyzed with every batch not to exceed 20 samples. Method blank concentration must be less than or equal to reporting limits unless the sample concentration is at least 20 times greater than the blank concentration or less than reporting limits. Corrective action for method blank contamination involves determining the source of the contamination and re-prepping the affected samples in the batch. The analyst may rerun the method blank once as the first step of corrective action.

DoD QSM -Method blank concentrations must be less than or equal to one-half the reporting limit. For common contamination analytes, method blank concentration must be less than or equal the reporting limit.

10.3 Calibration verification –

10.3.1 A mid-range ICV analyzed immediately after standards must be within ±10% of the true value. The ICV is an independent source standard purchased from a different vendor than the calibration standards.

10.3.1.1 Method 6010C recommends an additional calibration verification using a low-level ICV with a 70-130% criteria, however only the mid-range ICV is required to be from an independent source. In lieu of a second analysis, the response from the S3 standard can be entered back into the curve for verification.

10.3.2 The CCV is analyzed a minimum of every 10 samples in the analysis and at the end of the analytical sequence. If the closing CCV does not meet the criteria, the CCV and all analyses from the opening CCV must be re-analyzed after the problem has been eliminated.

10.3.3 The ICV and CCV analyses are followed by the ICB and CCB analyses, respectively. The ICV and CCV must pass the ±10% criteria or be re-analyzed.

10.4 Use of all standards made from a primary standard must not exceed the primary standard's expiration date.

10.5 Matrix spike (MS) samples - A matrix spike is processed with each batch of samples. Spike recoveries must be within 75-125% of the expected value. If the native sample results exceed 4x the spike added, no further action is needed. Note in narrative. Unless superceded by project requirements, it is not necessary to spike Na, K, Ca, and Mg for waters; or Na, K, Ca, Mg, Fe, Al and Mn for soil.

DoD QSM: Spike recoveries must be within 80-120%. Precision requirements are ≤ 20 % RPD for both aqueous and soil matrices. There is no corrective action for MS recoveries outside the acceptance range other than data qualification.

10.6 Matrix duplicate (DUP) or matrix spike duplicate (MSD) samples (Both options are allowed in method SW6010)- Duplicates are prepped and analyzed with every batch not to exceed 20 samples. Relative Percent Difference (RPD) is calculated for the results of duplicate samples.

10.6.1 A limit of 20% RPD shall be used for sample values greater than 5x the MRL

10.6.2 A control limit of (±) the MRL level must be used if either the sample or the duplicate value is less than 5x MRL.

10.6.3 If one result is above 5x MRL and one below, use the ± MRL criteria.

10.6.4 If both values are below the MRL, no RPD is calculated.

DoD QSM- DUP precision is evaluated for all analytes. Precision requirements are ≤ 20 % RPD for both water and soil. There is no corrective action for MS/MD precision outside the acceptance range other than data qualification.

10.7 Laboratory Control Sample (LCS) - is prepped with a minimum of every 20 samples of the same matrix.

10.7.1 For an aqueous LCS sample, mixed standards are spiked into a beaker of DI water resulting in concentrations approximately ½ the concentration of the high calibration standard and prepped as an aqueous sample. Recoveries must be within the established control limits. The ID of the aqueous LCS sample is LCSW.

10.7.2 For soils, approximately 1g of Teflon Chips (Chemware Ultra-pure PTFE boiling stones, acid washed) used to simulate solid matrix is spiked with standards at the approximate mid-point of the calibration curve. The LCS is then prepped as a soil. The ID for the solid LCS sample is LCSS.

DoD QSM –The acceptance range for both water and soil is 80-120% except silver which has a 75-120% range for soils. When Mitkem in-house LCS limit fall within DoD limit, in-house limits may be reported. If Mitkem in-house limits are wider than DoD limits, the 80-120 % limits must be used.

Laboratory control sample acceptance limits are based on control charts, which are established at Mitkem. These are referred to as in-house limits. Mitkem may also adopt limits from outside agencies when necessary.

10.8 Post digestion spike (PDS) addition - An analyte spike added to a portion of the sample digestate. A PDS is analyzed with each preparation batch of samples (PDS spiking of the same sample as used for matrix spiking is recommended) and is suggested when a new or unusual matrix is encountered within a batch. The acceptance criteria for PDS is 80-120%. The PDS is spiked at the same concentrations as the matrix spike. The PDS concentration should be >10 times the MRL, but < 100 times. If the PDS is not recovered within the specified limits, a matrix effect is confirmed.

DoD QSM: A PDS is required when a new or unusual matrix is encountered within a batch; or when the serial dilution test fails. In addition, a PDS is recommended for projects where all samples are below 50X MDL. The acceptance window is 75-125%.

Corrective action should be taken if the spike concentration <u>is at least 2x</u> the native sample concentration:

(a) If the MS fails for any analyte but the PDS passes, the sample may be redigested and analyzed, except when low MS recovery can be explained as a historically poor performer (example:Antimony (Sb) for soils) in that matrix. In these instances the analyst may want to discuss the outliers with the project manager or department supervisor. When redigestion is warranted, and the MS fails in the redigestate, a matrix issue is confirmed. If the MS passes for the redigestate, the initial results were due to human error during digestion or spiking.

(b) If both the MS and PDS fail, the laboratory will perform a dilution test, or may make a reasonable effort to address the matrix interference by performing the method of standard additions (MSA) or internal standard addition.

10.9 Dilution test (Serial Dilution) – should be performed with each preparation batch. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the MDL), an analysis of a 5X dilution should agree within ±10% of the original determination. If not,

a chemical or physical interference effect is confirmed. A serial dilution should be run whenever the PDS fails, to confirm matrix effect.

DoD QSM: A Serial Dilution is required once per preparation batch or when an unusual sample matrix is present. If the SD fails, a PDS must be run.

As a general rule, Mitkem performs a PDS and SD with each preparation batch of samples.

10.10 Instrument detection limits (IDLs) are established at the time the instrument is set up and every six months thereafter. Ten solutions of diluted acid (1% HNO3 and 1.5% HCl) are analyzed on three non-consecutive days. The IDL for a particular element is three times the average standard deviation of the measured concentration for that element. An IDL is valid in the condition that IDL on both instruments are less than MDL currently reported.

DoD QSM: IDLs are established at the time the instrument is set up and whenever significant changes are made

10.11 Method detection limits (MDLs) may be established once a year. Mandatory MDL determination has been removed from the SW846 6010C method however until regulatory agencies are in agreement, Mitkem will continue to perform MDL studies. The MDL is obtained by multiplying the standard deviation of seven analyses by the appropriate one-sided 99% t-statistic. The value of this statistic equals 3.143 if the number of analyses is seven. The concentration of the analyte in the analyzed solution should be between three to five times the calculated MDL. An MDL verification check is performed immediately following the MDL study. In lieu of an annual MDL study, the MDL verification can be run quarterly on each instrument. The MDL verification check sample is spiked at approximately 1-4 times the current MDL and prepared as if it were a sample.

10.12 Sample Dilutions: When analytes exceed the established linear dynamic range, the sample must be diluted sufficiently to bring those elements into the linear range. The dilution factor (Df) must be integrated into the final concentration calculation.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and sample results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory manager reviews 100% of the data prior to report generation. The QA Director or designee randomly reviews 10% of the data reported by the laboratory. Refer to Section 11 of the QAP for details.

11.2 Electronic files are validated by the analyst and transferred to the report generation group via the LIMS. Once data is in LIMS it is reviewed and validated again by the supervisor or his/her designee. The data are then locked and forms may be generated.

11.3 Reports are generated by the reporting group in conjunction with the metals lab. The data submitted for report preparation is dependent on project requirements.

12. Corrective Action Procedures

12.1 Corrective action to be implemented in the event QC results are outside of the acceptance range are covered in **Section 10**.

12.2 Corrective action reports (CARs) are initiated through the LIMS in the event of an out-of -control situation that cannot be corrected by the analyst. The procedure for initiating a CAR for the purpose of identifying the appropriate corrective action is covered in SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel on the bookcase between the GC/MS and OPrep labs. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan, and have read the Mitkem Contingency Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Always wear safety goggles or a face shield for eye protection when working with acids. If eye or skin contact occurs, flush with large volumes of water.

13.3 Many metal salts are extremely toxic if inhaled or swallowed. Use good housekeeping practices in areas where metal salts are being used and wash hands thoroughly after handling.

13.4 Inductively coupled plasma sources emit radio frequency radiation and intense UV radiation.

13.5 Basic good housekeeping practices such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's Quality Assurance Plan.

15. References

Fassel, V.A. et al. Simultaneous Determination of wear metals in lubricating oils by Inductively-Coupled Plasma Atomic Emission Spectrometry . Anal. Chem. 48: 516-519, 1976.

Patel, B.K.; Raab, G.A.; et al. Report on a single laboratory evaluation of Inductively coupled optical emission Method 6010; EPA contract No. 68-03-3050, December 1984.

U.S. Environmental Protection Agency. I*nductively Coupled Plasma Atomic Emission Spectroscopy* Method 6010C, SW-846 test methods for evaluating solids waste, Revision 3, Final Update IV, February 2007.

U.S. Environmental Protection Agency. Inductively Coupled Plasma-Atomic Emission Spectrometry Method for the Analysis of Waters and solids, EMMC, July 1992.

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Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

Attachments:

1. **Attachment 1**: Analytes of Interest (Table 1 from SW-846 6010C)
2. **Attachment 2:** DoD QC Requirements, Table B-6,D-18, D-19
3. **Attachment 3**: Standard Preparation Logs
4. **Attachment 4**: Instrument Run Logbook

Attachment 1
Analytes of Interest

TABLE 1

RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength[a] (nm)	Estimated IDL[b] (µg/L)
Aluminum	308.215	30
Antimony	206.833	21
Arsenic	193.696	35
Barium	455.403	0.87
Beryllium	313.042	0.18
Boron	249.678 x2	3.8
Cadmium	226.502	2.3
Calcium	317.933	6.7
Chromium	267.716	4.7
Cobalt	228.616	4.7
Copper	324.754	3.6
Iron	259.940	4.1
Lead	220.353	28
Lithium	670.784	2.8
Magnesium	279.079	20
Manganese	257.610	0.93
Mercury	194.227 x2	17
Molybdenum	202.030	5.3
Nickel	231.604 x2	10
Phosphorus	213.618	51
Potassium	766.491	See note c
Selenium	196.026	50
Silica (SiO_2)	251.611	17
Silver	328.068	4.7
Sodium	588.995	19
Strontium	407.771	0.28
Thallium	190.864	27
Tin	189.980 x2	17
Titanium	334.941	5.0
Vanadium	292.402	5.0
Zinc	213.856 x2	1.2

TABLE 1
(continued)

[a] The wavelengths listed (where x2 indicates second order) are recommended because of their sensitivity. Other wavelengths may be substituted (e.g., in the case of an interference) if they provide the needed sensitivity and are treated with the same corrective techniques for spectral interference.

[b] The estimated instrumental detection limits shown are provided for illustrative purposes only. Each laboratory must determine IDLs and MDLs, as necessary, for their specific application of the method. These IDLs represent radial plasma data and axial plasma IDLs may be lower.

[c] Highly dependent on operating conditions and plasma position.

Attachment 2:
DoD QC Requirements, Table B-6,D-18, D-19

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	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).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18).	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA	

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) (ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$ CVAA: $r \geq 0.995$	Correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within ± 10% of expected value GFAA: within ± 20% of expected value CVAA: within ± 20% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch	No analytes detected > ½ RL For common laboratory contaminants, no analytes detected > RL	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test Serial dilution	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	ICP: Perform post-digestion spike (PDS) addition GFAA: Perform recovery test CVAA: Perform matrix spike	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	The spike addition should produce a level between 10 - 100 x MDL.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x MDL	Recovery within 85-115% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard additions (MSA) or internal standard calibration	When matrix interference is suspected	NA	NA	NA	Document use of MSA in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	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 J-flag 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.
MSD or sample duplicate	One per preparatory batch per matrix	RPD ≤ 20% (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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7470 WATER MATRIX[28]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

[28] The as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7471 SOLID MATRIX[29]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

501L

[29] Some as-generated limits have been adjusted to reflect method requirements and acceptable calibration uncertainty. A number of sporadic marginal exceedances of the control limits are allowed for method 6010, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits.

Attachment 3:
Standard Preparation Logs

MITKEM LABORATORIES **METALS PRIMARY STD RECEIPT LOGBOOK:INSTRUMENT LAB**

DATE REC	PRIMARY STD ID	VENDOR	CATALOG#	LOT #	ANALYTE(S)	REC BY	DATE OPENED	EXPIR. DATE	MITKEM ID

100.0038-01/08

Page: _____

MITKEM LABORATORIES, A Division of Spectrum Analytical Inc.

METALS INTERMEDIATE STANDARD LOGBOOK

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Intermediate STD ID	Analyst	Exp. Date

Logbook ID: 100.0041 -01/08

Reviewed By: _____

MITKEM LABORATORIES **METALS WORKING STANDARD LOGBOOK**

Prep Date	Analyte(s)	Primary/Intermediate STD ID	Initial Conc.(mg/L)	Amt. Added (ml)	Final Volume (ml)	Final Conc.(mg/L)	Diluent Used	Working STD ID	Analyst	Exp. Date

Logbook ID: 100.0036 -12/07

Reviewed By: _____

Attachment 4:
Instrument Run Logbook

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/4300DV**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 100.0129-05/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

MITKEM LABORATORIES **SAMPLE RUN LOG: ICAP/3100XL**

Date: Analyst:

Spray Chamber: Minicone

POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID		POS	Lab ID	
1			21			41			61			81		
2			22			42			62			82		
3			23			43			63			83		
4			24			44			64			84		
5			25			45			65			85		
6			26			46			66			86		
7			27			47			67			87		
8			28			48			68			88		
9			29			49			69			89		
10			30			50			70			90		
11			31			51			71			91		
12			32			52			72			92		
13			33			53			73			93		
14			34			54			74			94		
15			35			55			75			95		
16			36			56			76			96		
17			37			57			77			97		
18			38			58			78			98		
19			39			59			79			99		
20			40			60			80			100		
												101		
												102		
												103		

Comments:

Lot #s

HNO3: _____

HCL: _____

CRI: _____

Logbook 00.0126-04/08

STANDARD: _____

ICV/CCV: _____

ICSA/ICSAB: _____

Reviewed by: _____

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

for

Handling Evidentiary Materials

SOP No. 110.0026
Rev. 6

	Signature	Date
QA Officer:	Sharon Lauk	2/23/06
Lab Director:	William D.	2/23/06

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

for

Handling Evidentiary Materials

SOP 110.0026
Rev. 6

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the procedures for handling all evidentiary materials associated with a sample delivery group, including air bills, SMO or client traffic report and chain-of-custody forms.

2. Personnel Qualifications and Responsibilities

All employees handling client information should have read and understood this SOP. All information in this SOP must be followed.

3. Summary of Procedure

All paperwork pertaining to the sample delivery group(s) of a case or project must be kept confidential.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Not applicable.

7. Reagents

Not applicable.

8. Procedure

All documents received with a sample delivery group (SDG) and/or generated in the course of the analyses of these samples must be kept confidential. Standard Business Confidentiality practices shall apply.

7.1 Documents specifically marked "Confidential" which may accompany an SDG, must be treated separately from other case-related documents.

7.2 Procedures for handling documents marked "Confidential"

7.2.1 Contact Sample Management Office (SMO) for EPA cases or the client to assure that receipt of these documents is correct and necessary for analysis of the samples. If not required for analysis, return the documents following SMO or client instructions.

7.2.2 If the documents are necessary for sample analysis, place them in a secure file separate from the regular files and under control of the Document Control Officer (DCO) or his/her designee. The DCO will keep this file of confidential documents secure at all times and only allow authorized personnel access on an as needed basis.

7.2.3 Receipt of confidential documentation, its use, duplication and ultimate disposition must be documented in a Confidential Document Log. Duplication of confidential documents must be kept to a minimum and done with concurrence of the Project Officer or his/her designee.

7.2.4 The DCO shall remove and retain for one year the cover sheet of any confidential material disposed of and note the disposition in the Confidential Document Log. Disposal of confidential documents should only be done at the direction of the EPA or client Project Manager and/or the EPA Contracting Officer.

8.4 Test results or data transmitted electronically or by electromagnetic means will be sent in such a way as to preserve client confidentiality. Confidentiality statements are included on Fax forms and e-mail transmissions. Confirmation of client address and/or individual personnel is required prior to transmission.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

Not applicable.

11. Data Validation and Reporting

Not applicable.

12. Corrective Action Procedures

Not applicable.

13. Health and Safety

Not applicable.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Not applicable.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

For

Sample Data Control for Inorganic CLP (ILM/ISM)

SOP No. 110.0034
Rev 1

	Signature	Date
QA Director:		3/13/09
Lab Director:		3/16/09

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MITKEM LABORATORIES,

A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Data Control for Inorganic CLP (ILM/ISM)
Rev 1

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the data management procedures used in support of report generation for CLP samples using the protocols described in ILM04.1, ILM05.4 and ISM01.1.

2. Personnel Qualifications and Responsibilities

Personnel responsible for inorganic data management must be familiar with the protocols outlined in this SOP and are required to follow these procedures.

3. Summary of Procedure

CLP forms 1 – 14 are generated from raw instrument data using the protocols outlined in the CLP Statement of Work (SOWs ILM04.1, ILM05.4 and ISM01.1) and **section 8** of this SOP, in preparation for use in the final data package.

4. Sample Preservation, Containers, Handling, and Storage

NA

5. Interferences and Potential Problems

NA

6. Equipment and Apparatus

6.1 Intel Based Workstation, operating in a Microsoft Windows 05.

6.2 Omega LIMS ILM CLP module (includes ILM, ISM, SW-846 and MCAWW report options).

7. Reagents

NA

8. Procedure

8.1 ThermoFisher PlasmaLab software is used for data acquisition of ICP-MS analyses. Perkin-Elmer WinLab Software is used for data acquisition of all ICP-AES analyses. Once a run has been completed, the data file associated with the run is assigned a filename that describes the instrument, date of run, and the daily run sequence (e.g. A091402 denotes the second data file generated by the Optima 3100 XL (ICP 2) on 9/14). This (.csv) file is loaded onto the file server Bernoulli. Path = \\ Bernoulli \\inorganic\CLPfiles\.

8.2 Subsequent file translation and data processing is done using the Omega ILM Module, Mitkem owns a site license. Currently a copy of the software is kept on three workstations. The workstations are connected to the file server via the Local Area Network (LAN). The servers are located in a secure area accessible by key code entry only to authorized personnel. Details about data storage and backup can be found in Mitkem SOP# 110.0029, Electronic Data Management.

8.3 Once all inorganic data for a Sample Delivery Group (SDG) are generated, a folder containing instrument printouts of all raw data associated with the SDG are delivered to a data report analyst for processing. ICP, Mercury, and Cyanide runs are captured and saved for direct upload by the report generation software.

8.4 The first step in generating a final report begins with the uploading of all runs into Omega LIMS. Before doing this the runs need to be uploaded into Omega Upload Tool so that they can be edited for any problems associated with the run, such as typographical errors, or the "z-ing" out of unwanted data points.

8.5 Enter the runs into Omega LIMS. This is done by opening the Omega LIMS program. At the main menu screen click on the DATA ENTRY button. this will bring up a new screen called Analytical Runs. In this screen enter in new runs, and update or revise any previous analytical runs.

8.6 To enter analytical runs press the Add key in the top tool bar so that there will be a blank screen. Enter the Instrument ID, Run Start Date, Analyst, and any comments. In the comments space enter any information that is important to the run and who uploaded it.

8.7 Once this information is entered press the Data Import button to bring the runs into LIMS. Once the run is uploaded it can be further edited for items such as ID's, turning off analysts that are not reported with the SDG, etc. Then the sequence can be calculated to get the final results.

8.8 The next step is to have the runs that were uploaded into Omega LIMS QA validated by an analyst who has QA authority. ***All runs that are entered into Omega LIMS have to be QA validated before entering them into <u>any</u> CLP module.***

8.9 After the runs have been QA'd by a senior analyst they can be uploaded into the ILM CLP module.

8.10 Open the module and enter an SDG number. Press return after the software has completed some querying.

8.11 Select the protocol that will be used <u>from the protocol drop down list</u>. If the protocol is SW6010<u> or 6020</u> select SW846. If the protocol is <u>CLP</u>, select ILM04.1, ILM05.<u>4 or ISM01.1 as appropriate</u>. <u>If the protocol is E200.7 or E200.8, select MCAWW. This same logic should be applied to the cyanide and mercury fractions as well.</u>

8.12 The next step for ILM04.1 SDGs is to select the IDL set that will be used. To do this left click in the blank IDL Key and a dropdown list will appear. Select the instruments that were used. Once the instruments for the IDLs have been selected, edit the IDLs to the analytes that are associated with the SDG that is being generated.

8.13 Click the "Get LIMS Data" button. This will do many calculations and put all the data on the correct CLP forms.

8.14 Now go to the "Review Data " tab and click on "Update Forms" This will update all the forms that were generated. In this screen the runs can be edited further by deleting the samples that are not needed in the SDG that is being worked on. The analytes that are not needed for the QC samples can be turned off. After all the runs have been edited click the "Update Forms" button again. This icon should be clicked after edits to the run have been made.

8.15 Now go to the tab "Report Options". This lists all the CLP forms that are generated through the module. Click on the icon "Edit Cover Letter" this will update the SDG cover page. Go through all of the forms to make sure that they look right and all the concentrations and percent recoveries appear to meet criteria. To view the forms click in the box next to the form that you want to view then press the "Preview Report". Form 11 ICP Interelement Correction should not be opened with the "Preview Report" icon. The reason for not opening this form is because it will not display properly so just go to "Generate Report" to print it out. After reviewing all the forms go back to the "SETUP: IDL and ICF" tab to import the IEC's. Click on the "Import from Excel" icon this will bring you to a directory where you can navigate to where IEC files are stored. The file is called "zzzzz6010_ICF". Next open the IEC table by clicking on "View Export" button and edit the list by choosing the instruments that are being used. The IEC's can also be uploaded after choosing the IDL's.

8.16 All ILM/<u>ISM</u> SDG's Form 1's need to have the color, clarity, and comments added to then you click on the button "Forms 1 Color, Clarity and Comments".

8.17 Now the report can be generated. Click on the button "Select All". This checks off all the forms that will be printed. If you do not need a form just click off the checkmark then press the "Generate Report" button. This will print out the entire SDG.

8.18 Initial Report Assembly:

Once all data forms have been generated, the entire data package is assembled for final review and contains all the required supporting forms including: all raw instrument data runs, copies of each preparatory log sheets, percent moisture log sheet (if required), a current revision of the laboratory work order that documents the EPA sample names, analyses required and other pertinent SDG related information, appropriate separator pages, and the "CLP/CLP-Like delivery checklist for inorganic analyses"(**Figure 1**). All deliverables shall be presented to the EPA as described in **Exhibit B, Section 2** of the ISM01.1 SOW.

8.19 Final Data Report Review:

Upon receipt of the data package and other supporting documents required in the complete SDG file (CSF), a designated data analyst reviews the final report. This review ensures that all required QA/QC operations as specified in **Exhibit D** of the ISM01.1 SOW have been followed. Errors in the format or content of the final data report are noted and the report is returned to the analyst who originally processed the data for correction.

8.20 Package Assembly:

When the Review/Corrections to the data report have been completed, and approved, the package with all the supporting documentation is delivered to Data Reporting for assembly into the final CSF with the reports (if required) from the organic laboratories. ISM reports also require the DC-2 forms (**Figure 2**) as outlined in **Exhibit B, Section 3.6** of the ISM01.1 SOW.

8.21 Software and Hardware systems are maintained on a daily basis. The network administrator, in addition to maintaining security and the integrity of all data on the main servers, also assists data technicians in the maintenance of each Windows based workstation, as outlined in **Exhibit F, Section 2.6** of the ISM01.1 SOW.

8.22 Software Updates:

Omega LIMS periodically updates the software. IT will also make periodic updates and therefore prior to using any module you should update it from the main menu.

9. Data Reduction and Calculations

NA

10. Quality Assurance/Quality Control

NA

11. Data Validation and Reporting

See **Section 8** of this SOP.

12. Corrective Action Procedures

All out-of-control situations must be corrected or qualified prior to submission of the final report. Any initial deviations should be noted in the prep/analysis notes section of the "CLP/CLP-Like Deliverable Checklist for Inorganic Analysis" so that corrections may be made or the report is returned to the laboratory for final correction.

13. Health and Safety

All employees must read and be familiar with the Mitkem Health and Safety Plan, and are required to comply with the safety practices described therein.

14. Pollution Prevention, Waste Management, and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. The Mitkem Quality Assurance Plan
2. US EPA CLP Statement of Work (SOW), for Inorganics Analysis, Multi-Media/Multi-Concentration, ILM04.1.
3. US EPA CLP Statement of Work (SOW), for Inorganics Analysis, Multi-Media/Multi-Concentration, ILM05.4.
4. US EPA CLP Statement of Work (SOW), for Inorganics Superfund Methods, ISM01.1.

Attachments:

1. **Figure 1**: CLP/CLP-Like Deliverable Checklist for Inorganic Analysis.
2. **Figure 2:** ISM01.1 DC-2 Forms

Figure 1

CLP/CLP-Like Deliverable Checklist for Inorganic Analysis

MITKEM LABORATORIES

CLP/CLP-like Deliverable Check List for Inorganic Analysis

Project Number:_____
Client._____
Input by/date:_____
Forms generated on/date:_____

Analysis:_____
Category:_____
Reviewer:_____
(1) Date Reviewed:_____
(2) Date Reviewed:_____
Corrections by:_____

Elements Required:

Al	Sb	As	Ba	Be	Cd	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Ni	K	Se	Ag	Na	Tl	V	Zn	Sn	CN	Hg

Items:	Pages	Check	OK/Unusual Observation
Sample Log-In Sheet			
Prep Log Sheet (AQ/SL)			
% Solid Bench Sheet			
Tumbling Log (TCLP/SPLP)			

	Check	Lab ID	OK/Unusual Observation/Deviation/Flags
ICV / CCV			
Matrix Spike (N)			
Duplicate Samples (*)			
Serial Dilutions (E)			
Post Digestion Spike			
LCS			
ICP Interference			
CRA / CRI			

Prep/Analysis Notes:

	Yes	No
Client ID Check:		
ID Truncation:		
Special Request:		

Figure 2

ISM01.1 DC-2 Forms

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LABORATORY NAME _____

CITY/STATE _____

CASE NO. _____ SDG NO. _____

SDG NOs. TO FOLLOW _____ _____ _____

MOD. REF. NO. _____

CONTRACT NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.6)

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
1.	Inventory Sheet (DC-2)				
2.	SDG Narrative				
3.	Sample Log-In Sheet (DC-1)				
4.	Traffic Report/Chain of Custody Record(s)				
5.	Cover Page				
Inorganic Analysis					
6.	Data Sheet (Form I-IN)				
7.	Initial & Continuing Calibration Verification (Form IIA-IN)				
8.	Blanks (Form III-IN)				
9.	ICP-AES Interference Check Sample (Form IVA-IN)				
10.	ICP-MS Interference Check Sample (Form IVB-IN)				
11.	Matrix Spike Sample Recovery (Form VA-IN)				
12.	Post-Digestion Spike Sample Recovery (Form VB-IN)				
13.	Duplicates (Form VI-IN)				
14.	Laboratory Control Sample (Form VII-IN)				
15.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)				
16.	Method Detection Limit (Annually) (Form IX-IN)				
17.	ICP-AES Interelement Correction Factors (Annually) (Form XA-IN)				
18.	ICP-AES Interelement Correction Factors (Annually) (Form XB-IN)				
19.	Internal Standard Association (Form XI-IN)				
20.	Preparation Log (Form XII-IN)				
21.	Analysis Run Log (Form XIII-IN)				
22.	ICP-MS Tune (Form XIV-IN)				

FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

	PAGE NOs.		CHECK	
	FROM	TO	LAB	REGION
23. ICP-MS Internal Standards Relative Intensity Summary (Form XV-IN)				
24. Initial Calibration (Form XVI-IN)				
25. ICP-AES Raw Data				
26. ICP-MS Raw Data				
27. Mercury Raw Data				
28. Cyanide Raw Data				
29. Preparation Logs Raw Data				
30. Percent Solids Determination Log				
31. USEPA Shipping/Receiving Documents				
Airbill (No. of Shipments_____)				
Sample Tags				
Sample Log-In Sheet (Lab)				
32. Misc. Shipping/Receiving Records (list all individual records) Communication Logs				
_____				
_____				
33. Internal Lab Sample Transfer Records & Tracking Sheets (describe or list)				
_____				
_____				
34. Internal Original Sample Prep & Analysis Records (describe or list)				
Prep Records _____				
Analysis Records _____				
PE Instructions _____				
Description _____				
35. Other Records (describe or list)				
Communication Logs				
_____				
_____				

36. Comments:

_____

_____

_____

Completed by:
(CLP Lab) _____ _____ _____
(Signature) (Print Name & Title) (Date)

Audited by:
(USEPA) _____ _____ _____
(Signature) (Print Name & Title) (Date)

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

For

Sample Data Control for CLP (SOM01.2)

SOP No. 110.0035

Rev. 1

	Signature	Date
QA Director:		4/8/08
Lab Director:		4/9/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Sample Data Control for CLP (SOM01.2)

SOP 110.0035 Rev. 1

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the data management procedures used in support of forms generation for CLP samples using the protocols in SOM01.2.

2. Personnel Qualifications and Responsibilities

The personnel responsible for the data management must be familiar with the protocols outlined in this SOP and follow these procedures.

3. Summary of Procedure

The CLP forms are generated using the **instructions, procedures and/or** protocols outlined in the **USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organic Analysis Multi-Media, Multi-Concentration SOM01.2 (April 2007)** and **section 8** of this SOP.

4. Sample Preservation, Containers, Handling, and Storage

N/A

5. Interferences and Potential Problems

N/A

6. Equipment and Apparatus

6.1 Intel based workstation, operating in a Windows OS.

6.2 Target Software from ThruPut.

7. Reagents

N/A

8. Procedure

HP EnviroQuant Software is used for data acquisition for all of the organic CLP analyses performed in the laboratory. The software is resident in each of the PCs (as part of a company wide network) that are password accessible Analysts are assigned different security levels depending on the job requirement.

8.1 Upon completion of each data acquisition, the raw data are copied directly to the primary file server. The data are then evaluated using Target. The Target reviewed data will then be uploaded into LIMS. The data in LIMS will need to be QA'd for forms generation (**Forms I through VIII for volatiles and semivolatile, Forms I through X for pesticides and Aroclors**) and supporting documents including chromatograms, quantitation reports, **tentatively identified compounds (TIC)** library search and spectra generation.

8.2 The laboratory owns several copies of the data processing software Target that is installed on individual workstations. The workstations are connected to a file server via the company's local area network (LAN). For more details, see SOP No. 110.0029, Electronic Data Management.

8.3 Target data and methods reside on a system with name "Organics". The system is configured as follows:

8.3.1 Volatiles analysis:

Organic/VOA/Vi/Batch/File, where i = 1,2,5,or 6.

8.3.2 Semivolatile Analysis:

Organic/SVOA/Sj/Batch/File, where j = 1,2,3 or 4

8.3.3 Pesticide and/or Aroclor Analysis:

Organic/SVOA/Ek/Batch/File, where k = 1,2, 3, 4 or 5.

Where Vi is the instrument that is used in the volatile lab; Sj is the instrument that is used in the semivolatile lab; and Ek is the instrument that is used in the Pesticide/PCB lab.
Batch is the batch of runs that are generated within the analytical batch. This is typically the date the mass spectrometric analysis is acquired. For pesticides/PCB, the batch is the date the analytical sequence was initiated.

Under the subdirectory **File** are the raw data files that are uploaded from the Hewlett Packard EnviroQuant Software. This subdirectory also contains the various method files that are used to process data including tunes, initial calibration, continuing calibration and sample analysis for mass spectral based analysis, and initial calibration, calibration verification and sample analysis for GC based analysis. These method files are as follows:

8.3.4 Volatiles (Trace and Low/Medium)Analysis:

Vi_BFB_SOM.m for evaluating BFB tunes for Vi.
Vi_TVOA_1.2.m for evaluating analysis of blanks and samples by trace volatiles or Vi_SOM1.2_LS for low/medium volatiles, where i = 1,2,5, or 6.

8.3.5 Semivolatile Analysis:

Sj_dftppSOM.m for evaluating DFTPP tunes for Sj.
Sj_SOM.m for evaluating analysis of blanks and samples, where j = 1,2, or 3.

8.3.6 Pesticide/PCB Analysis:

Ek_somPEST-F.m for evaluating sample analysis (including all standards) for Ek front column;Ek_somPEST-R.m for evaluating sample analysis (including all standards) for the rear column, where k = 1,2, 3, 4, or 5.

Once the data are processed, the analyst will evaluate the data to ascertain that all of the peaks of interest are properly integrated. If needed, manual integration will be performed to improve data quality. The analyst who performs the manual integration will initialize and date all such changes. In addition, hardcopy of the changes are generated as part of the audit trail. Most of the time, hardcopies of the re-integrations are self-evident and the analyst does not need to further justify the re-integrations. The lab supervisor or his/her designee will review and sign off the changes. See Mitkem SOP # 110.0008.

8.4 Procedures for Setting up Mass Spectral Based Analysis:

8.4.1 Mass spectral based analyses including the volatile and semivolatile organic analyses are similar and will be discussed together.

8.4.2 As discussed earlier, the various mass spectral processing methods are provided by the vendor and modified by the laboratory. On the day the samples are **to be** analyzed, the method file is created by going into Target/Edit/Copy to copy the methods from the previous batch of samples that were processed using the SOM01.2 approach.

8.4.3 **Make sure that both the tune and sample analysis methods are copied.** After the methods are copied, rename the methods to the date the actual batch of data are to be processed.

8.4.4 Use Target/Process/Select Method to select the tune method.
Use Target/Process/Process to process the tune analysis.
Use Target/Reports/Review Reports/Target Graphics to evaluate the tune analysis Whenever the ion abundance is outside the required criteria, it will be flagged with a qualifier "*". The analyst should re-analyze the tune sample. If the failure to meet tune criteria persists, the analyst should identify and resolve the problem by following the procedures outlined in the analytical SOP.

8.4.5 After the tune analysis passes criteria, proceed to the calibration standard analysis. Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the initial or continuing calibration standard(s). Use Target/Review/Target Review to evaluate the target analytes in the analysis for the initial or continuing calibration; use manual edition if needed.

8.4.6 For initial calibration, use Target/Process/Edit to open the method-editing window. Use View/Initial Calibration to evaluate the RRF and %RSD for each target analyte. An arrow qualifier will flag the target analyte(s) that does not meet criteria. Only up to two compounds may be flagged, and these compounds must have a RRF no less than 0.01 and a %RSD not greater than 40.0%. Corrections as noted in the analytical SOP should be taken to meet these criteria by re-analyzing one or more levels of the initial calibration.

8.4.7 For the continuing calibration, use Target/Reports/Review Reports/Target Sample to evaluate the RRF and %D for each target analyte. An arrow qualifier will flag the target analyte(s) that does not meet criteria. Only up to two compounds may be flagged, and these compounds must have a RRF no less than 0.01 and %D not greater than 40.0%. Corrections as noted in the analytical SOP should be taken to meet these criteria by re-analyzing the calibration standard. Once the daily calibration is processed, the RRF will automatically be updated in the method file. The same method file is then used to process the method blank, LCS, and samples run within the batch.

8.4.8 Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the method blank, LCS, and all samples. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and all samples; use manual edition if needed. Use Target/Reports/Review Reports/ISTD Monitor to evaluate the internal standard recovery. Use Target/Reports/Review Reports/Recovery Report to evaluate the recoveries for the system monitoring or surrogate compounds. For the LCS, the recoveries for the spiked compounds are listed as well. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and

samples. Some corrections as noted in the analytical SOP may be needed if the QC samples do not meet criteria.

8.4.9 After all the samples within the SDG, which may include several batches of samples, have been processed and reviewed, use Target/Reports/QuickForms to generate the required forms. The analyst should include all the files, which include all samples, tune, initial calibration, continuing calibration, method blank, LCS, and other necessary QC samples for the SDG using Add and Remove buttons.

8.4.10 Under the QuickForms Host: enter the case number and lab contract number into the appropriate boxes. Use Edit/Forms Control and then select SOM under Form Set. Make sure Fraction Type is set as VOA for volatile analysis or SV for semivolatile analysis; the analyst has the option of generation of all the required forms (**Forms I through VIII**) at once or one at a time. Use Edit/Edit SDG to evaluate all the files in the SDG. All the samples analyzed in the same batch will be organized into one QC group with their associated tune, initial calibration standard or continuing standards, and QC samples. Use Add Sample, Delete Sample, Delete QC Group, and New QC Group to rearrange samples or QC groups if needed. Use Edit/Blank Test to associate sample contamination with the corresponding method blanks. This step must be performed before producing any Form **I** to ensure that the compound has been flagged with a "B" qualifier if the same compound is also detected in the associated method blank. Use Generate/Generate Reports to produce all the form as selected. Use Generate/Review Reports to evaluate the forms.

8.5 Procedures for Setting Up Pesticides and/or Aroclor Analysis:

8.5.1 As discussed earlier, the Pesticides/PCB processing methods are provided by the vendor and modified by the laboratory. On the day the samples are to be analyzed, the method file is created by going into Target/Edit/Copy to copy the methods from the previous batch of samples that were processed using the SOM01.2 approach. **Make sure that both methods for the front and rear columns are copied.** After the methods are copied, rename the methods to the date the actual batch of data are to be processed.

8.5.2 Only the front column is discussed below, since the procedures are the same for the rear column.

8.5.3 Use Target/Process/Select Method to select the front column method. Use Target/Process/Process to process the initial calibration in the same order as the standards are analyzed, i.e. starting with the resolution check and ending with the INDABH. Use Target/Review/Target Review to evaluate the standard analyses to make sure they conform to method specifications as discussed in the Pesticide Analysis SOP. Some corrections, such as re-analysis of some of the levels of the calibration standards, may be needed to complete the initial calibration, as the method requires. Use manual edition if needed. Use Target/Process/Edit to open the method-

editing window, then use View/Initial Calibration to evaluate the %RSD for each target analyte in the initial calibration.

8.5.4 Once the initial calibration is processed, the CF will automatically be updated in the method file. The same method file is then used to process the method blanks, LCS, continuing calibration, and all samples run within the batch. Use Target/Process/Select Method to select the sample analysis method. Use Target/Process/Process to process the method blank, LCS, and all samples. Use Target/Review/Target Review to evaluate the target analytes in the analyses for the method blank, LCS, and all the samples; use manual edition if needed. Use Target/Reports/Review Reports/Target Sample to evaluate the CF and %D for each target analyte for the continuing calibration standard. Use Target/Reports/Review Reports/Recovery Report to evaluate the recoveries for the system monitoring compounds. For the LCS, the recoveries for the spiked compounds are listed as well.

8.5.5 After all of the samples within the SDG, which may include several batches of samples, have been processed and reviewed, use Target/Reports/Quick Forms to generate the required forms. The analyst should select all the files, that includes all samples, Resolution Check, PEM, instrument blank, initial calibration, continuing calibration, method blank, LCS, and other necessary QC samples for the SDG using Add and Remove buttons.

8.5.6 To generate Organics CLP forms, refer to the 'Organics Forms Reporting' document found in General Mitkem Omega Information on the Mitkem switchboard (http://switchboard.mitkem/docs.asp).

8.6 Data Package Assembly:

8.6.1 Once all of the required forms are generated; also generate all of the supporting documents. These include chromatograms and quantitation reports for the tune, initial calibration, continuing calibration, samples, blanks, LCS, TIC library search and spectra of positive hits for the mass spectral based analysis. For pesticides analysis, these include chromatograms, manual integration hard copies and quantitation reports for all initial calibration, calibration verification, florisil check, samples, blanks, LCS. All the information and documentation is forwarded to the document control officer with the following:

a. Copy of the extraction prep log.
b. Tracking log from sample receipt
c. Extraction transfer log.
d. Instrument run log.

The document control officer will assemble the data package per the SOW instructions.

8.7 Software Updates:

8.7.1 ThruPut is no longer updating the Target Software.

8.7.2 At Mitkem, the software and hardware systems are maintained on a daily basis. The laboratory has a full-time network specialist. This person is also responsible for monitoring the automatic copy/backup of all data each night.

8.7.3 At Mitkem, data base integrity (e.g. quantitation list, initial calibration and continuing calibration updates) is maintained by the analyst and his/her lab supervisor. Changes, if made, are reviewed by another analyst and/or supervisor.

9. Data Reduction and Calculations

N/A

10. Quality Assurance/Quality Control

N/A

11. Data Validation and Reporting

N/A

12. Corrective Action Procedures

All out of control situations must be recorded in the corrective action logbook and documented returned to control as described in SOP No. 80.0007.

13. Health and Safety

All employees should read and be familiar with Mitkem's Safety Manual and Chemical Hygiene Plan, and comply with the safety practices described therein.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories.

USEPA. Statement of Work, Method SOM01.2.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

SOP No. 110.0038

Rev. 7

	Signature	Date
QA Director:		2/19/09
Lab Director:		2/19/09

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Percent Solids Determination

As Required for Various SW846 and EPA Methods

Rev. 7

1. Scope and Application

This Standard Operating Procedure (SOP) describes the procedure for measuring the percent solids/percent moisture of many types of solid material. An undried portion of sample is weighed and dried at 105 ° C. The sample is cooled, reweighed and the percent of water evaporated provides the result.

Note: It may be necessary for some specifically identified samples to undergo a pre-drying process. See amendment.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Analysts and technicians are responsible for performing their work tasks following the procedures outlined in the SOP. Supervisors and managers are responsible for reviewing and approving new and revised SOPs. They are responsible for ensuring that SOPs are accurate and up to date, and are being implemented appropriately.

3. Summary of Procedure

Any standing water in the sample container is decanted. Samples are mixed to ensure sample homogeneity. Percent solid is calculated by the evaporation of water in a solid sample to a constant weight in a 103-105 ° C oven. .

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are stored prior to percent solids determination at a temperature between 2-6° C.

4.2 There is no "official" sample holding time. This analysis should be performed as soon as possible after sample receipt.

5. Interferences and Potential Problems

5.1 Temperature fluctuations will cause measurement errors.

5.2 Balance fluctuations can cause measurement errors. Also, if the balance is not properly calibrated, errors can occur.

5.3 Negligence in taring the balance prior to recording weights will also cause errors.

5.4 Results of less than 50% solids for ISM01.1 soils from EPA require immediate SMO notification and guidance.

5.5 If a SOM01.2 soil sample has greater than 65% moisture, the laboratory may use up to 50 g of soil sample in order to achieve the expected CRQLs. The amount of sample used and the % moisture should be noted in the SDG Narrative

6. Equipment and Apparatus

6.1 Aluminum weighing pans.

6.2 Top Loading balance, capable of reading to two decimal places, connected to a PC loaded with the Balance Talk software program.

6.3 PMOIST Binder to store photocopies of the Balance Talk worksheets

6.4 Scoopulas for portioning the sample.

6.5 Metal pans for moving samples in and out of the oven.

6.6 Class "S" or Class "2" weights for balance calibration.

6.7 Drying oven capable of maintaining 103-105 ° C (ISM01.1 allows 105 ± 5° C).

6.8 Class "1" weights for monthly calibration/verification .

6.9 Dessicator.

7. Reagents

Not applicable.

8. Procedure

Sample Prep: (***It maybe necessary to "pre-dry" some samples. See amendment, sec. 16.)***

8.1 Run the PMOIST backlong in LIMS by due date. Select the samples for PMOIST.

8.2 In LIMS, go to the Data Entry page. In the data entry page, click on **Export to Excel**. In the **Test Code** field, select PMOIST.

8.3 Remove the check marks from the **MBLK** and **LCS,** and select the **Excel** button. **SAVE** the file as a temporary excel file and select the **OPEN** button. When prompted for confirmation, select the **YES** button. The temporary excel file should be named *aaayymmddx*: where aaa are the initials of the person performing the PMOIST, yy is the last two digits of the year, mm is the month, dd is the day and x is A, B, C, etc., which is the sequence number. For example, the second temporary spreadsheet on January 14, 2009 would be called arh090114B.

8.4 In the temporary spreadsheet, delete any samples in which PMOIST will not be performed yet.

8.5 Add any sample DUPs now. Sample DUPs are performed every 20 samples. To add a sample DUP, highlight the row with the sample that a duplicate analysis is to be performed upon. Copy the highlighted row and select Insert Copied Cells option. Add the suffix DUP to the end of the ID for the sample that was just copied.. Be sure to save the changes made.

8.5.1 Duplicate samples for ILM05.4 projects must be done on the same sample that is chosen for Matrix Spike. The Duplicate analysis result is actually reported on Form 6 of the data package.

8.6 Label the aluminum weighing dishes with the Mitkem sample ID .

8.7 Minimize the Omega screen and the saved temporary spreadsheet. On the desktop, double click on the My Network Places icon. Double click on Public in Avogadro, Lims and then Pmoist. Scroll down and select file newestPmoist. If a macro warning box is displayed, choose the button **Enable Macros**.

8.8 Using the **Save As** option in the File Menu save the opened workbook using the following notation: YY =last two digits of the year; MM = numeric month example (June = 06, July = 07) DD = numeric value of the date of the month, X = letter corresponding to sequence number for the day. Do not forget to save this file to the Avogadro/ Public/Lims/**PMoist** directory.

8.9 Move to the **Header Information** page. Select the **Clear Data** button to remove any old data remaining from a previous batch. Complete the **Date and Time In/Out** and **Temp**erature fields, and save to the new **PMoist** workbook.

8.10 Copy Mitkem Sample IDs from the temporary Excel spreadsheet starting with cell A2 to D2 down to Ax and Dx, where x is the number of the last row of samples.

8.11 Paste Mitkem Sample IDs into the Sample field starting in cell (A2) of the Data Entry page of the new **PMoist** spreadsheet. The temporary spreadsheet should be closed at this point.

8.12 The balance calibration must be checked and recorded in the appropriate logbook. Use calibration class "S" check weights of 1.0, 10.0 and 100 grams. If the balance does not meet the calibration check criteria listed in the logbook, do not proceed, but inform the supervisor.

8.13 In the spreadsheet move the cursor to cell H2 (first cell under Tare Weight column). Begin to collect the weight of the weighing tin: make sure the balance is zeroed. Place a weighing tin on the balance. When the balance displays the "*" press the ''print'' button on the balance. The tare weight is then printed in the highlighted cell of the spreadsheet. The cursor automatically goes to the next cell (Tare + Sample).

8.14 Open the sample jar. If there is any free water standing on the surface of the sample, this is carefully decanted (poured) into a waste container. Using a clean scoopula, the sample in the jar is mixed to insure it is homogeneous (well mixed).

8.15 Collect the weight of the wet sample and weighing tin. Measure out approximately 5-10 grams of sample. The weights are recorded to the nearest 0.01 gram. When the balance displays the "*" press the print button on the balance.

The weight of the weighing tin and sample in then printed in the highlighted cell (Tare + Sample). The cursor automatically goes to the next Tare Weight cell

8.16 Proceed with **steps 8.13 to 8.15** until all samples are done. The spreadsheet should be saved periodically.

8.17 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.

8.18 As each sample is weighed, its tin is placed on a metal tray. As the tray of samples is complete, it is carefully placed on a shelf in the 103-105 ° C drying oven. Samples are dried overnight, optimally for at least 12 hours. ILM5.4/ISM01.1 soils have a 12 hour minimum drying time, and a 24 hour maximum.

8.19 If for some reason the samples cannot be dried overnight, a shorter drying time may be used, provided the sample is dried to a demonstrated constant weight. This procedure can only be used with the Supervisor's approval and must be carefully documented. The weighed wet sample is placed in the drying oven for at least four hours. Following four hours, the sample may be removed from the oven, cooled in the dessicator, weighed, and returned to the oven. After an additional 30 minutes of drying in the oven, the sample may be removed, cooled, reweighed, and replaced in the drying oven for an additional 30 minutes of drying. If two consecutive weights are equal (within 4%) then the sample has been dried to a constant weight, and the percent solids may be calculated.

This procedure must be documented in the PMOIST binder. A note is made for the sample that undergoes this procedure. The reverse side of the page is used to record the initial wet sample weight and time sample is first placed in the oven. When the sample is removed for weighing, the time and weight are also recorded on this page. There must be at least three weights and times recorded (initial and three consecutive constant weights).

ILM5.4/ISM01.1 samples which cannot be dried for the full 12 hours must also have demonstration of a constant final weight. Data must be recorded for a minimum of two repetitive 'weigh/dry/desiccate/weigh' cycles with a minimum of 1-hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight of the last cycle.

8.20 DAY TWO: Open the saved **PMoist** spreadsheet from the previous day. Enter date and time the samples were taken out of the oven and the oven temperature. Place the cursor so that it is in cell J2 (Final Wt). Ensure the balance has been calibrated and is zeroed as described above.

8.21 The tray of samples is carefully removed from the oven and the samples are placed in a dessicator to cool.

8.22 Put the cooled, dried sample and pan on balance. When the balance stabilizes press the **Print** button on the balance.

8.23 Select the **Calculate Pmoi**st button. Click again on the Data Entry sheet of the worksheet if the calculations appear acceptable. On the header information page select the **Copy Data** button. Click on the **Print Report** button. Save and close the Excel program.

8.24 If the Balance Talk computer program is not operational, discuss the issue with the Supervisor.

8.25 From the main page of Omega click on the **Data Entry** button.

8.26 Select the **Add** button. From the instrument ID dropdown box select Dry Weight Oven (DWO). Complete the Run Start Date field. From the Analyst field dropdown box select the analyst name. Select the **Data Import** button.

8.27 From the **Specification** dropdown box select **Omega Excel File. S**elect the **Run Import** button. From the **Open Dialog** box select the **PMoist** file and then select the **Open** button. Leave the value of 1 in the Text box and then select the **OK** button in the Spreadsheet Range box.

8.28 After Omega finishes importing the data, select the Data button. Of the three fields only the raw field should have value. Select the **Calc Seq** button and then select **Yes** when asked to continue.

9. Data Reduction and Calculations

The software performs all calculations. The results are reviewed by an independent analyst and then verified in the LIMS Omega System.

10. Quality Assurance/Quality Control

10.1. A duplicate sample must be performed at least every 20 samples.

10.2. The balance calibration must be within specifications.

10.3. The balance must be calibrated monthly with the Class "1" weights located in the QA/QC Office.

10.4. The drying oven temperature must be stable in the range of 103-105° C.

10.5. Times samples are put into and taken out of the oven are recorded.

11. Data Validation and Reporting

Results are checked first by the analyst and then by the supervisor. The print-out of the completed Excel spreadsheet is delivered to the Supervisor or designee for review.

Following Supervisor review and approval, all data are QA Validated in the Omega LIMS. The Excel spreadsheet is dated and sequentially numbered and scanned into the PMOIST optical file database for permanent storage. The original hardcopy record is saved in the PMOIST binder.

12. Corrective Action Procedures

12.1 If the balance calibration exceeds acceptance criteria, recalibrate it according to manufacturer's instructions.

12.2 If the drying oven shuts down during the night, the analysis will need to be restarted, but samples will not need to be re-weighed.

12.3 If drying oven temperature exceeds 110° C, samples will need to be reprepped and reanalyzed.

12.4 If a sample spills during the final weighing, it will need to be reprepped.

12.5 If a sample and duplicate RPD vary more than 35%, another independent analysis of the sample must be performed.

12.6 If sample drying time is not within 12 to 24 hours for ILM/ISM soils, the time must be noted for inclusion in the project narrative.

13. Health and Safety

Precautions to protect analysts include the nature of toxicity or carcinogenicity of samples used in the method. Lab coat, gloves and safety glasses must be worn at all times in the lab.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

APHA, AWWA, and WEF, "Standard Methods for the Examination of Water and Wastewater," 18th edition.

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Organic Analyses (Multi-Media, Multi-Concentration) SOM01.2

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Analyses (Multi-Media, Multi-Concentration) ILM05.4

U.S. Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration) ISM01.1

Balance Talk user manual

16. Amendment to SOP

16.1 Due to some sample matrixes and/or special client requests, it may be necessary to "pre-dry" samples prior to the sampling procedure. These samples will be specifically identified by the Laboratory Supervisor or Project Manager.

16.2 Visually determine or weigh out approximately 15 – 20 grams of each sample in an appropriate container. (An aluminum pan should be sufficient)

16.2.1 Place each sample in a drying oven set at 60°C overnight.

16.2.2 Follow the procedure as described in **Section 8**.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME.

SOP 20.0003

Rev. 3

	Signature	Date
QA Director:	Sharon B Lauk	4/9/08
Lab Director:	[illegible]	4/9/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE
For

Logging Workorders and Samples into Omega ME

Rev. 3

1 Scope and/or Applications

This Standard Operating Procedure (SOP) describes the procedures used to create workorders and log samples into Omega ME. Other steps in sample receipt and chain of custody documentation are described in separate SOP, Number 30.0003, entitled SOP for Sample Receipt, Storage and Tracking

2. Personnel Qualification and Responsibilities

Employees should have read and understood this SOP.

3. Summary of Procedure

This document assumes that a client and project profile has already been defined in Omega ME.

IMPORTANT: Omega ME generates unique sequence numbers (workorder numbers). These are assigned in order as samples are received at the laboratory. **It is essential that all new workorder numbers be assigned in sequence.** This number is assigned by the laboratory and is recorded on the chain of custody form **before** the samples are logged into Omega ME. The Omega ME work order number and the Mitkem project number are the same. If care is not taken, the information logged into Omega ME Work Order and the information in the Mitkem Project folder will not match.

4. Sample Preservation, Container, Handling and Storage

See separate SOP, Number 30.0003, entitled SOP for Sample Receipt, Storage and Tracking

5. Interferences and Potential Problems

Not applicable

6. Equipment and Apparatus

Not applicable

7. Reagents

Not applicable

8. Procedure

8.1 CREATING A WORKODER

8.1.1 From the main page of Omega ME click [WorkOrders] Figure 1.

8.1.2 The Omega ME - WorkOrders Form will open.

8.1.3 On the menu bar at the top of the screen click the [Add] button. This will add the next available WorkOrder and clear the screen of any prior information.

On the screen click the down arrow next to the [Client ID] button to open a list of all of Mitkem's current clients. Instead of searching through the long list of clients type in the first letters of the client name to shorten the selection. Select the proper client. The client name and address will automatically appear in the Client Information box. Figure 2.

Choose the appropriate project by pressing the down arrow on the [Client Project] button, Figure 3. If the client provides a purchase number (PO) enter it in the box provided.

Enter the received date (date samples received by the lab). It is necessary to assign the WorkOrder numbers in order by date samples received. The hardcopy due date [HC Due] is then filled in automatically by Omega based on the number of days (TAT HC/FAX) listed.

If a workorder requires a short turnaround time **(24,48 or 72 hours, etc.)**, click the checkmark for the [**Fax Results to Client**] box. The cursor will automatically move to the **(TAT HC/FX)** box which is the box on the right-hand side in which the turnaround time number will need to be entered: **24=1,48=2,72=3**. When this is complete tab down to the box that is highlighted red which will exhibit the date the workorder is due. If the hardcopy due date differs from the fax due date, enter this information now.

For EPA workorders the client is **EPAINV.** The project (I,II,III,IV,V,VI,VII,VIII,IX,X, SOM_PE) is next selected by looking at the Scheduling Notification Form. This form identifies the case number, the Region the samples are from, the analyses required and

turnaround time. Setting up an EPA workorder differs from a commercial workorder in that the Case Number and SDG fields must be completed from the Chain of Custody after protocol has been selected. In some cases, a particular Case will have samples for both water and soils. The soil samples and aqueous samples may be logged into separate Work Orders. The Mitkem project manager will provide this information. Some EPA / commercial projects are kept open for more than one day, so be careful to change the received date as the samples arrive at the lab in order to update the due date properly. Click the [Login] button to begin logging in the samples.

8.1.4 A new screen appears in order to log in the sample information. The fields that need to be completed include:

1. **Click on ADD Samp.** This will add the first sample to be logged in or the information entered into the other fields will not be saved. **Date Received:** Enter the date and time that the samples were received, **Figure 4.**
2. **Client Sample ID:** Type the client ID for each sample as it appears on the Chain of Custody. **Tag Number:** If the ID needs to be truncated because it is more than 10 characters long, type the truncated ID in the Tag Number field. Example: **SB29S-32-12302 = 29S3212302.**
3. **Collection Date:** Enter the date and time that the sample was collected, if a date and time are present. A date is necessary as this is used to keep track of hold times (for non-EPA work). The time of collection is not a necessity, unless the analysis has a 24-hour or 48-hour hold time.
4. **Matrix:** Select the down arrow and choose the appropriate matrix for the sample. If a solid matrix is chosen the PMOIST automatically appears to schedule a percent solids analysis.
5. **Bottle Type:** Choose the type of bottle for each sample fraction by clicking the down arrow and selecting the appropriate bottle.
6. **Containers:** Enter the total number of bottles received per sample.
7. **Storage/Status:** Type the location where the bottle will be stored in the Sample Receiving area. Most of the samples are stored in the walk-in (R1). VOA samples are stored in the refrigerators in the VOA lab. Aqueous metals are stored outside the walk-in cooler on a metal rack.
8. **COC ID:** Some clients require this information to be included. The Mitkem project manager will notify the Receiving department when this needs to be done. The COC ID is required for SOM01.2. If there is more than one COC, associate the correct COC ID to the correct sample. To input the COC ID, go to the AFCEE tab and type in the COC ID in COC ID. Figure 5
9. **SA (CODE):** If the workorder requires the COC ID field to be completed, then this field also must be completed. Select whether the sample is a FB (field blank),TB (trip blank), RB (rinsate blank), or EB (equipment blank). This information can be found on the COC. This field is found under the AFCEE tab
10. **Temp (C): The temperature of the cooler needs to be completed for SOM01.2. In EPA cases where there is more than one cooler received for a particular Case, the correct temperature needs to be associated with the correct samples.** This field is also found under the AFCEE tab.

11. When all the above information has been entered, the next step is to choose the correct test. The tests for a particular project may be found by clicking on the Test column and using the drop down arrow.
12. Click ADD Frac if another fraction needs to be added. Fractions are added when different bottles with different preservatives are present. For example, a client sends in a sample for volatile, semivolatile, pesticides, metals and cyanide analysis. There will be an "A", a "B", a "C" and a "D" fraction. For example, they will be logged in as "A" for volatile, "B" for semivolatile and pesticides, "C" for metals and "D" for cyanide. This is due to each sample bottle being different and having different preservatives. Figure 6.
13. To add additional samples, click on the ADD Samp button. Enter the sample ID, date and time of collection, matrix, type of bottle and storage location. Once all this info has been entered, choose the test. An easier method when logging in a number of samples with identical analyses is to use the Multilog button, Figure 6. Clicking the Multilog button automatically copies all information to the next sample except for Client ID, Date andTime of collection, and Tag Number. If multi-logging, type the client ID, date of collection and time of collection for each sample. Once this is done, click on POST. Figure 7.
14. If samples arrive with designated QC (duplicates and matrix spikes) then check the MS button on the right side of the screen after you have chosen the appropriate test Be sure to change the number of bottles for this sample, as there will be more sample bottles. Figure 8
15. If specific tests are included on the Chain of Custody but the analysis is on hold, then check the "Sample on Hold" box. Figure 9
16. Comments should be added if warranted. For example, if there is limited sample volume, or the Chain-of-Custody indicates that the samples are dirty. Go to the Comments: and type in the necessary comment.
17. Labels are printed and affixed to the bottles. Labels may be printed by clicking on the Label button. Click over the samples in which labels need to be printed. Click the OK button and then the printer icon.
18. Once the labels have been printed, a work order needs to be printed. Back out to the work order page. Click on WO Rpt. Place the work order into the folder. Figures 10 and 11
19. A preliminary invoice will also need to be printed and placed into the folder. Click on the Reports button. Click on Base Report. Click on Clear All. Check on Invoice and then click on Preview Report and print the invoice. Figures 12, 13 and 14

8.1 5 Any correction to the workorder that greatly impacts the workorder such as a significant change in holding time or turn around time, the addition of new tests, and/or the addition or cancellation of samples must also be verbally communicated to the appropriate lab supervisor by the project manager. An email is also an effective means of communication.

8.2 REVIEW of the Work Order

8.2.1 The Mitkem Project folders are handed out by the Project Managers. It is the responsibility of the project manager to ensure that the sample ID, date and time of sample collection, date of sample receipt and the correct tests are logged in for each sample. Corrections are made, if necessary, and a new Work Order printed out and placed in the red folder.

8.2.2 When adding a workorder that is not going to be invoiced (Mitkem work, PE, etc..) or if the workorder is cancelled one of the following steps must be performed:

1)The workorder client name is changed to Mitkem.

2)All samples are designated as non-billable

3)For PE samples the characters "PE" are added to the SDG number field.

4) If the workorder is to be cancelled with no billable fractions and/or tests, the following steps should be followed:

4.1 Fill in Log Review with the date.

4.2 Click on Cancel Work Order button

4.3 The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.

5)If the workorder is to be cancelled but some fractions and/or test codes can be billed, then follow these steps:

5.1 One sample should be logged in with NO-TEST and the correct priced added.

5.2 Fill in Log Review with the date.

5.3 Click on Cancel Work Order button

5.4 The Completed, Validated, Reported, Faxed, Mailed and Invoiced fields will be populated with the current date. The Project No:, Case No:, SDG No: and EDD By: fields will be populated with Canceled.

5.5 An invoice can be generated.

9. Data Reduction and Calculations

Not applicable

10. Quality Assurance / Quality Control

The Project Manager reviews the work order within one day of sample receipt. Any changes or corrections are implemented at this step.

11. Data validation and Reporting

Not applicable

12. Corrective Action Procedures

Not applicable

13. Health and Safety

Not applicable

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Not applicable

Figure 1

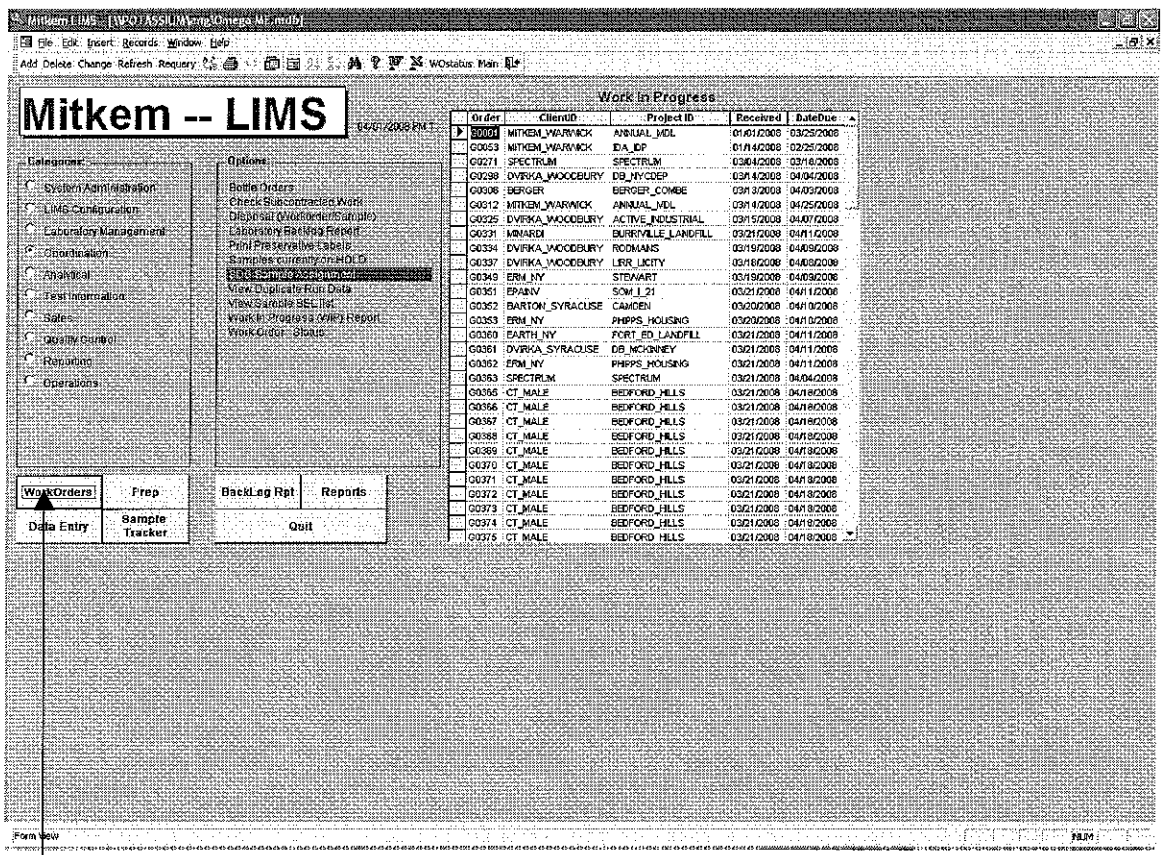
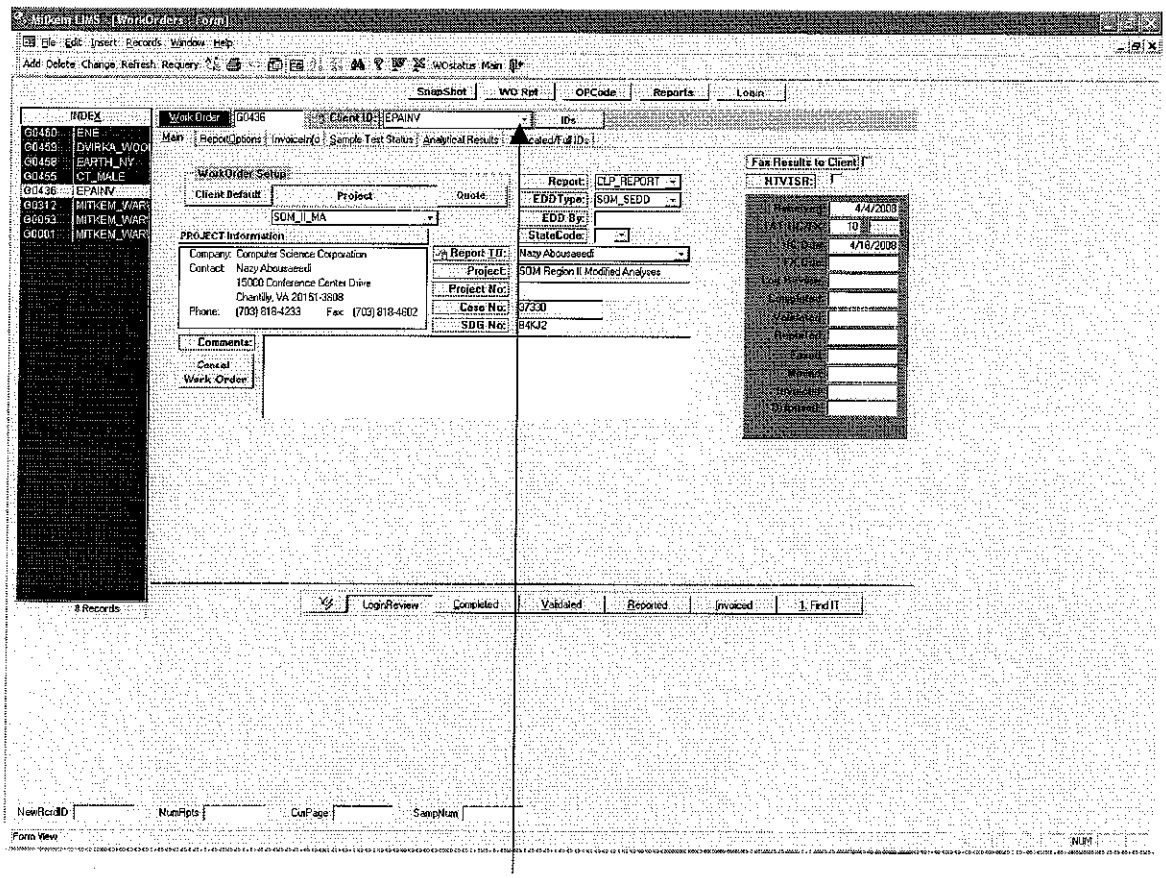


Figure 2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File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0093 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order
G0436
Client ID:
EPAINV
IDs
Main
Report Options
Invoicing
Sample Test Status
Analytical Results
Scaled/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousseedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233
Fax: (703) 818-4602
Report:
CLP_REPORT
EDDType:
SOM_SEDD
EDD By:
StateCode:
Report TO:
Nazy Abousseedi
Project:
SOM Region II Modified Analyses
Project No:
Case No:
37330
SDG No:
84KJ2
Comments:
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Fax Results to Client
NTVISR:
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4/16/2008
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Form View
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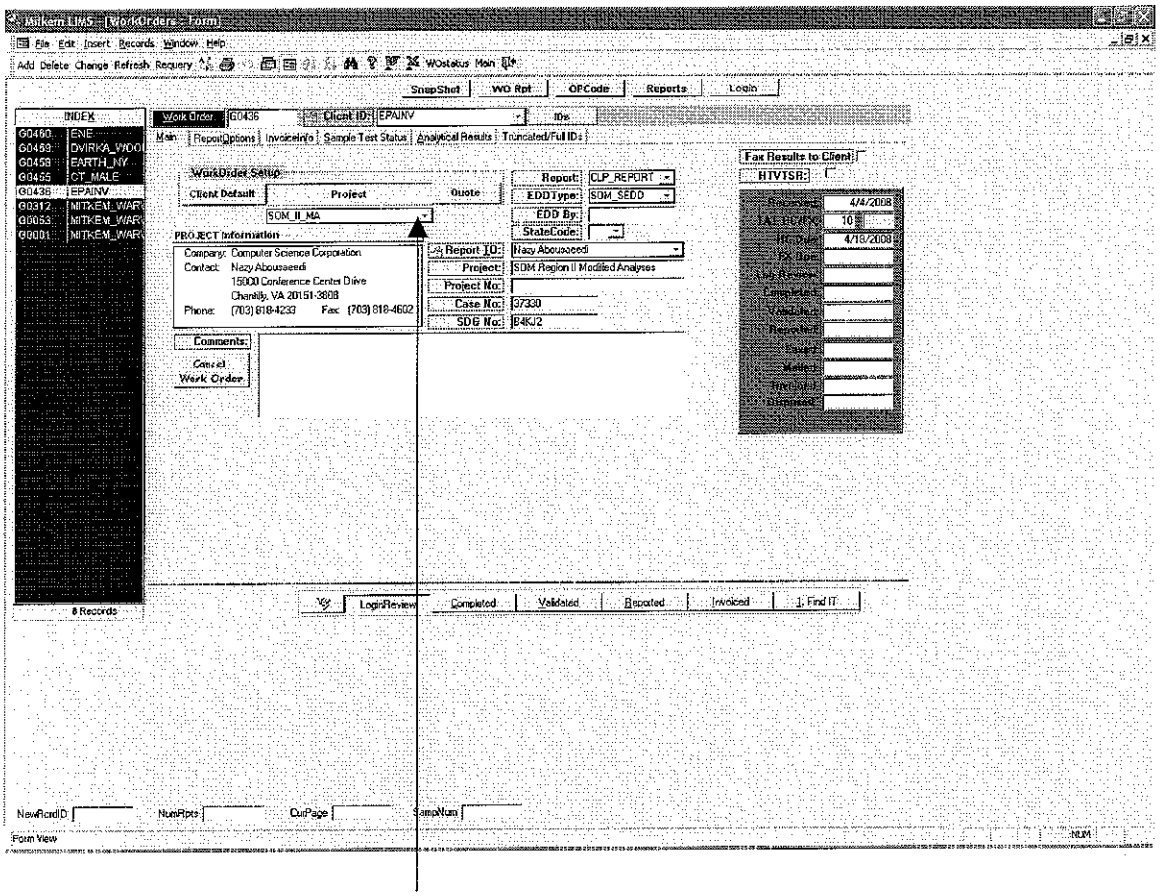
Figure 3


Mitkem LIMS - [WorkOrders - Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
SnapShot
WO Rpt
OPCode
Reports
Login
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
8 Records
Work Order G0436
Client ID: EPAINV
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousaeedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233 Fax: (703) 818-4602
Report: CLP_REPORT
EDDType: SOM_SEDD
EDD By:
StateCode:
Report TO: Nazy Abousaeedi
Project: SOM Region II Modified Analyses
Project No:
Case No: 37330
SDG No: B4KJ2
Comments:
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Fax Results to Client
HTVTSR:
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Validated
Reported
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Form View
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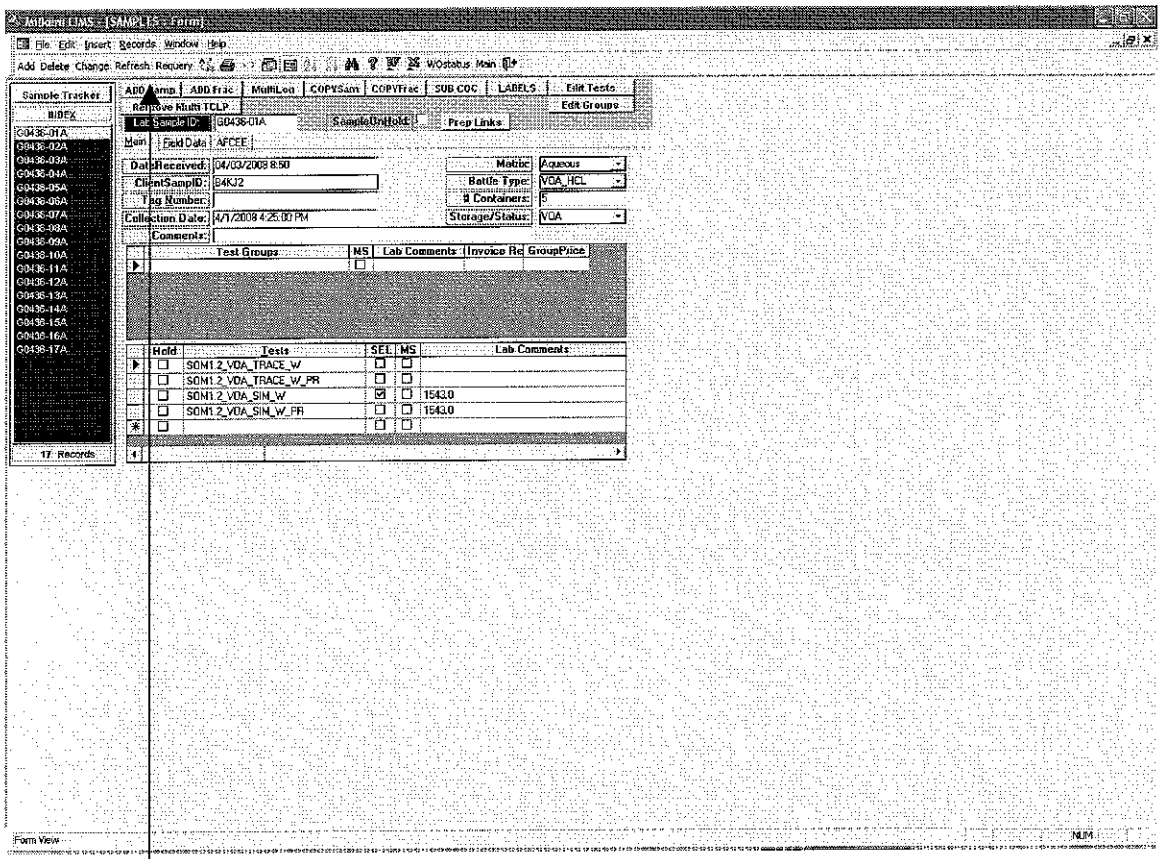
Figure 4


Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery W/Ostatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
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G0436-14A
G0436-15A
G0436-16A
G0436-17A
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ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Edit Groups
Remove Multi TCLP
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SampleOnHold
Prep Links
Main
Field Data
AFCEE
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ClientSampID:
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Tag Number:
Collection Date:
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Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
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Storage/Status:
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Invoice Re
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1543.0
Form View
NUM

Figure 5

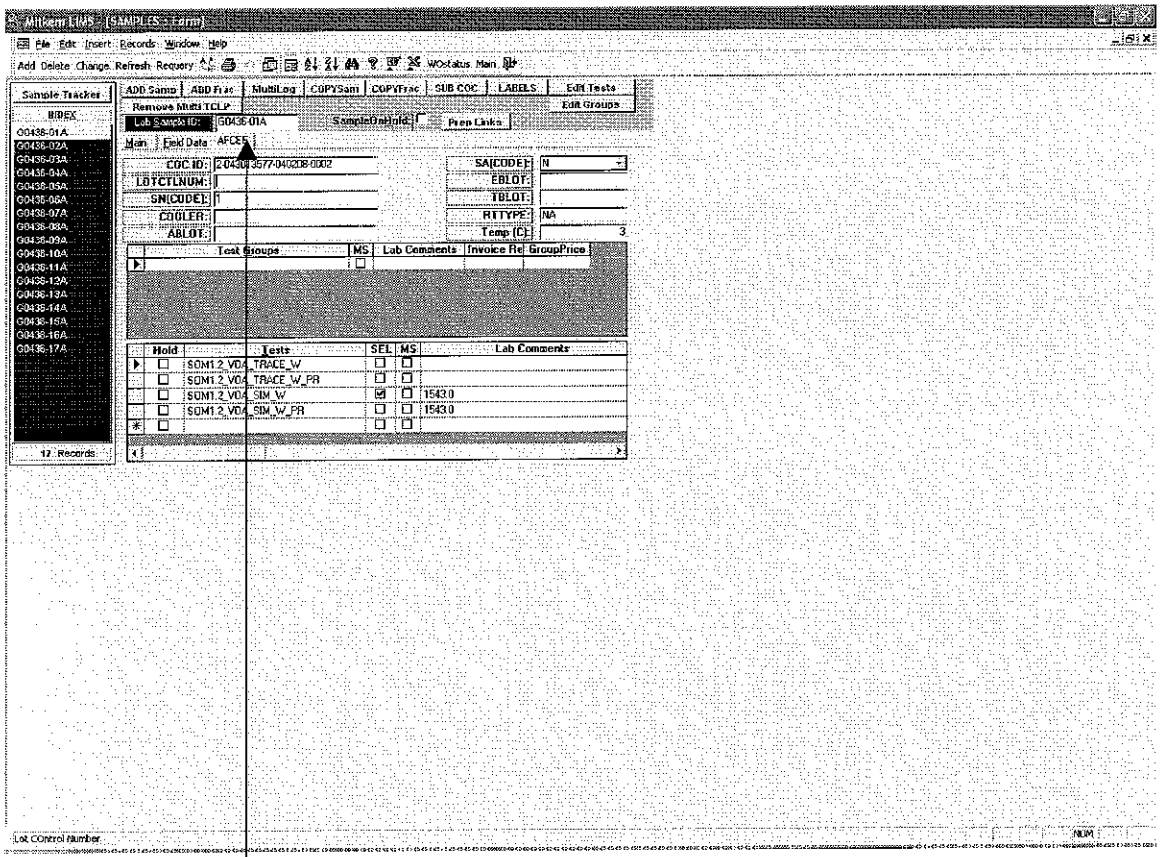

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleInstrId:
Prep Links
Main
Field Data
AFCEE
COC ID:
LOTCTLNUM:
SN(CODE):
COOLER:
ABLOT:
SA(CODE):
EBLOT:
TBLOT:
RETYPE:
Temp (C):
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
SOM1.2_VOA_SIM_W_PR
15430
15430
Lot Control Number
NUM

Figure 6

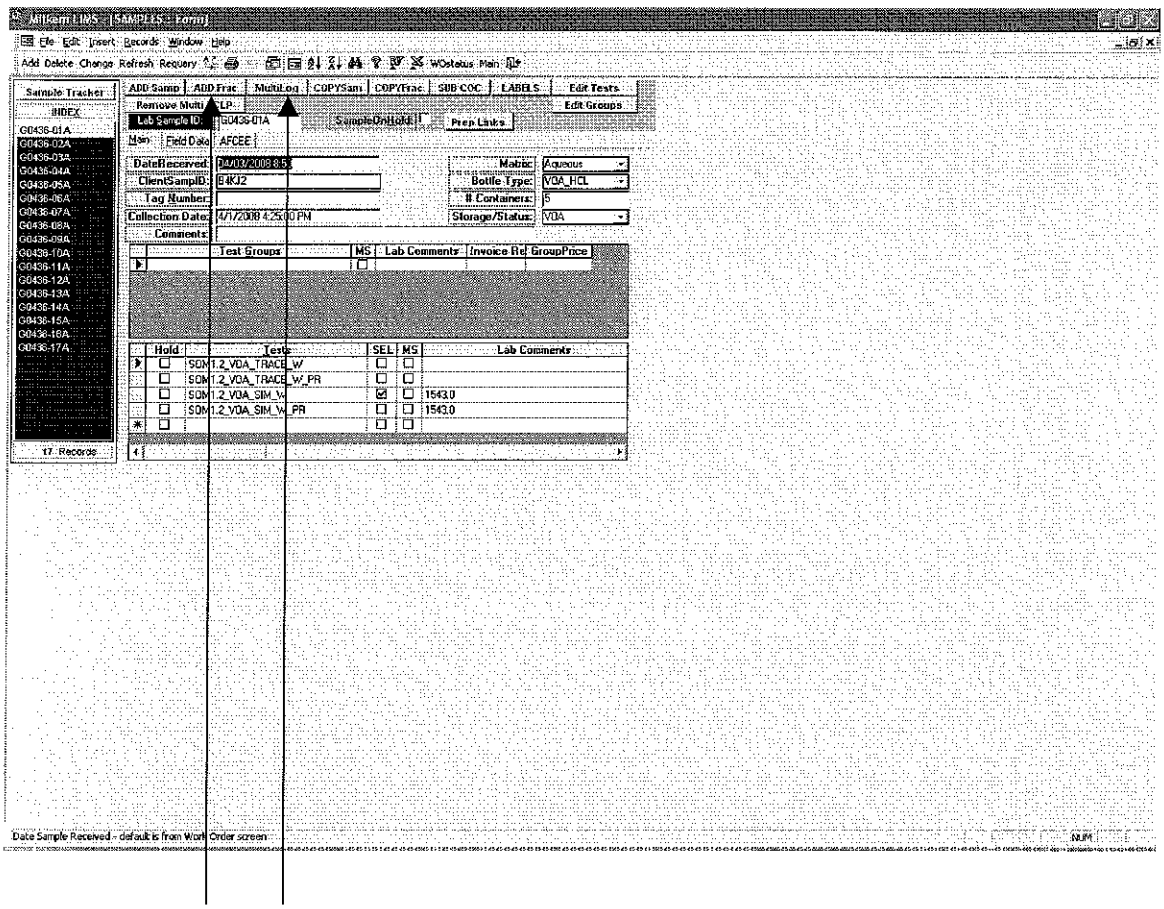

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Edit Groups
Remove Multi
Lab Sample ID:
G0436-01A
SampleOnHold
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:5
ClientSampID:
B4KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Date Sample Received - default is from Work Order screen
NUM

Figure 7

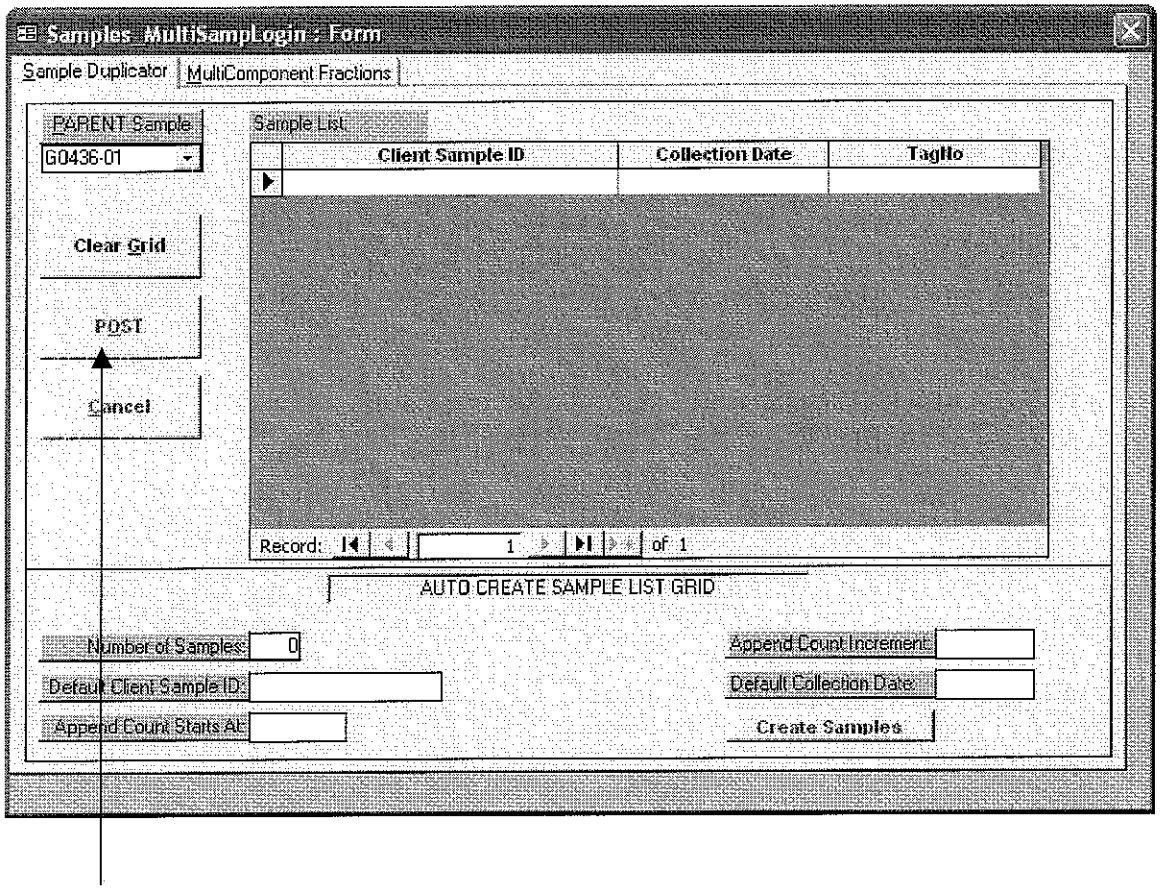

Samples_MultiSampLogin : Form
Sample Duplicator
MultiComponent Fractions
PARENT Sample
G0436-01
Sample List
Client Sample ID
Collection Date
TagNo
Clear Grid
POST
Cancel
Record:
1
of 1
AUTO CREATE SAMPLE LIST GRID
Number of Samples:
0
Default Client Sample ID:
Append Count Starts At:
Append Count Increment:
Default Collection Date:
Create Samples

Figure 8

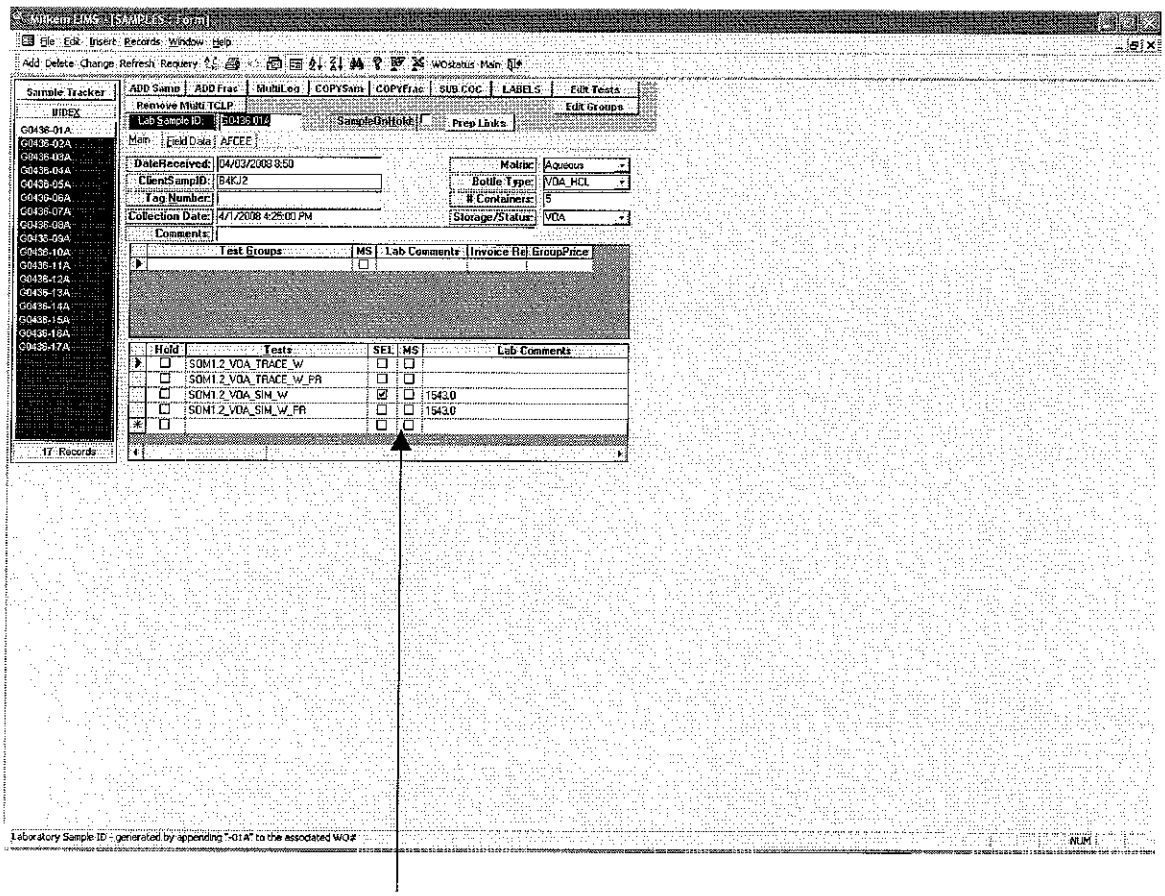

Mitkem EIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
SampleDuplicate
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
64KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 9

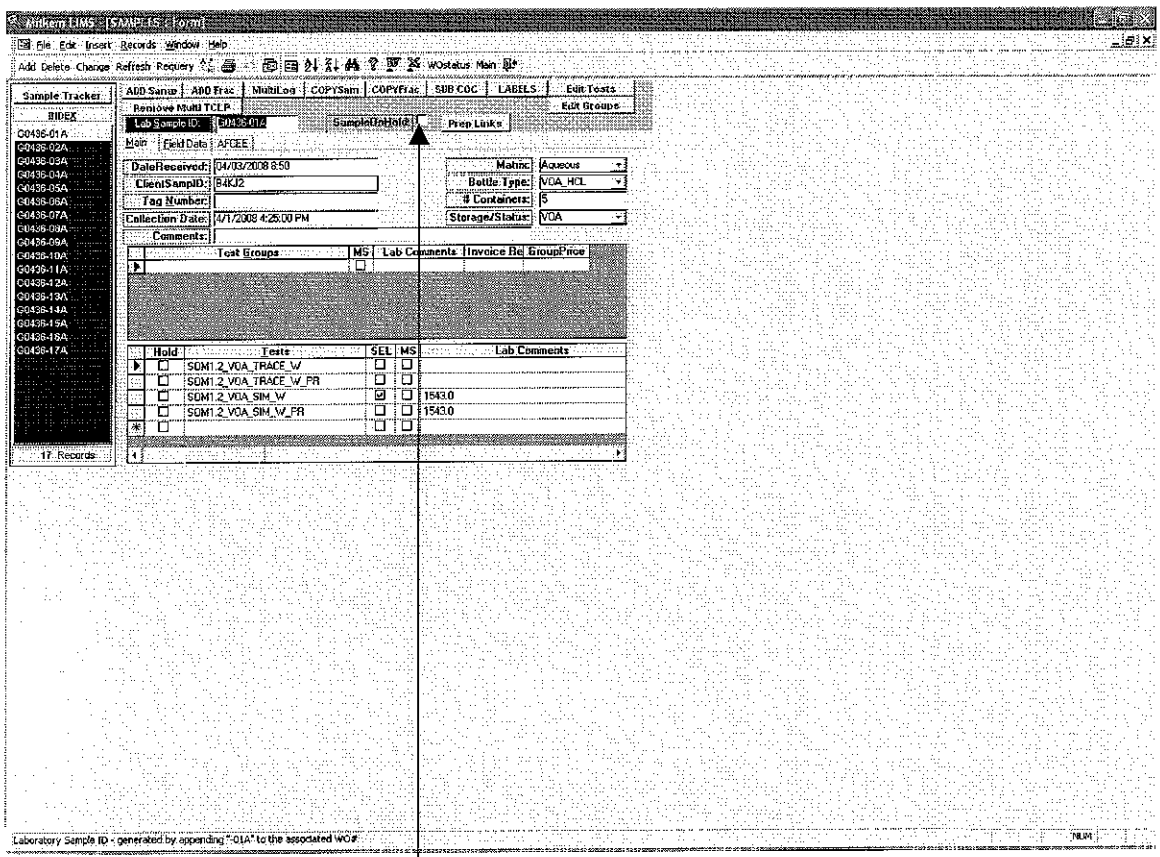

Mitkem LIMS - [SAMPLES : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Sample Tracker
INDEX
G0436-01A
G0436-02A
G0436-03A
G0436-04A
G0436-05A
G0436-06A
G0436-07A
G0436-08A
G0436-09A
G0436-10A
G0436-11A
G0436-12A
G0436-13A
G0436-14A
G0436-15A
G0436-16A
G0436-17A
17 Records
ADD Samp
ADD Frac
MultiLog
COPYSam
COPYFrac
SUB COC
LABELS
Edit Tests
Remove Multi TCLP
Edit Groups
Lab Sample ID:
G0436-01A
SampleOnHold
Prep Links
Main
Field Data
AFCEE
DateReceived:
04/03/2008 8:50
ClientSampID:
84KJ2
Tag Number:
Collection Date:
4/1/2008 4:25:00 PM
Comments:
Matrix:
Aqueous
Bottle Type:
VOA_HCL
Containers:
5
Storage/Status:
VOA
Test Groups
MS
Lab Comments
Invoice Re
GroupPrice
Hold
Tests
SEL
MS
Lab Comments
SOM1.2_VOA_TRACE_W
SOM1.2_VOA_TRACE_W_PR
SOM1.2_VOA_SIM_W
1543.0
SOM1.2_VOA_SIM_W_PR
1543.0
Laboratory Sample ID - generated by appending "-01A" to the associated WO#
NUM

Figure 10

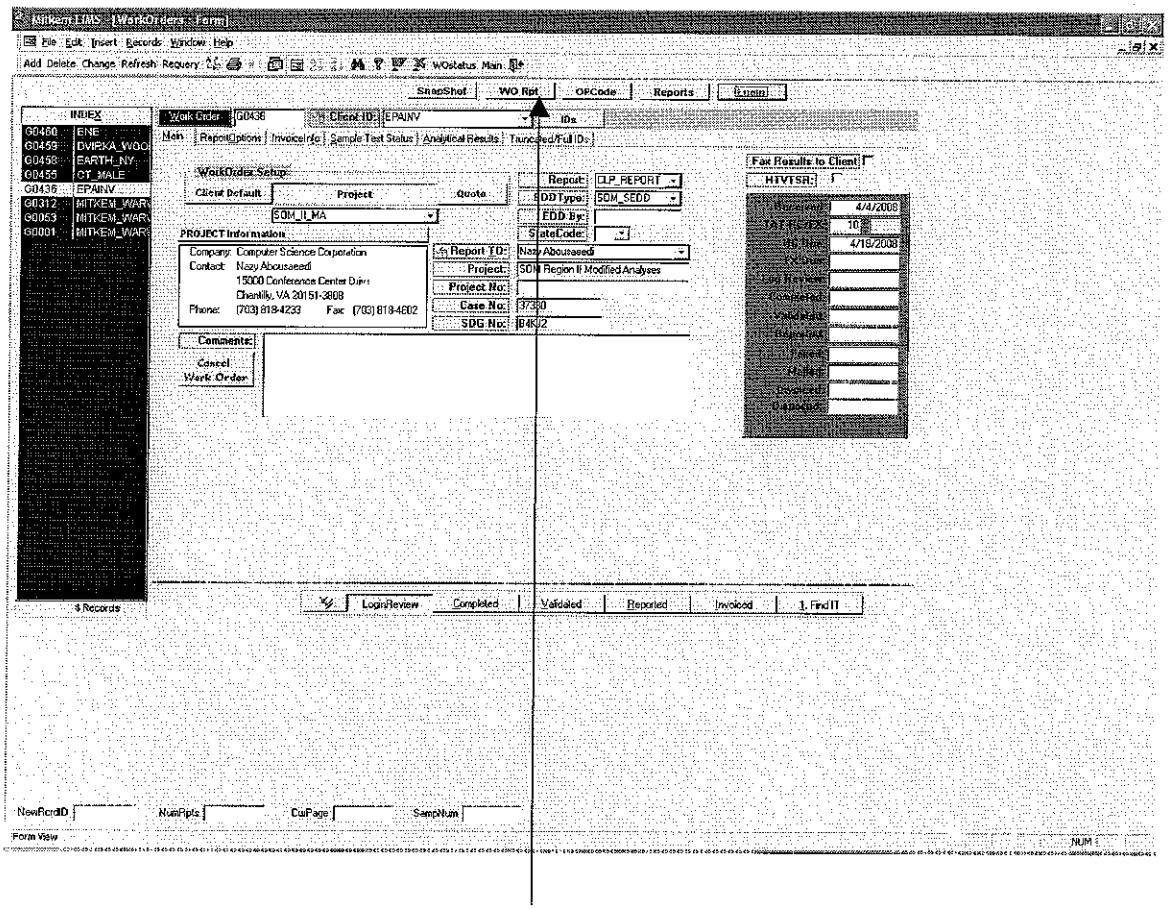

Mitkem LIMS - [WorkOrders : Form]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rpt
OPCode
Reports
INDEX
G0460 ENE
G0459 DVIRKA_WOO
G0458 EARTH_NY
G0455 CT_MALE
G0436 EPAINV
G0312 MITKEM_WAR
G0053 MITKEM_WAR
G0001 MITKEM_WAR
Work Order G0436
Client ID: EPAINV
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup:
Client Default
Project
Quote
SOM_II_MA
PROJECT Information
Company: Computer Science Corporation
Contact: Nazy Abousaeedi
15000 Conference Center Drive
Chantilly, VA 20151-3808
Phone: (703) 818-4233 Fax: (703) 818-4602
Report: CLP_REPORT
SDD Type: SOM_SEDD
EDD By:
StateCode:
Report TO: Nazy Abousaeedi
Project: SOM Region II Modified Analyses
Project No:
Case No: 37330
SDG No: B4K02
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
4/4/2008
10
4/19/2008
LoginReview
Completed
Validated
Reported
Invoiced
Find IT
NewRecID
NumRpts
CurPage
SampNum
Form View
NUM

Figure 11

Mitkem Laboratories *08/Apr/08 16:19* **WorkOrder: G0436**

Client ID: EPAINV
Project: SOM Region II Modified Analyses
Location:
Comments: N/A

Case: 37330
SDG: B4KJ2
PO: EP-W-05-030

Report Level: CLP_REPORT
EDD: SOM_SEDD
HC Due: 04/18/08
Fax Due:

Sample ID	HS Client Sample ID	Collection Date	Date Rec'd	Matrix	Test Code	Lab Test Comments	Hold	MS	SEL	Storage
G0436-01A	B4KJ2	04/01/2008 16:25	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-02A	B4KJ3	04/01/2008 12:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-03A	B4KJ4	04/01/2008 16:20	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-04A	B4KJ5	04/01/2008 17:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-05A	B4KJ7	04/01/2008 9:30	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-06A	B4KK0	04/01/2008 8:00	04/03/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA
					SOM1.2_VOA_TRACE_W		☐	☐	☐	VOA
G0436-07A	B4KH4	04/03/2008 11:10	04/04/2008	Aqueous	SOM1.2_VOA_SIM_W	1543.0	☐	☐	☑	VOA

Client Rep. Agnes R Ng

Page 1 of 3

Figure 12

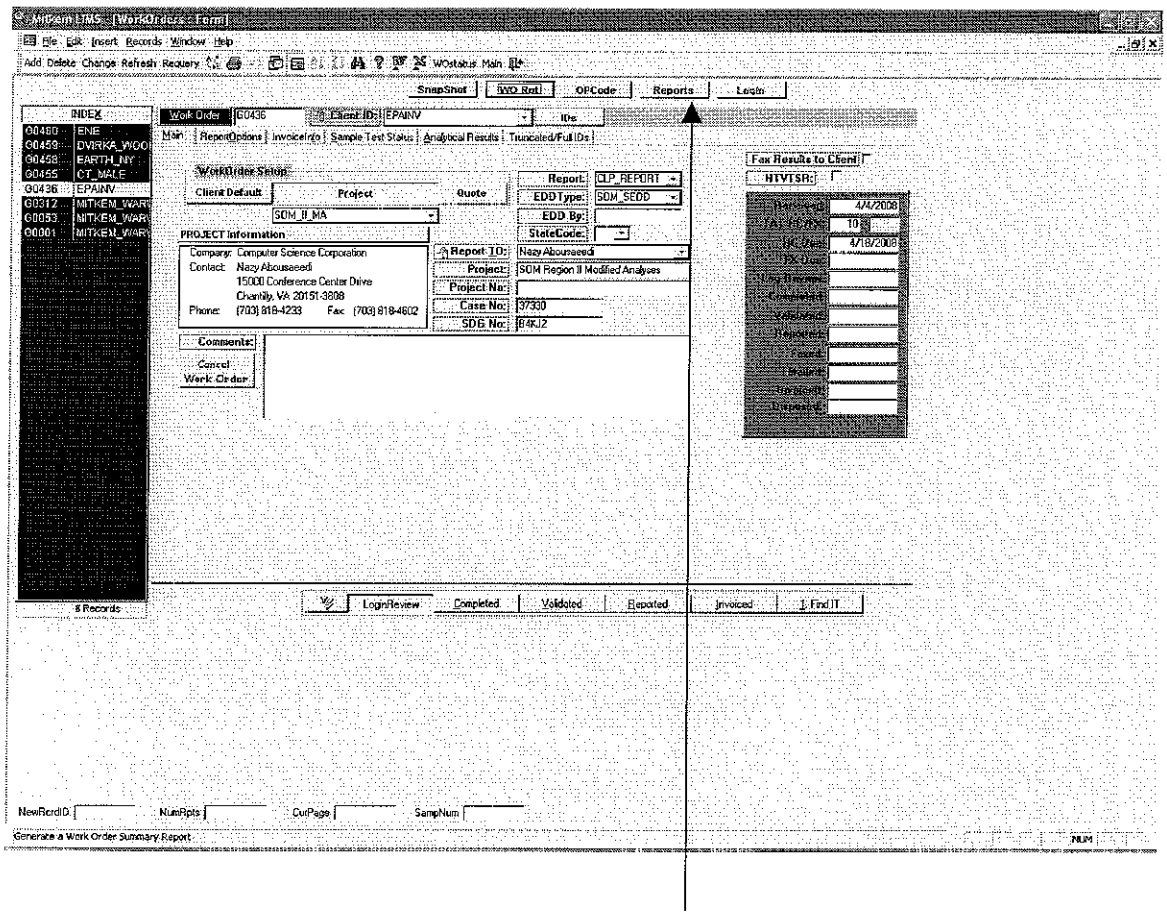

File Edit Insert Records Window Help
Add Delete Change Refresh Requery
SnapShot
WO Rptl
OPCode
Reports
Login
INDEX
Work Order
Client ID:
IDs
Main
ReportOptions
InvoiceInfo
Sample Test Status
Analytical Results
Truncated/Full IDs
WorkOrder Setup
Client Default
Project
Quote
Report:
EDD Type:
EDD By:
StateCode:
PROJECT Information
Report ID:
Project:
Project No:
Case No:
SDG No:
Comments:
Cancel Work Order
Fax Results to Client
HTVTSR:
LoginReview
Completed
Validated
Reported
Invoiced
Find IT
NewRcrdID
NumRpts
CurPage
SampNum
Generate a Work Order Summary Report
NUM

Figure 13

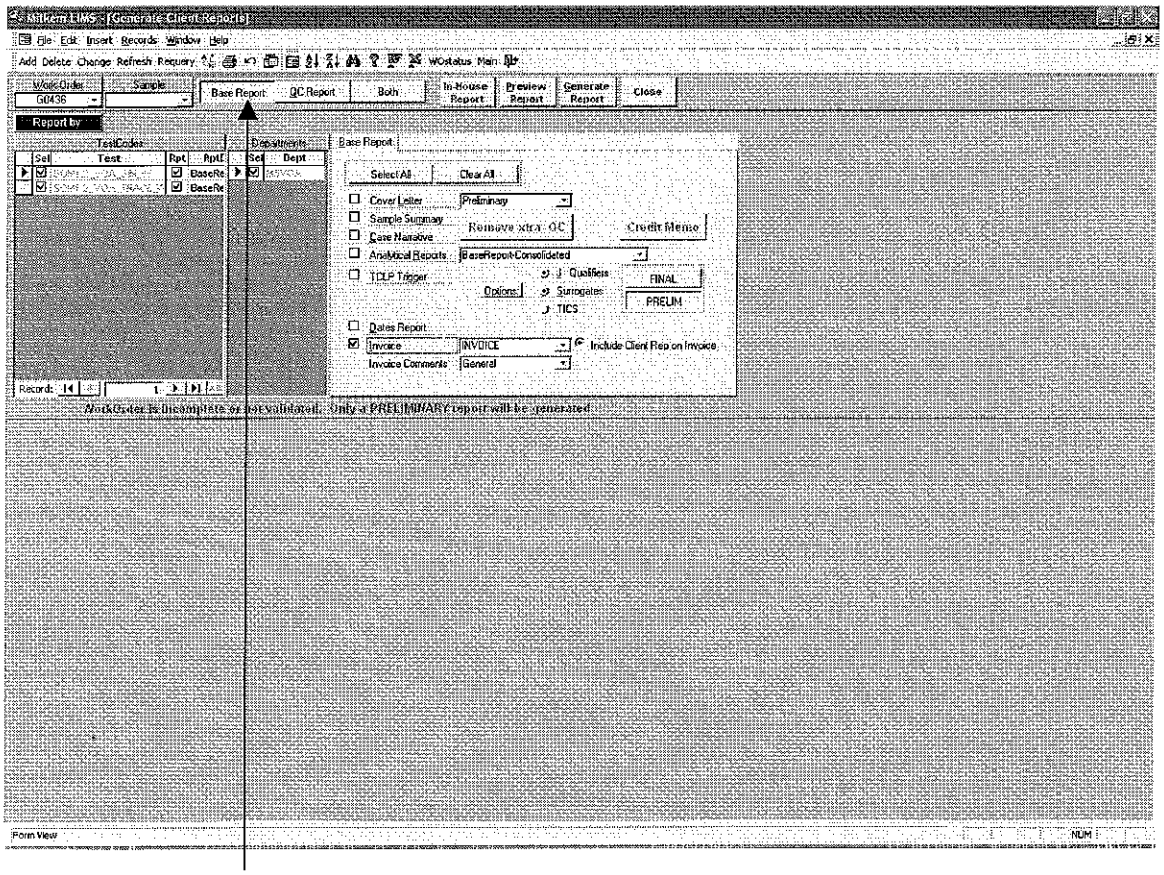

Mitkem LIMS - [Generate Client Reports]
File Edit Insert Records Window Help
Add Delete Change Refresh Requery
WOstatus Main
Work Order
G0436
Sample
Base Report
QC Report
Both
In-House Report
Preview Report
Generate Report
Close
Report by
TestCodes
Departments
Base Report
Select All
Clear All
Cover Letter
Preliminary
Sample Summary
Case Narrative
Remove xtra QC
Credit Memo
Analytical Reports
BaseReport-Consolidated
TCLP Trigger
Options
Qualifiers
Surrogates
TICS
FINAL
PRELIM
Dates Report
Invoice
INVOICE
Include Client Rep on Invoice
Invoice Comments
General
Records:
Work Order is incomplete or not validated. Only a PRELIMINARY report will be generated.
Form View
NUM

Figure 14

Bill To:

INVOICE

EPA Financial Management Center
Attn:
Research Triangle Park
Research Triangle Park, NC 27711

Number:
**G0436
Date:

Client Rep: Nazy Abousasedi
Work Order: G0436
PO Number: EP-W-05-030
Date Received: 04/04/2008

Project No:
Project Name: SOM Region II Modified Analyses
Case No: 37320
SDG No: B4KJ2

Test	Remarks	Matrix	Qty	Price	Test Total
SOM1.2_VOA_SIM_W	1543.0	Aqueous	17	$1.00	$17.00
SOM1.2_VOA_TRACE_W		Aqueous	17	$85.00	$1,445.00

Subtotal:	$1,462.00
Discount:	0.00%
Surcharge:	0.00%
Misc Charges:	$0.00
Payment Received:	$0.00
INVOICE Total:	$1,462.00

All invoices are due and payable net 30 days from receipt.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

SOP No. 30.0003

Rev. 12

	Signature	Date
QA Director:		10/17/08
Lab Director:		10/21/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample Receipt, Storage, Tracking and Disposal

Rev. 12

1. Scope and Application

This Standard Operating Procedure describes the procedures that must be followed upon receipt of samples at MITKEM. It describes the procedures to log-in and store samples. It also describes procedures for checking samples out of the storage area for analysis, and for final disposal of samples. Detailed procedures for entering work orders and samples into Mitkem's Laboratory Information Management System (LIMS) are described in a separate SOP Number 20.0003.

2. Personnel Qualifications and Responsibilities

The Sample Custodian should have a two-year degree in environmental science or a related field or have six months of on-the-job experience working with a trained and qualified Sample Custodian.

3. Summary of Procedure

When sample coolers are received, cooler custody seals and temperature are checked. The chain-of-custody forms are signed, and the condition of the samples are checked, including any sample custody seals. Each sample is assigned a specific individual sample number. Any discrepancies are documented and resolved. Samples are logged-in to the Omega LIMS following procedures in SOP 20.0003. Labels are generated, and affixed to samples. The pH of aqueous samples (Inorganic only, never VOA) are checked and if need be, either acid or base is added. The addition of preservative(and its lot number) is recorded on the Condition Form. Samples are stored in secure area under documented custody conditions. Certain types of VOC soil samples require special handling, as described in **Section 8.2.2**. Samples are kept at Mitkem for a specific time as called for by contract with the client, and then disposed of in accordance with federal, state and local regulations. The door to the sample receiving room is locked at all times, using a keypad code lock.

4. Sample Preservation, Containers, Handling, and Storage

Samples received at Mitkem are collected by our clients. Mitkem does provide preservatives and sample containers pre-cleaned by bottle suppliers.

Tables 7-1, 7-2 and 7-3 of Mitkem's Quality Assurance Plan (QAP) lists the type of container needed for a particular type of analyte and the proper preservative.

5. Interferences and Potential Problems

Any problem with sample condition including custody seals, breakage, discrepancies in chain of custody or other shipping documentation, cooler temperature, missing samples, etc. must be communicated to the client promptly and resolved. The Sample Custodian or assistant documents the problem and notifies the Mitkem Project Manager, who will then notify the client. For CLP the Project Manager will notify the Sample Management Office (SMO).

6. Equipment and Apparatus

6.1 NIST-calibrated temperature "gun".

6.2 Condition Forms: Non-CLP, CLP (DC-1).

6.3 Orange colored Sample Condition Notification forms.

6.4 Narrow range pH paper, capable of recording pH 0- 2.5, and 11-13.

6.5 Computer with Accessories to run the LIMS Omega system.

6.6 Label printer and labels.

7. Reagents

7.1 Hydrochloric Acid (HCl), Fisher Trace Metal Grade.

7.2 Nitric Acid (HNO_3), Fisher Trace Metal Grade.

7.3 Sodium Hydroxide (NaOH), Certified A.C.S.

7.4 Zinc Acetate $(CH_3CO_2)_2$ Zn $2H_2O$, certified.

7.5 Sulfuric Acid (H_2SO_4), Fisher Trace Metal Grade.

7.6 Phosphoric Acid (H_3PO_4), Fisher Trace Metal Grade.

Reagents are ordered by the inorganic laboratory section, and kept in the sample receiving and bottle prep areas. Zinc Acetate is stored in the volatile laboratory as it is only used with volatile sample bottle prep.

If the reagent needed is in neat form (NaOH or Zinc Acetate) a new bottle may be obtained from the chemical storage area. If you take the next to the last or the last bottle for login dept use, you must inform the inorganic lab supervisor so more can be ordered.

8. Procedure

8.1 Log-In Procedures:

Samples received at Mitkem are received from private courier (FedEx, UPS, etc.) or Mitkem courier or delivery by the clients. All samples received must be accompanied by a chain-of-custody (COC) record.

8.1.1 Prior to opening coolers the receiving hood must be turned on, and safety glasses, lab coat, and gloves worn.

8.1.2 Select the next sequential red workorder folder with a pre-assigned work order number. Work order numbers consist of one alpha character followed by a four digit number YXXXX, in which Y is the alpha character indicating the number of years that the Omega LIMS System has been operational and XXXX is a sequential number for this workorder for the current year. For example, 2006 is assigned the alpha character "E" and the 5th set of samples received during 2006 is assigned work order number E0005. Occasionally, a client wishes for a workorder number to be ongoing for several shipments of samples (i.e. EPA). The project manager will notify the sample custodian if additional samples are to be added to an existing workorder (an open Sample Delivery Group or SDG).

8.1.3 Before opening coolers, check the outside of the opening of the shipping cooler for the presence of custody seals. Remove any shipping air bills and note the work order number on them. This information should be noted on the Sample Condition Form (See **Attachments 1 and 2**).

NOTE: There are two different types of Sample Condition Forms; one for commercial samples, and one for the US EPA contract samples (see Attachments). Place air bills and all other paperwork for this shipment in the appropriate red workorder folder.

8.1.4 Open the cooler underneath the hood and remove the chain-of-custody form. This is typically located in a plastic zip-lock bag on the top of the samples or taped to the inside of the cooler lid. Write the Mitkem workorder number on

the COC. Sign the COC, noting the date and time of receipt of the samples at Mitkem. Look for return air bills to return coolers to the client, these are typically included in EPA projects. If the COC is missing, notify the Mitkem project manager using the Mitkem Sample Condition Notification (printed on orange paper) form immediately. Do not process the samples. Store the samples in the walk-in cooler until the client is contacted and responds.

8.1.5 Take the temperature of the contents using the temperature gun or thermometer.

8.1.5.1 Use the Temperature Gun as follows:

8.1.5.1.1 Unwrap a sample in the opened cooler. Unwrap enough of the sample in order to take a temperature reading.

8.1.5.1.2 Take the temperature by holding the gun no more than two inches from the sample container. Do not aim at the label or cap of the sample. Take several readings on at least 2 samples at opposite ends of the cooler. . Record the temperature on the lower right hand corner of the chain-of-custody or where noted on the Chain-of-Custody. The acceptable temperature range is 4° ± 2°C (2° to 6°C). If the temperature is acceptable, move on to **Section 8.1.6**. If the temperature is not within this range, enter the temperature on the Sample Condition Form, circle "yes" to the question regarding the Sample Notification form, and fill out the Sample Notification form (See **Attachment 3**). For CLP projects, if the temperature exceeds 10°C, the project manager will contact SMO for corrective action guidance. Give the entire folder and the Sample Notification form to the project manager. The project manager will contact the client for instructions on how to proceed and return the folder to the sample custodian indicating action to be taken. During the interim, the samples in question will not be processed, but the closed cooler will be stored in the walk-in cooler.

8.1.5.2 If a temperature blank is present, use a thermometer as follows:

8.1.5.2.1 Insert the thermometer in the temperature blank.

8.1.5.2.2 Let stand for up to five minutes.

8.1.5.2.3 Read the temperature and record in the lower right hand corner of the COC or where noted on the Chain-of-Custody. If the temperature is not within 4° ± 2°C (2° to 6°C), proceed as in **section 8.1.4** above. For CLP projects, if the temperature

exceeds 10°C, the project manager will contact SMO for corrective action.

8.1.5.3 A calibration check of the thermometer is to be done quarterly or as the thermometer is replaced by comparing it's reading to the NIST thermometer and recording the results in the QA/QC Thermometer Calibration Logbook. See **SOP No. 110.0006** for the calibration procedure.

8.1.5.4 The presence/absence of a temperature blank in the shipment is documented on the Form DC-1. Follow the instructions on the DC-1 Form for contacting SMO, and for documentation in the event a temperature blank is not present.

8.1.6 If the Sample Custodian is not present, the person receiving the samples should perform all steps through **Section 8.1.5.2.5**, then store the cooler in the walk-in refrigerator, R1, until the Sample Custodian returns, after removing any VOC samples. Samples for VOC analysis require special handling, as described in **Section 8.2.2**. In general, if not logged-in immediately, VOA vials must be removed from the cooler and stored in refrigerator R2 until logged-in to the LIMS. If VOC samples are received in Encore devices (typically contained in silver foil pouches), or are noted as requiring freezing to preserve, they must be removed and stored in the freezer F2. Encore samples must be extruded into pre-weighed VOC vials as soon as possible. Contact one of the following to insure this occurs: VOA Laboratory Supervisor, Sample Custodian, Project Manager, Laboratory Operations Manager or QA Director. Leave the paperwork in the red folder on the sample custodian's desk.

8.1.7 Once all of the coolers received have been inspected as above, prioritize them for logging into the LIMS with fastest turn-around times and short holding times processed first. The labs should be notified immediately about quick-turn analyses. This can be done either verbally, or by copying the COC and distributing that to the laboratories.

8.1.8 Remove and inspect samples from the coolers for the first workorder. Sample containers are removed from the cooler and lined-up on the counter in the same order as listed on the chain of custody form. Check to see that the correct number of samples is present. Check to see if any samples are broken. Check aqueous VOA sample vials for the presence of bubbles larger than the size of a pea. If bubbles are larger than the size of a pea, a Sample Notification form should be filled out and given to the Project Manager. Check to see that the quantity present is sufficient for the test requested. Make sure the client IDs on the bottles agree with the COC. Check the custody seals, if any, on the sample containers to ensure the all seals are present and intact.

8.1.9 For U.S. EPA samples, sign the COC and note the temperature on the bottom right corner of the COC. Place the project number in the top right hand corner of the COC. Record the following information on the Form DC-1.

- Sign DC-1. Note that the person signing should be the person who signed for receipt on the COC. There will be one DC-1 form for each cooler received for a particular case. Only the samples in that particular cooler will be listed on that DC-1 form.
- EPA Case number from the COC.
- List sample tag control numbers and compare these with the COC records.
- Document whether or not these numbers agree.
- If Sample tag numbers are not listed on the COC form, record this fact.
- Record the custody seal numbers if present.
- Write the work order number on the air bill and place it in the red project folder. Record the name of the courier and the air bill number on Form DC-1.
- Date of receipt.
- Time of receipt.
- EPA sample numbers.
- SDG-Final Sample must be documented on EPA TR/COC.
- SDG (Sample Delivery Group) number. The SDG is the lowest alphanumeric sample on the first shipment and remains the same until the work order is closed.
- EPA sample numbers.
- Assigned Lab ID numbers. Note that with EPA projects, shipments may be received over several days to be added to the same work order, up to twenty field samples, not including PE samples. The SDG number stays the same, but the sample ID numbers run consecutively.
- Whether samples were delivered by hand or not.
- Any problems and discrepancies in "remarks" box.
- The cooler temperature is entered in item #10 of the Form DC-1.

Note from the COC whether the work order is closed, or if more samples are expected to be added (work order/SDG is still open). EPA projects are closed automatically after one week from the date of receipt, for cases with a turnaround time of 14 or 21 days and three days for samples with a turnaround time of 7 days, of the first shipment or sooner if the client checks the box that the project is closed.

Discrepancies in EPA shipments should be reported to the Project Manager directly. Any irregularities in the above information should be documented and the client contacted by either Project Manager or the Sample Custodian. Until an answer is received from the client, samples should be stored in the walk-in cooler.

If there are any discrepancies, note them on the Sample Condition Notification Form and give the form and the workorder folder to the project manager. Samples with

issues will not be processed further, but stored in the walk-in cooler until a resolution has been reached with the client. For CLP (EPA) samples, the Sample Management Office (SMO) will be contacted for corrective action. Communication of the corrective action will be placed in the red project folder.

8.1.10 Log the workorder and samples into the Omega Laboratory Information Management System (LIMS) following procedures described in SOP 20.0003. Labels are generated and attached to each sample container. In the case of pre-preserved samples in VOA vials (sodium bisulfate, DI water and/or methanol), be sure the Mitkem sample ID label does not cover the tare weight of the vials.

8.1.11 Have the project manager or a second person check the Lab IDs on the labels vs. the client IDs on the COC to confirm the bottles have been properly labeled. Ensure the reviewer initials the proper line of the Sample Condition Form or DC-1 form.

8.1.12 Enter the workorder number, date received, and client name in the Sample Receipt and Tracking Logbook (**Attachment 6**). Occasionally, a client wishes for a project number to be ongoing for several shipments of samples (i.e. EPA). In such cases the date of arrival of each subsequent shipment should be noted for the original work order number.

8.1.13 Inorganic preserved samples must now be tested for pH as follows:

For metals, wet chem, etc., pour a small amount of sample over a strip of pH paper that is suspended over the mouth of a waste container. The pH of the acidified samples are taken by using the narrow range pH strips which measures pH 0 to 2.5. The sample pH should be less than two, as indicated by the color on the pH indicator package. The pH of the basic samples are taken by using the narrow range pH strips which measures pH 11.0 – 13.0. The basic samples should have a pH 12 or greater, as indicated by the color of the pH indicator package. (See **Attachment 4** for instructions on the use of the pH paper.) Acidified samples must have a pH of less than 2. Any pH adjustments that need to be made are recorded on the Sample Condition Form for the workorder. Record the lot number and ID of the reagent used and the approximate volume used to adjust the pH. For metals samples requiring acidification, alert the inorganic laboratory to allow 24 hours before metals preparation to allow enough time for the acids to dissolve any metals that adsorb to container walls.

8.1.14 Some samples (i.e. fecal coliform bacteria) which require subcontracting must be done so immediately as the analysis has an extremely short hold time. A list of analyses typically subcontracted can be printed from the LIMS Omega system. An Outside Services Purchase Order is also generated from the LIMS system when the batch of samples to subcontract is created.

8.1.15 Receiving personnel make one copy of the sub contract COC. This COC accompanies the samples and is signed by the laboratory that will perform the analysis. A copy of the COC is placed into the red workorder folder and one copy is sent to Accounting.

8.2 Storage and Location of Samples:

8.2.1 Semivolatile Organic, Pesticides/PCBs, and Wet Chemistry Samples:

Samples are stored in the main walk-in refrigerator (R1) in the sample receipt room at 4° ± 2°C. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook . The person putting the samples into the walk-in must put their initials in the logbook beside the location entries.

8.2.2 Volatile Organic Samples:

Samples for VOA are stored separately from all other samples, in refrigerators or freezers dedicated for this purpose. These refrigerators and freezers are located in the VOA Laboratory. A soil sample for VOA may contain one or more of the sample types listed below. Care must be taken to insure each sample vial type receives the proper handling. Soil samples for volatiles analysis must have special handling performed by the VOA lab staff.

A storage blank must accompany all VOA samples requiring EPA/CLP or New York State ASP analyses when placed in the VOA refrigerators. The storage blanks are prepared in the volatiles department using the VOA Type I water. The 40ml vial is filled so there is **no** headspace, capped immediately and tightly, and labeled with the date prepared and any preservative added. The storage blanks for unpreserved low/medium soil samples are prepared by placing approximately 5 g of inert sand in dry closed-system purge-and-trap vials. These inert sand storage blanks will be stored in the same freezer with unpreserved low/medium soil samples.

8.2.2.1 Samples suspected of containing high concentrations of volatile organics must be stored in a separate VOA refrigerator designated for such samples. This isolates these high concentration samples from other samples, and helps to control cross-contamination.

8.2.2.2 Soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial) should be stored in a VOA freezer (containing no standards or sample extracts). These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.3 Soil samples that contain up to 5 mL of water. These vials are also to be stored in a VOA freezer. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.4 Soil samples that contain 5mL of water containing a sodium bisulfate preservative. These vials are also to be held in a VOA freezer (specifically for EPA CLP samples). Samples for Method 8260 analysis may be held in a VOA refrigerator. These vials are to be weighed by VOA lab staff prior to analysis.

8.2.2.5 Soil samples received in the Encores (typically contained in silver foil pouches), the samples must be extruded into tared, dry closed-system purge-and-trap vials by the VOA lab staff, then re-weighed to obtain the final sample weight. This should happen immediately (for EPA CLP samples), but no longer than 48hours of sample collection. The vials are then to be placed into a VOA freezer.

8.2.2.6 Soil samples may also arrive as methanol preserved samples. These samples do not require freezing, but they shall be held in a VOA refrigerator to be weighed by VOA lab staff prior to analysis. If possible, check that the methanol covers the soil samples within the vial. If the methanol is insufficient, note this on the Sample Condition Form and relay the information to the VOA lab.

8.2.2.7 Unpreserved VOA soil samples should be stored in a VOA freezer (EPA CLP samples) or a VOA refrigerator (for Method 8260 samples) until the time of analysis.

8.2.2.8 Water samples are stored in VOA refrigerators. Trace Volatile samples for the EPA/CLP SOM01.2 contract must be stored in a separate refrigerator from all other samples. At present, this has been designated as **R13 and R4**.

8.2.2.9 Log the samples into the VOA lab. Enter the workorder and other pertinent information in the Volatile Receiving Logbook (See **Attachment** 7) and sign off that the samples have been relinquished. If the samples are ever removed from VOA they must be signed out of the VOA lab as well.

8.2.3 Metals Samples:

Aqueous preserved samples for metals analyses are stored on the rack in the sample receipt area. Once the samples have been placed on a shelf, or shelves, record the location(s) in the Sample Receipt and Tracking Logbook . The

person putting the samples onto the rack must put their initials in the logbook beside the location entries.

Standards must be stored in separate freezers or refrigerators isolated from samples and sample extracts.

8.3 Storage and Location of Extracts:

Extracts for semivolatile analyses must be stored, shielded from light, at $4^{o} \pm 2^{o}C$ in a separate refrigerator stored either in the laboratory or in Unit 3. Extracts for Pest/PCB are stored in refrigerators in the same manner.

Extracts must be stored separately from standards.

Extracts for semivolatiles and Pest/PCB must be stored until the proper time to dispose of them per client approval. Mitkem normally retains sample extracts for a period of three months from the date of delivery of the final report to the client

US EPA CLP and New York State ASP extracts must be stored for 365 days after delivery of a complete data package to the client.

8.4 Temperature Monitoring:

The temperature of the storage areas must be monitored on a daily basis, including weekends and holidays. This temperature must be entered in the Temperature Logbook. If any refrigerator fails, first investigate the problem. Was the door ajar? Is something preventing the door from shutting fully? If the situation can be rectified, do so and record the temperature again after a sufficient period of time. If the deviation occurred at the end of the day, re-record the temp as soon as possible the following morning. When the temperature meets criteria, document the issue and note that the refrigerator/freezer has been "*returned to control*". If there is no obvious issue with the refrigerator/freezer, or the refrigerator does not maintain the correct temperature, the QA Director or Lab Director must be notified immediately to correct the problem. Fill out a report in the LIMS Omega Corrective Action Report option. Acceptable temperature ranges are $2^{o} - 6^{o}C$ for refrigerators and $-10^{o}C$ to $-20^{o}C$ for freezers. The Sample Receiving supervisor or his/her designee will check and record the storage area temperatures daily.

8.5 Tracking of Samples and Extracts:

8.5.1 Samples:

8.5.1.1 Sample Log-out:

When samples are taken from their storage areas for extraction, they must be signed out using the appropriate Sample Receipt Logbook. The log-out procedure is a two-person procedure that requires samples to be relinquished by either the Sample Custodian or Authorized Laboratory Personnel

The analyst retrieves the samples from the walk-in cooler and the Custodian or Authorized Laboratory Personnel checks the sample numbers. The Mitkem sample numbers as well as the bottle # are recorded (i.e. E0140-02B#2). Both people initial and date the Receiving Logbook in the appropriate places. When the sample is logged-out by an analyst, it is considered to be in the custody of that analyst until he/she returns the sample to its storage area.

Upon the sample's return to the storage area the samples are signed into Receiving using the same procedure. This time the analyst relinquishes the samples and the Custodian or Authorized Laboratory Personnel checks the sample numbers to be returned. Each person signs and dates in the appropriate places in the Receiving Logbook.

Authorized laboratory personnel are employees who are knowledgeable in regard to the receipt and log-in of samples. A list of Authorized Laboratory Personnel is posted on the Receiving bulletin board. (See **Attachment 5**).

8.5.1.2 Sample Extraction:

If a sample is signed out for extraction, the sample is logged into the lab by entering the Lab ID into the appropriate extraction logbook. After extraction, any remaining sample volume is returned to the storage area and signed back into the Receiving area for storage as noted in **Section 8.5.1.1**.

When working with samples in the preparation laboratories, the following procedures must be adhered to:

- All activities performed on the sample must be recorded.
- Entries in the logbooks must be signed and dated (mm/dd/yy).
- All entries must be made in ink.
- Corrections and additions must be made by drawing a single line through the errors, entering the correct information, and initializing and dating the new information.
- Unused portions of the logbooks must be lined-out or "Z-ed" out.
- Logbook entries must be made in chronological order.
- For US EPA/CLP samples, entries are recorded for only one SDG on a page, except in the event where SDGs share quality control samples.
- Information inserted in the logbooks must be permanently affixed, signed, and dated across the insert.
- Copies of all pertinent logbook pages must be placed in the workorder file.

° Proper subsampling techniques can be located in the Subsampling SOP, 110.0039.

8.5.1.3 Extracts/Digestates:

The organic sample extracts, once completed by the preparation laboratory, are transferred to the appropriate instrumentation lab and signed into the extract transfer logbook. The analyst will sign for the extracts and enter storage location information. Inorganic metals and mercury digestates are transferred to the instrumentation lab. The analyst signs off on the preparation logbook at time of transfer.

When the time period for organic extract storage has expired, extracts may be disposed of in the same manner as would used/expired standards. CLP extracts must be stored for one year. The transfer and disposal for these extracts must be documented in the Extract Disposal Logbook.

When the time period for inorganic digestate storage has expired, digestates are disposed in the same manner as would used/expired inorganic standards.

See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the actual disposal of above extracts/digestates.

8.5.1.4 Sample Disposal:

The unused portion of sample is returned to the Receiving area for long term storage. Such portions must be stored for a certain length of time as required by client contract and then properly disposed. Unused sample is disposed with the use a licensed hazardous waste contractor, currently Univar. Routinely, one of the sample custodians runs the LIMS Omega Disposal program to produce a list of samples ready for disposal. See Mitkem SOP No. 30.0024 Sample and Waste Disposal for details on the different wastestreams and disposal options.

All soil samples received from outside of the 48 contiguous United States must be disposed of following special procedures outlined by the USDA, as detailed in Mitkem SOP No. 30.0024 Sample and Waste Disposal section 8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone.

9. Data Reduction and Calculations

Not applicable.

10. Quality Assurance/Quality Control

10.1 The Sample Custodian will calibrate the temperature gun using an NIST calibrated thermometer on a quarterly basis. The correction factor will be noted on the temperature gun and recorded in the NIST Thermometer Calibration Logbook. The serial number of the NIST thermometer will also be recorded in the logbook.

10.2 On a daily basis, the Project Managers will review that the Sample Condition Forms are filled out correctly.

10.3 Twice daily (typically early morning and mid-day) the temperatures of the refrigerators and freezers will be checked and the temperatures recorded. Deviations will be noted in the logbook. If after 36 hours the temperature problem cannot be corrected with manual adjustments, the refrigerator or freezer will be replaced.

10.4 The Hazardous Waste Inspection Logbook will be completed weekly, after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11. Data Validation and Reporting

A copy of the chain of custody and Sample Condition Notification form accompanies the data reports to the client. Also any communication records are to be included in the data package (telephone conversations, e-mail communication, etc.).

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range. Notification reports (orange sheets) are generated in the event of an out-of-control situation occurs at sample log-in that cannot be corrected. Project Managers contact the client in order to ensure what type, if any, corrective action needs to be taken. Other corrective actions concerning the Receiving Area are recorded using the Omega LIMS Corrective Action Report.

13. Health and Safety

The sample custodian must wear a lab coat, safety glasses, and gloves when handling samples and preservative chemicals. Samples coolers should be opened under a hood. All samples should be handled as though they are potentially hazardous. The outside of sample containers should also be handled as though they are contaminated. Any broken sample containers must be handled very carefully to prevent exposure to the sample.

14. Pollution Prevention, Waste Management, and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

US EPA CLP SOW SOM01.2
US EPA CLP SOW ILM04.3
US EPA CLP SOW ILM05.4
US ACOE Shell for Chemical Analytical Requirements, Appendix H

Attachments:

1. **Attachment 1**: Mitkem Sample Condition Form.
2. **Attachment 2**: EPA Sample Condition Form (DC-1).
3. **Attachment 3**: Sample Condition Notification Form.
4. **Attachment 4**: Instructions for the use of pH paper.
5. **Attachment 5**: Example Authorized Personnel list.
6. **Attachment 6**: Sample Receipt and Tracking Logbook.
7. **Attachment 7**: VOA Receiving Logbook

Attachment 1
Mitkem Sample Condition Form

MITKEM LABORATORIES
Sample Condition Form

Received By:	Reviewed By:	Date:	MITKEM Workorder #:
Client Project:		Client:	

		Lab Sample ID	Preservation (pH) HNO_3	H_2SO_4	HCl	NaOH	H_3PO_4	VOA Matrix	Soil Headspace or Air Bubbles ≥ 1/4"
1) Cooler Sealed	Yes / No								
2) Custody Seal(s)	Present / Absent								
	Coolers / Bottles								
	Intact / Broken								
3) Custody Seal Number(s)									
4) Chain-of-Custody	Present / Absent								
5) Cooler Temperature									
Coolant Condition									
6) Airbill(s)	Present / Absent								
Airbill Number(s)									
7) Sample Bottles	Intact/Broken/Leaking								
8) Date Received									
9) Time Received									

Preservative Name/Lot No:

VOA Matrix Key:

US = Unpreserved Soil	**A** = Air
UA = Unpreserved Aqu.	**H** = HCl
M= MeOH	**E** = Encore
N = $NaHSO_4$	**F** = Freeze

See Sample Condition Notification/Corrective Action Form yes / no

Rad OK yes/ no

Attachment 2

EPA Sample Condition Form (DC-1)

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name				Page 21 of 32
Received By (Print Name)				Log-in Date
Received By (Signature)				
Case Number		Sample Delivery Group No.		Mod. Ref. No.

Remarks:			Corresponding		
		EPA Sample #	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists	Present/Absent*				
4. Airbill	Airbill/Sticker Present/Absent*				
5. Airbill No.	_____ _____				
6. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
7. Sample Condition	Intact/Broken*/ Leaking				
8. Cooler Temperature Indicator Bottle	Present/Absent				
9. Cooler Temperature	_____				
10. Does information on TR/COCs and sample tags agree?	Yes/No*				
11. Date Received at Laboratory	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution.

Reviewed By	Logbook No.
Date	Logbook Page No.

SAMPLE LOG-IN SHEET

Lab Name	Page 22 of 32
Received By (Print Name)	Log-in Date
Received By (Signature)	

Case Number	Sample Delivery Group No.	SAS Number

Remarks:		EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab #	Remarks: Condition of Sample Shipment, etc.
1. Custody Seal(s)	Present/Absent* Intact/Broken				
2. Custody Seal Nos.	_____ _____				
3. Chain of Custody Records	Present/Absent*				
4. Traffic Reports or Packing Lists	Present/Absent*				
5. Airbill	Airbill/Sticker Present/Absent*				
6. Airbill No.	_____ _____				
7. Sample Tags	Present/Absent*				
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody				
8. Sample Condition	Intact/Broken*/ Leaking				
9. Cooler Temperature	_____				
10. Does information on custody records, traffic reports, and sample tags agree?	Yes/No*				
11. Date Received at Lab	_____				
12. Time Received	_____				
Sample Transfer					
Fraction	Fraction				
Area #	Area #				
By	By				
On	On				

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

Attachment 3
Sample Condition Notification Form

Sample Condition Notification

Mitkem Project#:_____ **Date of Receipt:**_____
Client:_____ **Received By:**_____
Client project #/name:_____

Unusual Occurance Description:

_____

_____

_____

_____

_____

_____

Client Contacted:

Contacted via: Phone/Fax/E-mail
Date:_____ **Time:**_____
Contacted By:_____
Name of person contacted:_____

Client Response:

Responded via: Phone/Fax/E-mail
Date:_____
Name of person responding:_____
Responding to:_____

_____

_____

_____

_____

_____

Mitkem Action Taken: _____

_____

_____

_____

_____

Attachment 4

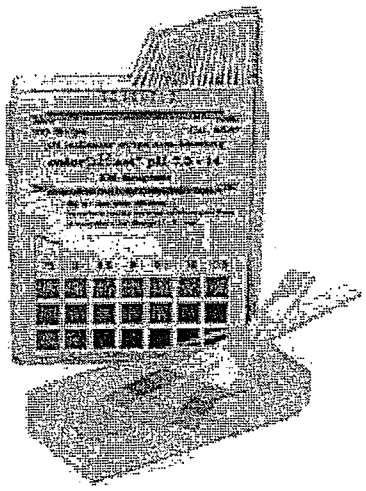
Instructions for use of pH paper

General Procedure for the Use of colorpHast® pH Test Strips

1. Remove a strip from the box.
2. Immerse the reaction zone of the strip into the solution for approximately 30 seconds to 1 minute, allowing enough time for the reaction to take place.
3. Remove the strip from the solution, wiping the strip along the edge of the vessel to remove excess liquid from the strip.
4. Compare the strip's reaction zones to the color chart on the box while holding the strip to the top of the box and the reaction zones to the bottom of the box.
5. Record the results of the closest matching color to the strip.

- *For additional application or product information, ordering information or other technical questions, please call our Technical Service Department at:*

  1-800-222-0342 *or* **1-856-423-6300**



EMD Chemicals Inc.
480 South Democrat Road
Gibbstown, NJ 08027

Phone: 856-423-6300
Fax: 856-423-4389
www.emdchemicals.com

Attachment 5

Example Authorized Personnel list

Sample log-in/out: **Authorized Personnel**		
Name	**Dept**	**Initials**
Anderson, Courtney	Inorganics	*CJA*
Badura, Karolina	PEST/PCB	*KB*
Chiu, Kin	Mitkem	*KC*
Cardoso, Antonio	Oprep	*AC*
Ding, Yihai	Mitkem	*YD*
Ding, Xiangdong	VOA	*XD*
Gaudreau, Veronica	Receiving	*VEG*
Jaffer, Nathan	Inorganics	*NJ*
Healey, Josh	Receiving	*JH*
Kaczorowski, Przemyslaw	Oprep	*PK*
Lawler, Andrew	Oprep	*AEL*
Lawler, Edward	Project Management	*EL*
Lawler, Sharyn	QA/QC	*SBL*
Leveillee, Roy	Oprep	*ROY*
Luo, Wei	VOA	*WL*
Lucas, Derek	PEST/PCB	*DL*
Maczewska, Beata	SVOA	*BM*
Marquis, Ashley	VOA	*ALM*
McGorty, Cassandra	Inorganics	*CJM*
McGovern, Kerri	Inorganics	*KLM*
Mosher, Cassie	SVOA	*CLM*
Montmarquet, Timothy	Mitkem	*TM*
Nadeau, Cory	Receiving	*CAN*
Ng, Agnes	Project Management	*ARN*
Ng, Shirley	Project Management	*SN*
Rosadzinski, Tomasz	Oprep	*TR*
Sawyer, Tom	Inorganics	*TS*
Smart, Dawne	Inorganics	*DES*
Wetherbee, John	Inorganics	*JRW*
Wortley, Stephen	SVOA	*SW*
Veilleux, Brian	Inorganics/Oprep	*BV*
Wieczorek, Agnieszka	PEST/PCB	*AW*
Zharkova, Sofya	PEST/PCB	*SZ*
Zhao-Anderson, Heather	VOA	*HZA*

Most common personnel using log-in/out

Attachment 6

Sample Receiving and Tracking Logbook

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc.

Sample Receiving Logbook

Workorder No. _____

Client Name: _____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

Date Recv'd_____ **Sample #s**_____ **Storage Locations:**_____

OUT		IN	
Relinquished By	**Received By**	**Relinquished By**	**Received By**
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			
Date: Init:	Date: Init:	Date: Init:	Date: Init:
Samp. #s			

Comments: _____

_____

Please record analyst's initials, date, and sample #s removed. Add any comments if necessary (broken bottles, empty jars, etc.)
Include the abbreviated name of the test to be performed., ie: SVOA, PCB...near the "samp. #s".
Include bottle or jar number when more than one.

Reviewed:_____

Logbook ID: 30.0287-09/08

Attachment 7

Volatiles Receiving Logbook

MITKEM LABORATORIES: VOLATILE SAMPLES RECEIVING LOGBOOK

VOA Log-In Date	Workorder	Client ID	Sample Numbers	Relinquished by:	Received by:	Pres. Used	F/R	Returned to R1

Logbook ID 90.0191-06/08

Reviewed By:

"Preservative Used" Key

UA = Unpreserved Aqueous	**H** = HCL	**A** = Air	**M** = MeOH	**E** = *Encore
US = Unpreserved Soil	**N** = $NaHSO_4$		**F** = Freeze	**T** = Trace, HCL

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP No. 30.0024

Rev. 8

	Signature	Date
QA Director:		10/17/08
Lab Director:		10/21/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Sample and Waste Disposal

SOP 30.0024 Rev. 8

1. Scope and Application

This Standard Operating Procedure (SOP) outlines the procedures for the disposal of the unused portion of samples submitted for analysis, as well as solid and aqueous wastes and sample extracts to minimize the amount of waste in the lab and organize the manner in which it is handled.

2. Personnel Qualifications and Responsibilities

Personnel handling samples must be aware of the potential dangers posed by the samples, and handle them properly to minimize this danger. Personnel handling samples must also maintain the documented chain of custody of the samples from the time they are received at Mitkem until they become laboratory waste. Laboratory waste must be handled in a safe manner to minimize any exposure to this material.

3. Summary of Procedure

Waste stream manifest profiles have been identified by Mitkem and the waste-disposal company. These profiles dictate how to dispose of each individual waste, as described in this SOP. If any question arises regarding the disposal or classification of waste, the Waste Disposal Technician, or senior laboratory management should be consulted.

Once sample analyses are complete and a final report has been issued to the customer, samples are stored for a specified length of time before disposal. This timeframe is usually dictated by the client. Strict chain of custody documentation is maintained for these samples until samples are disposed into one of several waste streams. This waste is removed from Mitkem by a licensed waste-hauler for final disposal.

Safety equipment will be worn prior to and during any waste disposal. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

4. Sample Preservation, Containers, Handling and Storage

Not applicable.

5. Interferences and Potential Problems

Not applicable.

6. Equipment and Apparatus

Waste drums. Polyethylene drums are used for liquids (acids or basic/spent CN solutions) and steel open top drums are used for waste solid materials.

Burp-free Ultra Funnel, various sizes, polyethylene funnel used to allow safe pouring of liquid wastes into drums. Product is vented and has locking lid.

Spill kits

Drum Pump

Drum Lift

Satellite Nalgene storage containers with lidded funnels for interim liquid waste storage in Wet Chem, Volatiles and Metals Labs.

Satellite storage containers (red metal Justrite Oily waste Can ,10G capacity) for interim solids storage in Organic Prep, Semivolatiles and Volatiles Labs.

Satellite storage container (Justrite Safety Disposal Can, 2G capacity) for solvent waste in Volatile Lab.

7. Reagents

Not applicable.

8. Procedure

8.1 Classification of Waste:

Laboratory waste products fall under one of the following categories:

8.1.1 Methylene Chloride, Methanol, Water:

Waste solvents are emptied into a solvent drum located in the organic sample preparation laboratory. This waste consists primarily of methylene

chloride with lesser portions of methanol and water. Trace amounts of other solvents may also be included from time to time. Due to the high proportion of methylene chloride in the waste, the material is flammable, and is classified as D001-type waste. Whenever a new drum is started, a Hazardous Waste label is attached to the outside.

When the drums are full the waste drum is taken to the Hazardous Waste storage shed. An accumulation date is added to the Hazardous Waste label on the drum.

Empty solvent bottles are evaporated to dryness and discarded into the general solid waste dumpster (garbage). Glass containers that present a risk of injury should be discarded into a broken glass container for disposal.

Any lab apparatus, gloves, paper towels, etc. exposed to methylene chloride must be discarded into the red solid waste drums in the Organic Prep Lab. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.2 Acids, Water:

Acid waste consists of approximately 98% water and 2% acid. This waste stream also includes the remainders of spent or expired metals standards, digested samples, TCLP and SPLP leachates and undigested aqueous samples preserved with acid. This waste is emptied into polyethylene acid waste drums in the hazardous waste area. Acidic waste from the Lachat analysis of nitrate/nitrite or chlorides are stored temporarily in Unit 3 in a small waste container equipped with a closing safety funnel. The container is routinely emptied into this same wastestream. A Hazardous Waste label is affixed to the side of the drum and an accumulation date is recorded on it the first time the drum is started. This waste is classified as D002 type waste. The drum must be removed by the Waste Hauler within 90 days of the accumulation date.

8.1.3 Scintillation Vials, Methylene Chloride, Hexane:

This waste stream consists of Organic analysis vials used for GC and GC/MS analyses. Spent or expired organic standards in solvent are also included in this wastestream. The vials are disposed of unopened. Vials are initially disposed in the GC Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.4 Lab Solids, Broken Glass, Methylene Chloride, Acetone:

This waste stream contains the remainders of solid and soil samples. This waste stream also includes laboratory solid waste that has been stored in the red metal drums in the Organic Prep Lab. When full the laboratory red metal containers are taken to the Hazardous Waste shed and the contents dumped into the waste solids drum. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and is later removed by the Waste Hauler within 90 days of the accumulation date. This waste has no RCRA Characterization code.

Any solid matrix samples received from outside of the 48 contiguous United States, or from any other area identified by the client as a US Department of Agriculture Soil Quarantine Area must have special disposal in order to kill any quarantined organism. Samples identified as such, along with their entire container including jar cover, are placed in a disposable aluminum pan and placed in the kiln (in Unit 3) and heated to 400 degrees for 30 minutes. After the pan has cooled, the sample(s) and aluminum pan(s) are disposed in the waste soil/solid drum.See **Attachment 1** for incineration procedures.

8.1.5 Glass Vials, Methanol, Water, Soil:

This waste stream consists of primarily 40-ml organic analysis vials used for VOA analyses. The vials are disposed of unopened. Vials are initially disposed in the VOA Analysis Laboratory in a red metal drum. When this drum is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D001 waste.

8.1.6 Spent Carbon with Methylene Chloride:

This waste stream contains the remainders of Mitkem's discharge. Discharge is run through tanks of carbon material as a means of removing methylene chloride. When the tanks are fully deactivated, the carbon is removed and dumped into a steel drum. The tanks are then filled with activated carbon. A Hazardous Waste label with an accumulation date is affixed to the outside of the drum and the drum is later removed by the Waste Hauler within 90 days of the accumulation date. This waste stream is not associated with any RCRA Characterization code.

8.1.7 Electronic Device with Beryllium Oxide:

This waste stream contains the power tube of an ICP spectrophotometer. Because of beryllium in the tip of the tube, it cannot be disposed of with

ordinary trash. The tube is placed in a sealed cardboard box and a Hazardous Waste label with an accumulation date is affixed to the outside of the box. The box is later removed by the Waste Hauler. This waste stream is not associated with any RCRA Characterization code.

8.1.8 COD Vials:

This waste stream consists of glass tubes that contain a mixture of mercuric sulfate, silver sulfate, sulfuric acid, and chromic acid. The tubes are used for COD analyses and are disposed unopened in their entirety. Vials are initially disposed in the Wet Chemistry Laboratory in a covered cardboard box with a Hazardous Waste Label. When this box is full, the contents are removed to the Hazardous Waste Shed and dumped into a steel drum. A Hazardous Waste label is affixed and the drum removed by the Waste Hauler within 90 days of the accumulation date. This waste is classified as D002, D009, D011 waste.

8.1.9 Lab Pack Waste:

From time to time, waste or unused chemicals (non-solvents) will require disposal. These materials are segregated and placed in a lab pack container. An inventory of materials is maintained for the lab pack drum. The materials are stored in the laboratory in a closed cardboard box with a Hazardous Waste label attached. Consult the waste hauling company when sufficient volume of material is present in the laboratory to initiate a lab pack.

8.1.10 PCB Waste:

Any PCB-containing material (at a concentration of greater than 50 ppm) must be segregated and disposed according to PCB-specific procedures. This material would generally consist of any oil sample containing over 50 ug/g (ppm) of PCB. Samples containing this high concentration of total PCB will be identified by the GC Laboratory Manager or the Laboratory Director. The container for this sample will be prominently labeled "for return to client" when the holding period (as identified in **Section 8.2.1** below) is reached. The Mitkem project manager will be notified, as will the client, with a notation in the report Project Narrative or Cover Letter that the sample will be returned.

8.1.11 Water/Sodium Hydroxide/Pyridine:

This waste stream consists of sulfide and cyanide digestates and spent standards. Pyridine waste from the analysis of Cyanide (Lachat), and basic pH Ion Chromatography (IC1) wastes are stored temporarily in Unit 3 in several small waste containers equipped with closing safety funnels.

The containers are routinely emptied into a polyethylene Basic Waste drum in the hazardous waste area. This waste is classified as D002, D038 waste

8.2 Procedure for Disposal of Sample Remainders:

8.2.1 Determination of Disposal Date:

Samples are held for a period of time following receipt at the laboratory or release of the final report to the client. Mitkem's policy is to hold samples for a specified length of time. On a case-by-case basis, arrangements may be made between the client and Mitkem through discussion with the Mitkem Project Manager. This agreement is then saved in LIMS in the individual Client Project. Default values are used otherwise. To review the Sample Hold Times and Disposal Options on a particular workorder, go to the Report Option at the Workorder Main screen.

For CLP, samples are held for 60 days after delivery of the reconciled data package. Sample extracts must be stored at 4°C until 365 days after delivery of a complete, reconciled data package to EPA.

8.2.2. Documentation of Sample Disposal:

When the specified hold time has passed since the receipt date, the samples can be disposed. Sample receiving personnel select sample remainders from their storage locations and remove these to a designated location outside of the sample receiving area for disposal by Sample Receiving or laboratory personnel. Documentation that sample remainders have been disposed is maintained in the Mitkem Omega LIMS. When samples are removed from storage for disposal the person removing them enters the disposal date and their initials into LIMS.

8.2.3. Final Disposal and Record keeping:

All samples are to be disposed of according to **Section 8.1**.

Once waste drums are full, approximately bi-monthly, the waste hauling company is called to schedule a pick-up. Mitkem currently uses Univar to arrange for disposal of laboratory waste. Univar will remove the waste from the Mitkem storage area, and arrange with Pollution Control Industries (PCI) in Michigan for ultimate disposal of the waste material. Waste manifests are initiated by the hauler, signed by the QA Director or his designee when drums are loaded on the hauler's truck, signed and mailed back to Mitkem following final disposal. Manifests are filed

according to date in the QA office. Mitkem's Generator Number is **RI5000009621**.

9 Data Reduction and Calculations

Not applicable.

10 Quality Assurance/Quality Control

The Hazardous Waste Inspection Logbook will be completed weekly after an inspection of the Hazardous Waste shed by a Sample Custodian. Major problems will be reported to the Hazardous Waste Coordinator.

11 Data Validation and Reporting

After sample disposal, the disposal dates are entered into the LIMS for each workorder. In addition, the samples'storage location is changed to "disposed".

12 Corrective Action Procedures

Corrective actions concerning sample or waste disposal are recorded using the Omega LIMS Corrective Action Report under the Receiveing Department or another lab area involved in the issue. Corrective actions should implemented in the event of an out-of-control situation involving sample or waste disposal that cannot be easily corrected. The sample custodian should contact the Safety team, Hazardous Waste Coordinator or Lab Director for help determining what corrective action needs to be taken.

13 Health and Safety

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the Mitkem Safety Manual. Bottle or container labels also provide important information that must be noted. Proper gloves, safety glasses and lab coats or protective outerwear must be worn during all waste disposal activities.

All laboratory employees who either sign a Hazardous Waste manifest or handle hazardous waste directly must attend an annual RCRA training class. At this time Summit Technologies of New Hampshire provides the necessary on-site training for Mitkem's laboratory personnel.

14 Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15 References

None.

Attachments:

1. **Attachment 1**: Instructions for the Kiln.

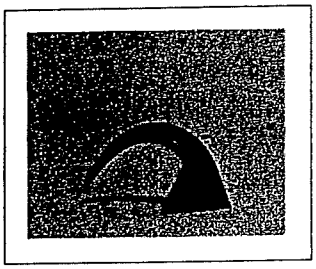
Attachment 1

Instructions for the Kiln

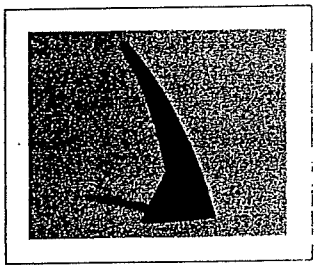
To use Cone-Fire Tuning, **IdLE** must display. Press **ENTER**, then **1. ConE** will appear. Press **999, ENTER. rSLt** (for result) will display alternating with the current Tuning number. Enter the new number, then press **ENTER. ConE** will appear. Continue entering the values for the Cone-Fire program in the usual way. Once you change the Tuning number, Cone-Fire will remain adjusted to that number until you change it again.

The large cone on the kiln shelf should be visible through a peephole. Avoid exposure to cool air by keeping the cone at least 3" away from the peephole. Program the DTC 800C for the cone on the shelf and fire. After cooling, check the cone:

The cone bent to 6 o'clock: In this case, the controller is matched to your kiln.

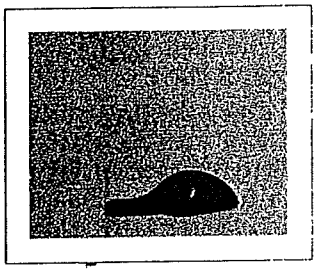


The cone did not bend far enough: Use a lower Tuning number for the next firing.



The cone bent too far: Use a higher Tuning number for the next firing.

Do not be overly concerned with achieving an exact 6 o'clock bend. The difference between a 3 o'clock and a 6 o'clock bend is only a few degrees. Cones, even from the same box, also vary slightly.



Ramp-Hold Mode

Ramp-Hold is available on both the DTC 800C and DTC 800.

In Ramp-Hold, the kiln fires in segments, or stages. Each segment has a firing rate, firing temperature, and hold (or soak) time. Ramp-Hold includes eight segments. Use only the number of segments you need per firing, from one to eight.

Rate is degrees of temperature change per hour, from 1° to 9999°F. (e.g., to increase temperature by 500° per hour, enter a rate of 500).

To control cooling, set the segment to a lower temperature than that of the preceding segment.

Storing User Programs in Ramp-Hold

A user program is a set of firing instructions to run the kiln in Ramp-Hold. A user program includes number of segments needed; firing rate, temperature, and hold (if any) for each segment; and an alarm temperature (if any).

Ramp-Hold Programming

As the program prompts you for segments, rate, temperature, etc., you will see values from the last firing. To use these again, just touch **ENTER**.

To fire without the Delay feature: Follow steps 1 through 9. Then press **START** twice.

1 Turn controller safety switch ON.

2 If controller displays **ErrP** or a flashing temperature, touch **ENTER. IdLE** will appear.

3 Touch **ENTER** then **4. USEr** will appear. Enter a number from 1 to 6 for the stored program desired. ENTER 1

4 Touch **ENTER. SEGS** will appear. Enter number of segments you will use. ENTER 1.

5 Touch **ENTER. rA 1** will appear. Enter firing rate for segment 1 (temperature change per hour: any temperature from 1° to 9999° F.). ENTER 400°C.

6 Touch **ENTER. °F 1** will appear. Enter the temperature you will be firing to in segment 1. ENTER 250°C

7 Touch **ENTER. HLd1** will appear. Enter segment 1 hold (soak) time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). ENTER 00.20

8 Touch **ENTER.** Continue entering values for all segments.

9 Touch **ENTER. AlAr** will appear. Enter alarm temperature. (Enter 9999 to turn alarm off.) Then touch **ENTER.**

IdLE will appear.

10 To set Delay Fire, touch **ENTER** then **3. dELA** will appear. Enter delay time in hours and minutes (e.g. 12 hours and 30 minutes = 12.30). Then touch **ENTER.** (Delay zeroes out after each completed firing.) NO DELAY

11 To start program, touch ENTER twice. **-On-** will appear, then kiln temperature. If a delay was programmed, **-On-** will appear, then time remaining until start.

To stop the program during the firing cycle, touch **STOP** or turn the safety switch OFF. When program fires to completion, **CPLt** will appear alternating with total firing time in hours and minutes. Turn safety switch OFF when **CPLt** appears. To shut off the alarm when it sounds during a firing, press **ENTER.** When Temp decreases after the 20 minutes @ 250°C, hit STOP. LET COOL DOWN ON ITS OWN.

Ramp-Hold can store up to six user programs even when the kiln is unplugged. Potters may want to store a program for crystalline glaze, another for a favorite red glaze, etc. Glass artists might want to store several programs for fusing their favorite types of glass and another for slumping.

When you enter Ramp-Hold mode, the first prompt to appear is **USEr.** The **USEr** prompt means, "Select a stored user program for this firing". If you are using Ramp-Hold for the first time, press 1. Then enter num-

ADDENDUM TO COMPLIANCE AGREEMENT WITH MITKEM CORPORATION
DECEMBER 18, 1996
CONTINUED

7. "USDA" soil samples may not be shipped to other facilities unless such facility has a valid permit and compliance agreement for imported soil or a valid compliance agreement for domestic soil. (See Item No. 3 for a list of such facilities)

8. "USDA" soil samples may not be used as a growing medium for plants and for the isolation and/or culture of organisms imported with the soil.

9. It is further agreed that the PPQ Officer may conduct an inspection of the laboratory facility and records concerning soil shipments at any time during normal working hours. All reasonable recommendations of the PPQ Officer to eliminate the spread of plant pests will be followed. Failure to comply with any of the stipulations of this Compliance Agreement may result in its revocation and the facility's authority to handle foreign and domestic regulated soil.

ALL SOIL RESIDUES, THEIR STORAGE CONTAINERS, FILTERS, ETC. SHALL BE TREATED WITH ONE OF THE FOLLOWING SCHEDULES:

Used soil storage containers may be treated by incineration.

DRY HEAT: Soil to be spread in layers 0.5 inches in depth to ensure uniform heat penetration.

TEMPERATURE		EXPOSURE PERIOD
110 - 120.5 C	(230 - 249 F)	16 HOURS *
121 - 154 C	(250 - 309 F)	2 HOURS *
154.5 - 192.5 C	(310 - 379 F)	30 MINUTES *
193 - 220 C	(380 - 429 F)	4 MINUTES *
221 - 232 C	(430 - 450 F)	2 MINUTES *

* Do not start counting time until the mass reaches the required temperature.

STEAM HEAT: 250 F at 15 lbs. pressure for 30 minutes.

Preheat laboratory autoclaves. Do not start counting time until pressure reaches 15 lbs.. Restrict soil depth to 2 inches when treating quantities of soil in trays. Restrict each package weight to 5 pounds or less when treating individual packages. Load with adequate spacing. Large commercial steam facilities which operate at pressures up to 60 lbs. psi will permit treatment of greater soil depth.

For large filter & soil — use 250° for 2 hours

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication (Method 3550B)

SOP No. 50.0052
Rev 2

	Signature	Date
QA Director:		1/31/08
Lab Director:		2/4/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Sonication
(Method 3550B)

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples for analysis by EPA SW846 methods SW8081A, SW8082, SW8015 and SW8270C. Discussions include sample extraction, sample cleanup references, sample concentration technique and the preparation of standard spiking solutions for the analysis of Total Petroleum Hydrocarbons (TPH), Deisel Range Organics (DRO), pesticide/PCB or semivolatile organic compounds in soil samples. Highly contaminated soils for MA DEP EPH analyses may also use this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. The sample is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using TurboVap or Kuderna-Danish (KD) apparatus followed by Nitrogen blowdown. In addition, this method can be used for Medium/High Concentration Semivolatile samples. A 1-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted once using ultrasonic extraction. The extract is separated from the sample by gravity filtration. The extract is dried through anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration.

4. Sample Preservation, Containers, Handling, and Storage

4.1. The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection.
- Sample extracts must be analyzed within 40 days of sample extraction.

4.2. Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3. Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1. Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2. Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3. Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1. Ultrasonic Disrupter – pulsing horn type with 375 watts maximum and ¾" standard solid disrupter horn and ⅛" standard tapered microtip probe(medium soils only). Fisher Scientific Sonic Disrupter 550., VWR Scientific Branson Sonifier 450.

6.2. Sonabox – ultrasonic disrupter box designed to reduce exposure to ultrasonic sound.

6.3. Beakers – 400mL

6.4. 500mL Erlenmeyer flask

6.5. Dessicator

6.6. Balance capable of weighing +/- 0.1 gram.

6.7. Glass syringes for delivering spike and surrogate solutions – 0.25mL, 0.5mL, 1.0mL.

6.8. Volumetric flasks for making up surrogate and matrix spike solutions – 50mL, 250mL.

6.9. Aluminum Foil (Industrial Grade)

6.10. Anhydrous sodium sulfate: J. T. Baker, ultra resi analyzed grade, granular for drying the samples and sample extracts. Baked at 400°C for four hours.

7. Reagents

The following chemicals including solvents and standards are extensively used in the lab:

7.1. Methylene chloride: J. T. Baker, ultra resi analyzed, to be used for glassware rinsing and sample extraction.

7.2. Methanol: Fisher, Optima, to be used for rinsing glassware and preparing standards.

7.3. Acetone: J.T. Baker, ultra-resi analyzed, to be used for rinsing glassware.

7.4. 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5. Hexane: Fisher, Optima grade, to be used for solvent exchange of samples.

7.6. H_2O, deionized.

7.7. H_2SO_4, concentrated, Fisher, ACS trace metals.

7.8. Surrogate Standards *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200µg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL (Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9. Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

8. Procedure

8.1. Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1. All **primary standards** received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:

Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2. Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is

prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.8** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1mL of the o-Terphenyl primary standards in **Section 7.8** and diluting to 200 mL using methanol. The working standard contains the following compound:

Compound	Concentration(µg/mL)

5-α Androstane	50
o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3. Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 2.5mL of Base/Neutral Composite Set primary standard in **Section 7.9**, 2.5mL of Acid Composite Mix primary standard in **Section 7.9** and 2.5mL of Composite Mix #3 primary standard in **Section 7.9** and diluting to 100mL using methanol. The resulting concentration will be 50ug/mL for all SW8270 compounds. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2. The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See **Attachment 1** for a list of the individual compounds and their concentrations.

8.1.3.3. The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.5. The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10mL Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX

where:
SI = Semivolatile intermediate standard
yymmdd = date intermediate standard is prepared,
X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1mL of the intermediate standard above, and diluting to 100mL using Methanol. The working LCS/MS Standard contains the following:

Compounds	Concentration(µg/mL)
Diesel fuel	5000

The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.9** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.9** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1.**

A smaller or larger amount may be used to prepare the Lab Control Sample and Matrix Spike Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.2.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2. Sample Extraction Procedure:

8.2.1. Low Level Method: Unless Mitkem has documented history that the samples contain high concentration of target analytes (>10,000µg/Kg for the individual

analyte) all of the semivolatile samples are prepared using the low-level approach.

Before starting:

- The disrupter has a minimum of 375 watts power and must be inspected periodically to ensure that the ¾ inch tip has not experienced excessive wear.
- The disrupter is tuned each day of use. The tuning is documented.
- Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.1.1. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400mL glass beaker. A 30 gram ± 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopla. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.1.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing, however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.1.3. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes. According to the test method being used, add the following spikes to the samples in beakers:

- Use a syringe to add 1.0mL of the surrogate standard to the Blank, LCS, samples and any MS/MSD sample.

- Use a syringe to add 1.0mL of the LCS/MS standard to the LCS, and any MS/MSD sample.

8.2.1.4 Add 100mL of the 1:1 v/v methylene chloride/acetone mixture to the Blank, LCS, samples, and any MS/MSD for Semivolatile or Pesticide/PCB extraction, OR add 100mL of methylene chloride to the Blank, LCS, samples, and any MS/MSD for TPH/DRO extraction.

8.2.1.5 Place the bottom surface of the ¾ inch tip of the disrupter about ½ inch below the surface of the solvent, but above the solid layer.

8.2.1.6 Extract ultrasonically for 3 minutes, with the output control set at full power and the mode switch on Pulse, using a 50% duty cycle.

8.2.1.7 Remove the beaker from the disrupter. Decant the solvent layer into a 500mL Erlenmeyer flask, leaving the solid in the beaker.

8.2.1.8 Extract the sample two more times with 100mL of solvent and a 3 minute sonication time. Decant the solvent aliquots into the 500mL Erlenmeyer flask. Cover the flask with aluminum foil.

8.3. Semivolatile Medium Level Method: (Also refer to **SOP No. 50.0100** , Method SW846 3570 Microscale Solvent Extraction for another soil extraction option using small sample volumes.)

8.3.1. The sample is mixed to ensure sample homogeneity. As representative as possible, weigh about a 1 gram portion of the sample into a pre-weighed 15mL vial. The weight measurement is recorded to the nearest 0.1 gram.

8.3.2. Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. Mix the sample and anhydrous sodium sulfate to achieve a free flowing mixture.

8.3.3. Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.

8.3.4. Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples.

8.3.5. Add 9mL of methylene chloride to the Blank and samples. Add 8mL of methylene chloride to the LCS and any MS/MSD.

8.3.6. Sonicate the sample with the ⅛ inch tapered Microtip for 2 minutes at output control setting at 5 in the continuous mode.

8.3.7. Cap the vial.

8.3.8. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

8.4. The sample extract is now ready for filtration and concentration. These procedures are found in SOP No. 50.0054, Extract Filtration and Concentration.

8.5 After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:

8.5.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and

duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to SOP No. 50.0030 Method 3660B Sulfur Cleanup, for details on activiting copper and using it in a sample extract.

8.5.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to SOP No. 50.0031 Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.5.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP No. 50.0032 Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.5.4. Other cleanup methods may be found in SOP numbers 50.0033(Florisil) and 50.0034 (Silica Gel).

8.6 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.7 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.7.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.7.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the

Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1. Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2. Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- An aliquot of the surrogate standard prepared in **section 8.1.2** is added to the Method Blank.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2. Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2. Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- An aliquot of the surrogate standard prepared in **section 8.1.2** and the lab control standard prepared in **section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike

11. Data Validation and Reporting

11.1. Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries,

balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1. Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2. Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3. LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4. Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan; Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3540C, Ultrasonic Extraction, Revision 3, December 1996.

Attachments:

Attachment 1: Pesticide LCS/MS List
Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Re-extraction Request

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4′-DDD	0.4
4,4′-DDE	0.4
4,4′-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc. ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES

Date:	Analysis:	Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:

Blank ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	MeCL/Acetone 1:1 Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started: Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to Fractionation Date / Analyst	Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments:

Logbook ID: 50.0147-01/08

Surrogate Std. ID:

Matrix Spike/LCS Std. ID:

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour:_____

Drip Rate_____

Water Bath Temp:_____

Reviewed By:_____

Figure 2

Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonic) 3540C (Soxhlet) 3545(PFE) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH Number:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent Lot # Solvent Name	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst RV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst RV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

	PEST Matrix Spike Std ID:	Vol. Spiked:	
	PCB Matrix Spike Std ID:	Vol. Spiked:	
	PEST Surrogate Spike Std ID:	Vol. Spiked:	
	PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane Lot # / L/L Drip Rate:
	PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot# / Soxhlet Cycle/Hour:
	PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot # / Sonicator Tuned? Yes/No

Logbook ID: 50.0149-02/08

Reviewed By:_____

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-07/07

Reviewed By:_____

MITKEM LABORATORIES,

A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Soxhlet (Method 3540C)

SOP No. 50.0053
Rev. 2

	Signature	Date
QA Director:		1/31/08
Lab Director:		2/4/08

MITKEM LABORATORIES,

A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Soxhlet (Method 3540C)

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil and sediment samples for analysis by EPA SW846 methods SW8081A, SW8015, SW8082 and SW8270C. Discussions include sample extraction, sample cleanup references, sample concentration technique and the preparation of standard spiking solutions for the analysis of semivolatile, Total Petroleum Hydrocarbons (TPH), Deisel Range Organics (DRO), or pesticide/PCB organic compounds in soil samples. Soil samples to be analyzed for MA DEP EPH may also be extracted using this method.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 30-gram sample is mixed with anhydrous sodium sulfate to form a free-flowing mixture. This is solvent extracted overnight using soxhlet extraction. The extract is dried through powdered and granular anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Caliper TurboVap or Kuderna-Danish (KD) apparatus followed by nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection

- Sample extracts must be analyzed within 40 days of sample extraction

4.2 Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to **SOP No. 110.0039** for more detail on sub sampling techniques.

4.3 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest.

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this preparation method include:

6.1 Beakers – 400ml.

6.2 Boiling flask – 500ml.

6.3 Soxhlet extractor.

6.4 Condensers, 55/50.

6.5 Heating mantle, 500ml.

6.6 Balance capable of weighing +/- 0.1 gram, Ohaus CT1200.

6.7 Glass syringes for delivering spike and surrogate solutions – 0.25ml, 0.5ml, 1.0ml.

6.8 Volumetric flasks for making up surrogate and matrix spike solutions – 50ml, 250ml.

6.9 Aluminum Foil (Industrial Grade)

6.10 Anhydrous sodium sulfate: J. T. Baker, ultra resi analyzed grade, granular for drying the samples and sample extracts. Baked at 400°C for four hours.

7. Reagents

The following chemicals including solvents and standards are extensively used in the lab:

7.1 Methylene chloride: J. T. Baker, ultra resi analyzed, to be used for glassware rinsing and sample extraction.

7.2 Methanol: Fisher, Optima, to be used for rinsing glassware and preparing standards.

7.3 Acetone: J.T. Baker, ultra-resi analyzed, to be used for rinsing glassware.

7.4 1:1 v/v methylene chloride/acetone mixture, to be use for sample extraction.

7.5 H_2O, deionized.

7.6 H_2SO_4, concentrated, Fisher, ACS trace metals.

7.7 Surrogate Standards: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatiles Full Scan: Restek BN Surrogate mix (Cat. No. 31086)at 5000μg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000μg/mL in Methanol.
- Semivolatiles SIM: Cambridge Benzo(e)pyrene d-12 surrogate (Cat. No. DLM-257-S) at 200μg/mL.
- Deisel Range Organics (DRO)/TPH and MA EPH: 5-α Androstane at 10,000μg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000μg/mL(Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 μg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000μg/mL in Acetone.

7.8 Lab Control Sample and Matrix Spike: *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000μg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at

2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.

- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.
- MA EPH: An independent vendor than that used for the MA EPH calibration standards is used. The LCS consists of the 17 Aromatic Hydrocarbon compounds plus the 14 normal Aliphatic Hydrocarbons at 1000µg/mL in Methylene Chloride.

8. Procedure

8.1 Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes.

The standards are labeled ZPyymmddX,
where:
Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

8.1.2.1. Semivolatile Full Scan: The Semivolatile working surrogate standard is prepared by combining 2.5ml of the Base/Neutral primary standard in **Section 7.7** and 2.5ml of the Acid primary standard in **Section 7.7** and diluting to 250ml using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/ml)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

Semivolatile SIM_surrogate standard is prepared by diluting 1.25mL of the 200ug/mL primary standard in **Section 7.7** to 50mL in methanol. The resulting concentration is 5ug/mL of Benzo(e) pyrene d-12.

The working surrogate spike standard is labeled ZWyymmddX
where:
Z = S for Semivolatile or TPH/DRO, P for Pesticides or PCB
W=Working Standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.7** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)
Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

8.1.2.3. The TPH/DRO and EPH working surrogate standard is prepared by taking 1mL of the 5-α Androstane and 1mL of o-Terphenyl primary standards in Section **7.7** and diluting to 200 ml using methanol. The working standard contains the following compound:

Compound	Concentration(µg/ml)
5-α Androstane	50

o-Terphenyl	50

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile Full Scan working LCS/MS Standard is prepared by combining 2.5ml of Base/Neutral Composite Set primary standard in **Section 7.8**, 2.5ml of Acid Composite Mix primary standard in **Section 7.8** and 2.5ml of Composite Mix #3 primary standard in **Section 7.8** and diluting to 100ml using methanol. The working Semivolatile SIM LCS/MS Standard is prepared by a further 10 times dilution of the above standard.

8.1.3.2 The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See Attachment 1 for a list of the individual compounds and their concentrations.

8.1.3.3 The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.8** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

8.1.3.4 The TPH/DRO working LCS/MS Standard is prepared by first preparing an intermediate standard and making a dilution of it.

On an analytical balance, weigh out 5.00g of diesel fuel into a 10ml Class A volumetric flask and bring to volume with Methanol.

The intermediate spike standard is labeled SIyymmddX
where:
SI = Semivolatile intermediate standard

yymmdd = date intermediate standard is prepared,
X = the order that the intermediate standard is prepared on that date, in increasing alphabetical order.

The working LCS/MS Standard is prepared by taking 1ml of the intermediate standard above, and diluting to 100ml using Methanol. The working LCS/MS Standard contains the following:

Compounds	Concentration(µg/ml)
Diesel fuel	5000

8.1.3.5 The working MA EPH LCS/MS Standard is prepared by taking 2.5mL of the Aliphatic Hydrocarbons standard in Section **7.8** and 2.5mL of the Aromatic Hydrocarbon standard in Section **7.8** and diluting to 250mL in 1:1 methylene chloride: acetone mixture . The final concentration will be 50 ug/ml for each of the individual components.

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2.1**.

A smaller or larger amount may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

Before starting:

- The soxhlet extractor must be inspected periodically to ensure that the glassware does not have any cracks.
- Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.1 Add about 3 Teflon boiling chips to the boiling flask. Place the soxhlet extractor over the boiling flask. Be careful not to knock the extractor over. Use glass wool to plug the hole in the soxhlet extractor.

8.2.2 The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a pre-weighed 400ml glass beaker. A 30 gram ± 0.5 gram sample is transferred into the beaker using a clean stainless steel scoopla. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.3 Add enough anhydrous sodium sulfate to the sample, but not more than 1:1 w/w of the sample. When mixed with the anhydrous sodium sulfate, the solid material must be a free flowing, however, it should not contain an excessive amount of anhydrous sodium sulfate. If a sample matrix presents difficulty, discuss the issue with the OPREP Supervisor or Mitkem's Technical Director before proceeding.

8.2.4 Pour the sample and anhydrous sodium sulfate mixture into the soxhlet extractor. Be sure not to get any soil into the boiling flask.

8.2.5 Sonicate the surrogate standard and the lab control sample standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes.

- Use a syringe to add 1.0 mL of the surrogate standard to the Blank, LCS, all sample and any MS/MSD sample that are in the soxhlets.
- Use a syringe to add 1.0 mL of the LCS/MS standard to the LCS and any MS/MSD samples.

8.2.6 Cover the sample with glass wool to ensure that none of the soil splashes. Add 400ml of the 1:1 v/v methylene chloride/acetone mixture to the soil.

8.2.7 The boiling flask is placed in the heating mantle. The soxhlet is chained so it will not tip over. The condenser is placed on top of the extractor. Start the water running through the condensers. Plug the heating mantles in. Measure the cycle/hour rate and record this in the extraction logbook. Allow the sample to extract for a minimum of 18 hours.

8.2.8 At the end of the extraction, unplug the heating mantles. Allow the samples to cool to room temperature. Take the soxhlet out, drain the extraction solvent into the boiling flask and dump the soil mixture into solid waste container. Cover the boiling flask with aluminum foil.

8.2.9 Record all extraction information, including the Date and Time the soxhlet extraction began and ended, in the appropriate Extraction Logbook (**Figures 1 and 2**).

8.3 The sample extract is now ready for filtration and concentration. These procedures are found in **SOP No. 50.0054**, Extract Filtration and Concentration.

8.4. After concentration the extract may require cleanup. The following is a list of Cleanup Procedures:

8.4.1. **Sulfur cleanup** is mandatory for all Pesticide/PCB sample extracts containing sulfur. All QC samples including blanks, lab control samples, matrix spikes and duplicate matrix spikes must be subjected to the same cleanup as the field samples. Sulfur cleanup will be performed using activated copper powder. Refer to **SOP No. 50.0030** Method 3660B Sulfur Cleanup, for details on activiting copper and using it in a sample extract.

8.4.2. **Acid cleanup (PCB extract only)** is mandatory for all PCB sample extracts. Refer to **SOP No. 50.0031** Method 3665A Sulfuric Acid Cleanup, for details on using the acid cleanup in a PCB sample extract.

8.4.3. **GPC cleanup** is useful for both Pesticide and semivolatile samples. Refer to SOP **No. 50.0032** Method 3640A GPC Cleanup, for details on the procedure for cleanup and quality control criteria for GPC.

8.4.4. Other cleanup methods may be found in SOP numbers **50.0033**(Florisil) and **50.0034** (Silica Gel).

8.5 The extracts are transferred to the Semivolatile lab and documented in the appropriate Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.6 Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.6.1 Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.6.2 Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2 Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1 Frequency of LCS:

A LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or

- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2 Procedure for LCS:

- The LCS is prepared in identical fashion as the associated samples; and in addition:
- 1.0mL of the surrogate solution prepared in **Section 8.1.2** and 1.0 mL of the lab control sample prepared in **Section 8.1.3** is added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.

10.3 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2 Procedures for Duplicate Matrix Spikes:

- The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition
- 1.0 ml of the surrogate solution prepared in **Section 8.1.2** and 1.0 ml of lab control sample prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A re-extraction request will be filled out (**Figure 3**).

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**)..

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will instruct the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out (**Figure 3**).

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety

glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3540C, Ultrasonic Extraction, Revision 3, December 1996.

Attachments:
Attachment 1: Pesticide LCS/MS List
Figure 1: Semivolatile Extraction Log
Figure 2: Pest/PCB Extraction Log
Figure 3: Re-extraction Logbook

Attachment 1: Pesticide LCS/MS List

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES, A Division of Spectrum Analytical, Inc. ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES

Date:	Analysis:	Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: Soil: 3550B (Sonic) 3540C (Soxhlet) _____	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:

Blank ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	MeCL/Acetone 1:1 Lot#	H_2SO_4 Lot#	NaOH Lot#	Time/Date Started:
									Time/Date Ended:

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to Fractionation Date / Analyst		Fractionation Date / Analyst		Final Concentration Date / Analyst		Final Conc. Volume (mL)	Date Extract Trans.

Comments: _____

Logbook ID: 50.0147-01/08

Surrogate Std. ID: _____

Matrix Spike/LCS Std. ID: _____

Reviewed By: _____

Sonicator Tuned: Yes / No

Soxhet Cycle/Hour: _____

Drip Rate _____

Water Bath Temp: _____

Figure 2

Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonic) 3540C (Soxhlet) 3545(PFE) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH Number:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent Lot # Solvent Name	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst RV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst RV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

	PEST Matrix Spike Std ID:	Vol. Spiked:	
	PCB Matrix Spike Std ID:	Vol. Spiked:	
	PEST Surrogate Spike Std ID:	Vol. Spiked:	
	PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane Lot # / L/L Drip Rate:
	PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot# / Soxhlet Cycle/Hour:
	PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot # / Sonicator Tuned? Yes/No

Logbook ID: 50.0149-02/08

Reviewed By:_____

Figure 3

Re-extraction Logbook

Mitkem Laboratories, A Division of Spectrum Analytical Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-07/07

Reviewed By:_____

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques

	Signature	Date
QA Director:		2/26/09
Lab Director:		2/26/09

MITKEM LABORATORIES
A Division of Spectrum Analytical, Inc.

STANDARD OPERATING PROCEDURE

for

Organic Extract Filtration and Concentration Techniques

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the filtration and concentration of organic sample extracts that were prepared using EPA water and SW-846 solid hazardous waste methods, as well as OLC3.2, OLM4.3 and the current SOM version procedures.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

The sample extract is dried through granular anhydrous sodium sulfate and is made ready for cleanup and/or analysis following concentration using Kuderna-Danish (KD) apparatus or Caliper TurboVap apparatus and nitrogen blow down.

4. Sample Preservation, Containers, Handling, and Storage

Not Applicable

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation and concentration, therefore, these materials

must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results for the analytes of interest. See Mitkem SOP Nos. 50.0030 through 50.0034 for cleanup methods.

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation and concentration steps to minimize interferences from these compounds.

6. Equipment and Apparatus

Instrumentation used in this method include:

6.1 Teflon boiling chips.

6.2 Kuderna-Danish (KD)apparatus with a 10 or 15ml receiver tube.

6.3 80mm Glass funnel.

6.4 Glass wool.

6.5 Three ball Snyder column.

6.6 Water bath, capable of maintaining 60 °C to 90°C.

6.7 Boiling chips, carbon.

6.8 Nitrogen blow-down apparatus, N-EVAP Model No. 111.

6.9 Receiving vial with Teflon septa – 2ml.

6.10 15ml storage vials with screw caps.

6.11 Caliper TurboVap apparatus with 200mL collection tube

6.12 Aluminum Foil (Industrial Grade)

6.13 Glass Pipettes

6.14 20X150mm Disposable Culture Tubes

7. Reagents

The following chemicals including solvent, standards and gasses are extensively used in the lab:

7.1. Methylene chloride: J. T. Baker, ultra-resi analyzed grade, to be used for glassware rinsing and sample extraction

7.2. Hexane: Fisher, Optima grade, to be used for solvent exchange of samples.

7.3. Anhydrous sodium sulfate: J. T. Baker, ultra-resi analyzed grade, granular for drying the sample extract.

7.4. Acetone: J. T. Baker; ultra-resi analyzed, to be used for glassware rinsing.

8. Procedure

8.1 Sample Extract Filtration:

All sample extracts are dried through anhydrous sodium sulfate prior to further sample concentration.

8.1.1. A drying funnel is first cleaned with methylene chloride to remove any contaminants. The drying funnel is then prepared by plugging an 80mm diameter filtering funnel with glass wool and adding approximately 20 grams of anhydrous granular sodium sulfate.

8.1.2. Sample extracts are dried through the drying funnels and collected directly into the KD concentrators or Labconco collection tubes depending on which method of concentration is going to be used. Rinse the empty boiling flask and drying funnel with methylene chloride after the initial transfer to ensure the qualitative transfer of the extract.

8.1.3. The KD concentrator apparatus consist of a 500ml KD flask and a 10 or 15ml concentrator tube. The concentrator tube is attached to the flask by placing it on and then giving a gentle twist. The concentrator tube should be on snug. Place a blue Keck clamp to ensure the tube will not fall off. Continue with **Section 8.2.1**.

8.1.4. The Caliper TurboVap 200mL collection tubes are placed underneath the drying funnel and the extract is collected directly into it. Continue with **Section 8.2.2**.

8.2. Sample Extract Concentration:

8.2.1. Procedure for concentration of sample extracts using **KD** apparatus without further Gel Permeation Chromatography (GPC) cleanup.

8.2.1.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.1.2. Attach a three-ball Snyder column to the KD flask and submerge the evaporator into the hot water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.1.2.1. Concentrate the **SV (or TPH/DRO)** extract down to about 4 ml.

8.2.1.2.2. Concentrate the **Pest/PCB** extract down to about 4ml and then add approximately 20ml of hexane through the top of the Snyder column to exchange the solvent. Continue to concentrate the extract to about 4ml. REPEAT.

Caution: It is important that the extract in the KD apparatus not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.1.3. Remove the evaporator from the water bath. Let the evaporator cool for at least 10 minutes, allowing the solvent in the Snyder column to drain back into the KD concentrator tube.

8.2.1.4. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a 20x150mm disposable culture tube and place the culture tube into the nitrogen blow down bath, set at 30-35°C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.1.5. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.1.5.1. Concentrate the **SV (or TPH/DRO)** extract to between 0.5 and 0.8ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 1.0ml mark. Transfer the 1.0ml extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.1.5.2. Concentrate the **Pest/PCB** extract to 5ml. Transfer the extract to a 15ml vial marked at the 10ml mark. Rinse the concentrator tube with 1ml of hexane and add to vial. Rinse the concentrator tube once more. Bring the final extract volume up to 10ml with hexane. Mark the meniscus with a permanent marker.

8.2.2. Procedure for concentration of sample extracts using TurboVap (TV) apparatus without Gel Permeation Chromatography (GPC) cleanup:

8.2.2.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C(recommended).

8.2.2.1.1. Concentrate the **Semivolatile** extracts to 0.5 – 0.8mL. Allow the collection tube to stand without nitrogen flow to cool. Using a 1.0 ml syringe pull up the extract and rinse the interior of the collection tube with the extract. Pull up the extract again with the 1.0 ml syringe. Rinse the collection tube with additional methylene chloride and pull up in the syringe. If needed, add additional methylene chloride to bring the extract to 1.0 ml. Transfer the extract to a 2 ml crimp top autosampler vial. Mark the meniscus with a permanent marker.

8.2.2.1.2. Concentrate the **Pesticide/PCB extract** to about 4ml and then add 20ml of hexane. Raise the temperature to 80 °C(recommended), if necessary, to complete the concentration. Concentrate to about 4 ml again, and add 20 ml of hexane. Continue to concentrate the extract as in **Section 8.2.1.5.2.**

Caution: it is important that the extract in the collection tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.2.2. The extracts are transferred to the GC or GC/MS lab and documented in the appropriate Extract Transfer log. The extracts are stored in the appropriate refrigerator, at 4°C ± 2 °C until analysis.

8.2.3. Procedure for concentrating sample extracts using the **KD** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.3.1. Add 2 boiling chips to the KD flask containing the extraction fluid.

8.2.3.2. Attach a three-ball Snyder column to the flask and submerge the evaporator into the water bath set at medium heat (recommended temperatures are: 60-70 °C for SV (or DRO) and as high as 80-90°C for Pest/PCB during and after hexane exchange) with the tip of the KD concentrator tube in the water.

8.2.3.3. Concentrate the extract down to about 4ml and remove the evaporator from the water bath.

8.2.3.4. Let the evaporator cool for at least 10 minutes, and allow the solvent in the Snyder column to drain back into the concentrator tube.

8.2.3.5. Remove the three-ball Snyder column and the KD flask from the concentrator tube. Transfer the extract into a disposable culture tube and place the culture tube into the nitrogen blow down bath, with the temperature set at 30-35°C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8. Rinse the concentrator tube with a small amount of methylene chloride to ensure a full transfer of the extract.

8.2.3.6. Start a low flow stream of nitrogen across the surface of the extract solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulates inside the tube. Concentrate the samples to 2ml. This is to reduce the concentration of acetone in the Pre-GPC soil extract.

8.2.3.7. Transfer the 2ml extract into a 15ml vial with a line at the 10ml mark. Rinse the concentrator tube with 2ml of methylene chloride and combine the rinsate into the vial. Rinse the concentrator tube one more time with 2ml of methylene chloride and pour into the vial. Bring up to the 10ml mark with methylene chloride.

8.2.3.8. The extract is now ready for GPC cleanup. Refer to SOP 50.0032 for how to perform GPC.

8.2.4. Procedure for concentrating sample extracts using the **TV** apparatus with further Gel Permeation Chromatography (GPC) cleanup:

8.2.4.1. Place the 200mL TV collection tube into the TurboVap. Set the nitrogen to 10 psi, and the temperature to 60 °C (recommended).

8.2.4.2. Concentrate the extract down to about 2-3 ml and remove the collection tube from the nitrogen blow down bath. Allow to cool without nitrogen flow.

8.2.4.3. Pipet or carefully pour the extract into a 15mL vial marked with a line at the 10ml mark. Rinse the concentrator tube with 2mL of methylene chloride and pour into the vial. Rinse the concentrator tube one more time with 2mL of methylene chloride and pour into the vial. Bring up to the 10mL mark with methylene chloride.

8.2.4.4. The extract is now ready for Gel Permeation Chromatography (GPC) cleanup. Refer to SOP 50.0032 for how to perform GPC Method SW3640.

8.2.5. Post GPC the extracts require additional concentration steps.

8.2.5.1. For **Semivolatile** extracts, 5ml of the original 10 ml extract was used for cleanup and the final extract volume must be 0.5ml in methylene chloride.

8.2.5.1.1. Semivolatile extracts (TPH/DRO samples do not undergo GPC Cleanup) are re-concentrated using the KD until approximately 4ml.

8.2.5.1.2. The extract is then transferred to 20x150mm disposable culture tubes and placed into the nitrogen blow down bath, set at 30-35°C(recommended). The dial should be set to 5 to attain this temperature range. Pressure should be set to 8.

8.2.5.1.3. Start a low flow stream of nitrogen across the surface of the extraction solvent. The flow rate should be such that no solvent will splash from the tube or any condensation accumulate inside the tube.

Caution: it is important that the extract in the culture tube not be allowed to go dry to minimize volatilization of the more volatile target compounds.

8.2.5.1.4. Concentrate the **SV** extract to a final volume of 0.5ml. Pull the extract up with a 1.0ml syringe. Rinse the collection tube with a small volume of methylene chloride and pull up the rinsate filling the syringe to the 0.5ml mark. Transfer the extract to a 2ml auto-sampler vial. Mark the meniscus with a permanent marker.

8.2.5.2. For **Pesticide/PCB** extracts, 5ml of the original 10 ml extract was diluted to a 10ml final volume using methylene chloride, and was used for GPC cleanup. The final extract should be solvent exchanged to hexane and concentrated to 5ml.

8.2.5.2.1. The post GPC extract is transferred to a TV apparatus with the temperature set to 60 °C(recommended). Concentrate to about 4 ml. Raise the temperature to 80 °C(recommended), if necessary, to complete the concentration. Add 20ml of hexane to exchange the solvent. REPEAT. Continue to concentrate the extract until it has reached 2-3ml. Transfer the extract to a 15mL vial marked at the 5mL mark. Rinse the collection tube with hexane several times to ensure a full transfer. The final volume must be 5ml. Mark the meniscus with a permanent marker.

8.2.5.2.2. The extract is ready for florisil cleanup. Refer to SOP 50.0033 for detailed instructions on florisil cleanup, Method SW3620.

8.2.5.2.3. Other cleanup methods may be employed if needed. Possible procedures may be: Silica Gel Cleanup, Method SW3630 (SOP 50.0034), Acid Cleanup, Method SW3665 (SOP 50.0031) or Sulfur Cleanup, Method SW3660 (SOP 50.0030).

8.2.6. Excess volume of extracts are stored in screw cap vials and labelled with color coded lab tape. The color of the tape records the extract holding time until disposal. See chart in laboratory.

8.3. Sample and Extract Disposal:

All sample extracts are disposed of in accordance with applicable OSHA and state regulations. All sample extracts must be protected from light and stored at 4°C ± 2°C.

8.3.1. Sample Extracts – All sample extracts are kept for 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of.

8.3.2. EPA CLP/SOM sample extracts- All sample extracts are kept until 365 days after delivery of a reconciled, complete data package.

9. Data Reduction and Calculations

Not Applicable

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. In order to trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and contains the manufacturer's reference number given in the standard preparation logbook.

10.1. Method Blank – A method blank is a liter of organic free reagent water that is carried through the entire analytical procedure. It is used to determine the level of contamination associated with the analytical processing and analysis of samples.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank

- The Method Blank is prepared in identical fashion as the associated samples.

- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a numerical value, which increases with every batch of twenty samples or less.

10.2. Surrogate – Surrogate standards are added to all samples including the Method Blank, Lab Control Sample and matrix spikes to assess the efficiency of the sample preparation and analysis procedures.

10.3. Lab Control Sample (LCS) – A Lab Control Sample is a liter of organic free reagent water that is spiked with all target analytes and the surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1. Frequency of LCS:

An LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.3.2. Procedure for LCS:

An LCS is prepared in identical fashion as the associated samples; in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the corresponding numerical value as the associated method blank.

10.4. Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix. For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1. Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Once every 20 samples, or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.4.2. Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples, in addition:

- An aliquot of surrogate solution prepared in **Section 8.1.2** and lab control spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. The Quality Control Officer will perform periodic and unscheduled reviews. These data consist of the final volume of sample extracts, the volume and lot number of solvents used, and extract transfer dates.

11.2 Reporting of the data will include review by the Organic Preparation Laboratory Supervisor of the data listed in **Section 11.1**, time of extraction, sampling handling procedures, and extract handling procedures.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples associated with a non-compliant Method Blank are re-extracted and reanalyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when method blanks have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out. The re-extracted samples will be labeled with the suffix RE.

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out .

The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects.

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. Any sample(s) that is/are associated with a non-compliant LCS will require re-extraction and re-analysis. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A re-extraction request will be filled out .
The re-extracted samples will be labeled with the suffix RE.

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, IV, or On-line Revisions of 3500 and 3600 series for Organic Extraction, Sample Preparation and Cleanup.

USEPA Statement of Work, Current OLC, OLM and SOM Methods.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3570)

SOP No. 50.0100

	Signature	Date
QA Director:		1/31/08
Lab Director:		2/4/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Organic Preparation of Soil Samples by Microscale Solvent Extraction (Method 3570)

1. Scope and Application

This Standard Operating Procedure (SOP) pertains to the preparation of soil, sediment, tissues, biota and any sample considered solid in nature. Discussions include sample extraction, sample cleanup references, sample concentration technique references and the preparation of standard spiking solutions for the analysis of semivolatile or pesticide/PCB organic compounds in soil samples.

2. Personnel Qualifications and Responsibilities

This method is restricted to use by or under the supervision of trained analysts in the Organic Preparation Laboratory (OPREP). Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Mitkem's training requirements for analysts before performing this extraction method.

3. Summary of Procedure

A 2 to 20-gram sample is weighed directly into a 40 or 60-mL VOA vial. The sample is solvent extracted first with acetone. Then it is extract three times with methylene chloride by either a manual shake approach or via rotation or spinning of the sample. For analysis of Pesticides, PCBs and Petroleum samples hexane rather than methylene chloride can be used. The extract is separated from the sample by gravity filtration. The extract is dried through powdered anhydrous sodium sulfate and is ready for cleanup and/or analysis following concentration using a Kuderna-Danish (KD) or TurboVap apparatus.

4. Sample Preservation, Containers, Handling, and Storage

4.1 The sample holding times are as follows:

- Sample must be extracted within 14 days from the time of sample collection
- Samples can be held for one year if they are frozen

- Sample extracts must be analyzed within 40 days of sample extraction

4.2 Sediment/Soil samples – Decant and discard any water layer on the sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves and rocks. Refer to SOP No. 110.0039 for more detail on sub sampling techniques.

4.3 Waste samples consisting of multiple phases must be prepared by separating the phases and performing the appropriate extraction technique on the phase(s) of interest.

5. Interferences and Potential Problems

5.1 Solvents, reagents, glassware and other sample processing hardware can yield interferences during sample preparation; therefore, these materials must be demonstrated to be free of interferences under the conditions of the analysis by analyzing method blanks.

5.2 Interferences may be co-extracted from the sample. Additional cleanup steps may be necessary to give improved results of analysis of the analytes of interest

5.3 Phthalate esters can contaminate sample extracts, as many products found in the laboratory contain these esters. Plastics must be avoided during the preparation steps to minimize interferences from these compounds.

6. Equipment and Apparatus

6.1 Rotary extraction apparatus

6.2 40 and 60 mL VOA vials and caps.

6.3 Balance capable of weighing +/- 0.1 gram.

6.4 Glass syringes for delivering spike and surrogate solutions.

7. Reagents

The following chemicals including solvents and standards are extensively used in the lab:

7.1 Methylene chloride: J. T. Baker, ultra resi analyzed, to be used for glassware rinsing and sample extraction.

7.2 Methanol: Fisher, Optima, to be used for rinsing glassware and preparing standards.

7.3 Acetone: J.T. Baker, ultra-resi analyzed, to be used for rinsing glassware.

7.4 Hexane: Fisher, Optima grade, to be used for solvent exchange of samples

7.5 H_2O, Deionized.

7.6 Copper, Fisher, purified. Fine or Granular.

7.7 H_2SO_4, concentrated, Fisher, ACS trace metals.

7.8 Surrogate Standards:

- Restek BN Surrogate mix (Cat. No. 31086)at 5000µg/mL in Methylene Chloride; Restek Acid surrogate mix (Cat. No. 31087) at 10,000µg/mL in Methanol.
- Deisel Range Organics (DRO)/TPH: 5-α Androstane at 10,000µg/mL (Made from neat source(Sigma)), and o-Terphenyl at 10,000µg/mL(Made from neat source(Aldrich)).
- Pesticides/PCB: Ultra Decachlorobiphenyl (DCB)(Cat. No. PPS-150) at 1000 µg/mL in Toluene, and Ultra 2,4,5,6-Tetrachloro-m-xylene (TCX)(Cat. No. IST-440) at 2000µg/mL in Acetone.

7.9 Lab Control Sample and Matrix Spike:

- Semivolatile: Restek 8270 MegaMix (Cat. No. 31850) at 1000µg/mL in Methylene Chloride; Restek 3,3' Dichlorobenzidine (Cat. No. 31026) at 2000µg/mL in Methanol, and 8270 add-on compounds from neat. An 8270 add-on Intermediate standard is prepared for the following compounds at 1000µg/mL in Methanol: Benzaldehyde, 1,1' Biphenyl, Caprolactam, Acetophenone, Atrazine.
- Pesticides: Restek Single/Dual Column Organochlorine Pesticides Mix AB#2 (Cat. No. 32292) at 8- 80µg/mL.
- PCB: Restek Aroclor 1016/1260 (Cat. No. 32039) at 1000µg/mL in Hexane.
- TPH/DRO: LCS uses Diesel/gasoline.

8. Procedure

8.1 Standards Preparation: All standards for the Organic Preparation Laboratory (OPREP) are prepared in the associated Instrumentation Laboratory (GC or GC/MS). The GC or GC/MS Laboratory analyzes the standards at dilution for quality control purposes prior to relinquishing the standards to OPREP. When more than one bottle is prepared, only one bottle is transferred at a time to OPREP. The OPREP technician will notify the GC or GC/MS Laboratory Analyst when new standard is needed, or the last bottle is being taken.

8.1.1 All primary standards received from vendors are logged into the Semivolatile or Pesticides/PCB Primary Standard Logbooks. These include standards for surrogates, LCS and matrix spikes. The standards are labeled ZPyymmddX, where:
Z = S for Semivolatile, P for Pesticides or PCB
P= Primary standard
yymmdd = date standard is received, and
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

The expiration date for ampulated solutions shall not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first.

8.1.2 Preparation of Surrogate standard:

8.1.2.1. The Semivolatile working surrogate standard is prepared by combining 2.5mL of the Base/Neutral primary standard in **Section 7.8** and 2.5mL of the Acid primary standard in **Section 7.8** and diluting to 250mL using methanol.

The working standard contains the following compounds:

Compound	Concentration(µg/mL)
1,2-Dichlorobenzene-d_4	50
2-Fluorobiphenyl	50
Nitrobenzene-d_5	50
p-terphenyl-d_{14}	50
2-Chlorophenol-d_4	75
2-Fluorophenol	75
Phenol-d_6	75
2,4,6-Tribromophenol	75

The working surrogate spike standard is labeled SWyymmddX where:
SW = Semivolatile working standard
yymmdd = date working standard is prepared,
X = the order that the working standard is prepared on that date, in increasing alphabetical order.

8.1.2.2. The working Pesticide/PCB surrogate standard is prepared by combining 1.2mL of the DCB mix and 0.3mL of the TCX mix in **Section 7.8** , and diluting to 1000mL using acetone. The working standard contains the following compounds with the following concentrations:

Compound	Concentration(µg/mL)

Decachlorobiphenyl	1.2
2,4,5,6-Tetrachloro-m-xylene	0.6

A smaller or larger amount may be used to prepare the surrogate. The final volume of the surrogate solution will be adjusted accordingly.

The expiration date for the surrogate standard is six months from the date of preparation.

8.1.3 Preparation of Lab Control Sample (LCS) and Matrix Spike (MS) Standard:

8.1.3.1. The Semivolatile working LCS/MS Standard is prepared by combining 2.5mL of Base/Neutral Composite Set primary standard in **Section 7.9**, 2.5mL of Acid Composite Mix primary standard in **Section 7.9** and 2.5mL of Composite Mix #3 primary standard in **Section 7.9** and diluting to 100mL using methanol. All compounds have a final concentration of 50 ug/mL.

8.1.3.2 The working Pesticide LCS/MS Standard is prepared by taking 1.25mL of the Pest Mix AB#2, and diluting it to 50 mL in methanol. The working standard contains the full list of SW8081 individual response compounds at 2-20 µg/mL. See Attachment 1 for a list of the individual compounds and their concentrations.

8.1.3.3 The working PCB LCS/MS Standard is prepared by taking 0.8mL of Aroclor 1016/1260 mix in **Section 7.9** and diluting it to 200mL in acetone. The working standard contains the following compounds with the following concentrations:

Compounds	Concentration(µg/mL)
Aroclor 1016	4.0
Aroclor 1260	4.0

The LCS/MS Standard is labeled using the same approach discussed in **Section 8.1.2**.

A smaller or larger amount may be used to prepare the LCS/MS Standard. The final volume will be adjusted accordingly.

The standard solution is placed in amber bottles and stored in the freezer in the GC or GC/MS lab at –10 to –20°C. One bottle is transferred to the OPREP Lab

and stored in the freezer at –10 to –20°C. The bottles are stored in a separate location from samples or sample extracts to make sure that there is no cross contamination.

The expiration date for the ampulated solutions is discussed in **Section 8.1.2.**

All of the appropriate standard preparation information is to be recorded in the appropriate Lab's working Standard Logbook.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8.2 Sample Extraction Procedure:

8.2.1. Remove the surrogate and LCS/MS spiking standards from the freezer and allow them to reach room temperature.

8.2.2. Sonicate the surrogate standard and the LCS/MS standard prepared in **Sections 8.1.2** and **8.1.3** for about 10 minutes.

8.2.3. The sample is mixed to ensure sample homogeneity. A representative portion of the sample is measured into a 40 mL VOA vial. The sample is weighed into the tared VOA vial. The weight on the sample will vary with the testing procedure and matrix. Tissues usually are weighed in the 3-5 gram range. Soils and sediments are usually in the 5-7 gram range. The data quality objective of the work will dictate if there should be variations from these recommended target weights. The weight measurement is recorded to the nearest 0.1 gram. Calibrate the balance prior to use. Refer to **SOP No. 110.0007** for direction.

8.2.4. Semivolatile Extraction Procedure:

8.2.4.1. Spike the samples and QC with the appropriate surrogates and spike solutions.

- Use a syringe to add 1.0mL of the Semivolatile surrogate standard to the Blank, LCS, samples and any MS/MSD sample that are in the vials.
- Use a syringe to add 1.0mL of the Semivolatile LCS/MS standard to the LCS and any MS/MSD samples.

8.2.4.2. Add 5-10 mL (7 mL) of acetone to Blank, LCS, samples, and any MS/MSDs. The volume should be enough to **completely cover the sample**. There are instances where samples will appear to absorb the solvent (humic materials for example). In these cases add enough solvent to cover the sample.

8.2.4.3. If sediments are being extracted, added activated copper to aid in the removal of sulfur after the acetone has been added.

8.2.4.4. Cap the vials and shake vigorously for 30 seconds.

8.2.4.5. Allow the samples to settle after agitation.

8.2.4.6. Decant the solvent layer into a 60mL VOA vial, leaving the solid in the 40mL VOA vial.

8.2.4.7. If the samples are extremely wet sediments or tissues, repeat the acetone step from above. At this step, water has been removed from the sample by the acetone.

8.2.4.8. For sediments sulfur is always suspected. Activated copper can be added to the acetone in the 60 mL vials to remove copper.

8.2.4.9. Extract the sample three more times with 5-10 mL (7 mL) of methylene chloride. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.

8.2.5. Procedure for Pesticides, PCBs and Petroleum Hydrocarbon:

8.2.5.1. Spike the samples and QC with the appropriate surrogates and spike solutions.

- Use a syringe to add 1.0mL of the surrogate standard into the Blank, LCS, sample and any MS/MSD sample that is in the 40mL vial.
- Use a syringe to add 1.0mL of the lab control sample standard into the LCS and any MS/MSD samples that is in the 40mL vial.

8.2.5.2. Continue as in **Sections 8.2.4.2 through 8.2.4.8**.

8.2.5.3. Extract the sample three more times with 5-10 mL (7 mL) of Hexane. Generally the samples will be placed on a rotator and spun for 30 minutes. Decant the solvent between spins into the 60 mL VOA vial. Cap the vials in preparation of transferring the solvent extract through the drying funnel to the KD apparatus.

8.2.5.3.1. For the chlorinated Pesticides and PCBs which are readily extractable, the samples can be extracted by shaking the samples in hexane for three minutes rather than spinning the samples.

8.2.5.4. If the samples contain a petroleum odor, the sample size should be reduced to one to two grams.

8.2.6. Record all extraction information in the appropriate Extraction Logbook (see **Figures 1 and 2**).

8.3. Sample Filtration and Concentration: Refer to SOP 50.0054, Extract Filtration and Concentration.

8.4. Extract Cleanup: Refer to SOPs 50.0030 Method 3660B Sulfur Cleanup, 50.0031 Method 3665A Acid Cleanup, 50.0032 Method 3640A GPC Cleanup, 50.0033 Method 3620B Florisil Cleanup, and 50.0034 Method 3630C Silica Gel Cleanup for details on the different cleanup methods available.

8.5. The final extracts are transferred to the Semivolatile lab and documented in the Extract Transfer Log. The extracts are stored in the refrigerator at 4°C until analysis.

8.6. Sample and Extract Disposal:

All samples and sample extracts are disposed of in a way in accordance with applicable OSHA and state regulations.

8.6.1. Samples – All unused portions of samples are returned to the respective storage area. Such portions are kept for 60 days after data submission. After such period, the remainder of the samples is disposed of by the Sample Custodian or his/her designee.

8.6.2. Sample Extracts – All sample extracts are kept for at least 60 days after the submittal of data for the last sample. After such period, the sample extracts are disposed of by the GC or GC/MS labs.

9. Data Reduction and Calculations

Data reduction for calculation of standard preparation:

$$\text{Concentration of working standard} = \frac{\text{(Concentration of ampule)(amount used)}}{\text{Final Volume}}$$

10. Quality Assurance/Quality Control

Quality assurance and quality control (QA/QC) procedures are implemented to ensure generation of data of known and documented quality. QA/QC procedures associated with the Organic Preparation Laboratory include preparation of Method Blanks, surrogate spikes, lab control sample, matrix spikes and balance checks. To trace spiking standards, the original manufacturer's list of standard compounds and concentrations are filed and the manufacturer's reference number is documented in the standard preparation log book.

10.1. Method Blank – A method blank is a weight of a clean reference matrix (granular sodium sulfate) that is carried through the entire analytical procedure.

10.1.1 Frequency of Method Blank:

A Method Blank is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within an SDG, or
- Each extraction procedure within an SDG, or
- Whenever samples are extracted.

10.1.2 Procedure for Method Blank:

- The Method Blank is prepared in identical fashion to the associated samples.
- The Method Blank is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The Method Blank is labeled **MB** and is given a sequential number for every batch of twenty samples or less.

10.2 Lab Control Sample (LCS) – A Lab Control Sample is a weight of a clean reference matrix (granular sodium sulfate) that is spiked with all appropriate target analytes and surrogate spikes, and carried through the entire analytical procedure.

10.2.1 Frequency of LCS:

A LCS is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG, or
- Whenever samples are extracted.

10.2.2 Procedure for LCS:

The LCS is prepared in identical fashion as the associated samples; and in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS Spike prepared in **Section 8.1.3** are added to the LCS sample.
- The LCS is subjected to similar extraction, cleanup, concentration and analysis procedures.
- The LCS is labeled **LCS** and is given the same numerical value as the corresponding method blank.
- The LCS is analyzed and the results are calculated for the recovery of all spiked analytes in the LCS.

10.3 Duplicate Matrix Spikes – Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.3.1 Frequency of duplicate matrix spikes:

A duplicate set of matrix spikes is extracted once for the following, whichever is more frequent:

- Each SDG analysis (not to exceed 20 samples), or
- Each matrix within a SDG, or
- Each extraction procedure within a SDG.

10.3.2 Procedures for Duplicate Matrix Spikes:

The duplicate matrix spikes are prepared in identical fashion as the associated samples; in addition:

- Surrogate solution prepared in **Section 8.1.2** and LCS spike prepared in **Section 8.1.3** are added to the duplicate matrix spike samples.
- The duplicate matrix spikes are subjected to similar extraction, cleanup, concentration and analysis procedures.
- The duplicate matrix spikes are analyzed and the results calculated for the recovery of the spiked analytes in the duplicate matrix spike.

11. Data Validation and Reporting

11.1 Data generated in the organic preparation laboratory will be reviewed by the supervisor. These data consist of, but are not limited to, extraction/preparation logbook entries, balance calibration logbooks, weights for soil samples, volumes and lot numbers of solvent used. The Quality Control Officer will perform periodic and

unscheduled reviews.

12. Corrective Action Procedures

Corrective actions are to be taken if the QA/QC as outlined in this SOP are not adhered to:

12.1 Method Blank Analysis:

All samples that are prepared with a non-compliant Method Blank will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when method blank contamination has occurred. A request for re-extraction will be filled out (**Figure 3**).

12.2 Surrogate Recovery:

All samples with surrogate recoveries outside of the control limits will be re-extracted and re-analyzed. The re-extracted sample is labeled with the suffix RE. If the re-extracted sample exhibits similar behavior, both data sets will be submitted to demonstrate matrix effects. The analysis laboratory will inform the Organic Preparatory Laboratory when surrogate recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.3 LCS Recovery:

All samples that are associated with the non-compliant LCS will be re-extracted and re-analyzed. The re-extracted samples will be labeled with the suffix RE. The analysis laboratory will inform the Organic Preparatory Laboratory when LCS recoveries have not met accepted criteria, and require re-extraction. A request for re-extraction will be filled out (**Figure 3**).

12.4 Matrix Spike Recovery and RPD:

These are used as advisory limits and do not trigger sample re-extraction.

13. Health and Safety

Health and safety hazards in the Organic Prep Lab include exposure to analytical standards and solvents. Always work in under a well-ventilated hood. Lab coats, gloves and safety glasses must be worn in the lab at all times.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Quality Assurance Plan, Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

U.S. Environmental Protection Agency. SW-846 Test Methods for Evaluating Solid Wastes, Update III, Method 3570, Microscale Solvent Extraction, Revision 0, November 2002.

Attachments:
Attachment 1: Pesticide LCS/MS list
Figure 1: Semivolatile Extraction Log
Figure 2: Pesticides/PCB Extraction Log
Figure 3: Re-extraction Request

Attachment 1: Pesticide LCS/MS list

Analyte	Spike conc in ug/mL
4,4´-DDD	0.4
4,4´-DDE	0.4
4,4´-DDT	0.4
Aldrin	0.2
alpha-BHC	0.2
alpha-Chlordane	0.2
beta-BHC	0.2
delta-BHC	0.2
Dieldrin	0.4
Endosulfan I	0.2
Endosulfan II	0.4
Endosulfan sulfate	0.4
Endrin	0.4
Endrin aldehyde	0.4
Endrin ketone	0.4
gamma-BHC (Lindane)	0.2
gamma-Chlordane	0.2
Heptachlor	0.2
Heptachlor epoxide	0.2
Methoxychlor	2

Figure 1

Semivolatile Extraction Log

MITKEM LABORATORIES, A Division of Spectrum Analytical Inc. ORGANIC PREP - SAMPLE PREPARATION: SEMIVOLATILES

Date:	Analysis:		Method #	Aq: 3510C (SepF) 3520C (Liq/Liq) Other: _____ Soil: 3550B (Sonic) 3540C (Soxhlet)	Matrix Aqueous Soil Wipe Oil Other:_____	Sodium Sulfate Lot #:
Blank ID	LCS ID	Analyst	Spiked By	Witness	MeCl Lot#	MeCL/Acetone 1:1 Lot#
H_2SO_4 Lot#	NaOH Lot#	Time/Date Started:	Time/Date Ended:			

Lab ID	Bottle #	Sample Wt (g) / Vol (mL)	Initial pH	Surrogate Spike Added(uL)	Matrix Spike Added(uL)	H2SO4 pH<2	NaOH pH>11	Emulsion	KD/RV Prior to Fractionation Date / Analyst	Fractionation Date / Analyst	Final Concentration Date / Analyst	Final Conc. Volume (mL)	Date Extract Trans.

Comments _____

Sonicator Tuned: Yes / No
Soxhet Cycle/Hour:_____
Drip Rate_____
Water Bath Temp:_____

Logbook ID: 50.0147-11/07

Reviewed By:_____

Figure 2
Pest/PCB Extraction Log

MITKEM LABORATORIES ORGANIC PREP-SAMPLE PREPARATION:PEST/PCB

Date:	Prep Method	Aq: 3510C (SepF) 3520C (Liq/Liq) Soil: 3550B (Sonic) 3540C (Soxhlet) 3545(PFE) 3570(MSE) Other:_____	Matrix: Aqueous Soil Wipe Oil Other:_____	BATCH Number:	Date/Time Started:

Analyst	Spiked By	Witness	Solvent Lot # Solvent Name	GPC Batch Number	Florisil Lot #	Analysis:	Date/Time Ended:

	Lab ID	Bottle #	Sample Wt (g) / Vol (ml)	Initial pH	Emulsion?	Initial CONC Date/ Analyst RV/ KD	Extract volume (ml)	GPC Cleanup Date / Analyst	FLORISIL Cleanup Date / Analyst	ACID (PCB only) Cleanup Date / Analyst	SULFUR (Copper) Cleanup Date / Analyst	Final CONC Date/ Analyst RV/ KD	Final extract volume (ml)	Extract Transfer Date /By

Comments:

	PEST Matrix Spike Std ID:	Vol. Spiked:	
	PCB Matrix Spike Std ID:	Vol. Spiked:	
	PEST Surrogate Spike Std ID:	Vol. Spiked:	
	PCB Surrogate Spike Std ID:	Vol. Spiked:	Hexane Lot # / L/L Drip Rate:
	PEST LCS Spike Std ID:	Vol. Spiked:	Sulfuric Acid lot# / Soxhlet Cycle/Hour:
	PCB LCS Spike Std ID:	Vol. Spiked:	Sulfur cleanup Copper lot # / Sonicator Tuned? Yes/No

Logbook ID: 50.0149-12/07

Reviewed By:_____

Figure 3
Re-extraction Request

Mitkem Laboratories, A Division of Spectrum Analytical Inc.
RE-EXTRACTION LOGBOOK
ORGANICS

REQUESTED BY:
DATE:

Mitkem Sample ID	Sample Matrix	Analysis	MB originally associated with sample	Reasons for Re-extraction

PREP LAB APPROVAL:

DATE:

Logbook ID 50.0190-07/07

Reviewed By:_____

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8082

SOP No. 60.0003

Rev. 8

	Signature	Date
QA Director:		9/15/08
Lab Director:		9/15/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Polychlorinated Biphenyls by Gas Chromatography/Electron Capture Detector

Analysis by SW846 Method 8082

Rev. 8

1. Scope and Application

This SOP describes the procedures applicable to the analysis of the compounds listed in **Table 1**. This SOP describes the analysis of polychlorinated biphenyls (PCB) as Aroclors in solid or aqueous sample extracts, using a gas chromatograph equipped with an electron capture detector. This SOP gives specific information to perform the analysis according to protocols discussed in USEPA SW846 Test Methods for Evaluating Solid Waste Method 8082A, and Department of Defense Quality System Manual for Environmental Laboratories, Final Version 3. All matrices including ground water, aqueous samples, TCLP and SPLP extracts, petroleum oil, wipes, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require extraction and/or clean-up prior to analysis. **Section 8.2.1** provides the SOP references for sample extraction and clean-up procedures to be used with this analytical procedure. **Section 10** provides the quality control (QC) requirements required by Method 8082. A list of acronyms used in this SOP is included in **Table 2**.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors and Peer analysts** review the logbooks and data generated from this procedure and approve all reported results. The **Data Reviewer** evaluates all laboratory reports for reasonableness of the results. The **Project Manager** reviews the final report and signs the narrative. The **QA**

Director reviews all quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure

3.1 A sample extract is analyzed by injecting a 1-2µL aliquot into a gas chromatograph (GC) using an auto-sampler. Concentrations of various PCB aroclors are determined by separation of the analytes using a GC equipped with fused silica, open-tubular, megabore columns and electron capture detectors (ECD). Hewlett Packard's Chem-Station (G1034C Version C03.00) is used to handle data acquisition. Target software from ThruPut (Revision 4.14) is used for data reduction and forms generation is through Omega LIMS.

3.2 While SW846 Method 8082 is also applicable to determining polychlorinated biphenyls as congeners, this SOP specifically analyzes PCB as aroclors.

3.3 PCB aroclors are determined using dual-column system with dissimilar phases. The method allows for the option of dual columns joined to a single injection port and individually connected to two ECDs

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by clients and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients provide their own containers. For PCB analysis by USEPA SW846 Method 8082, water samples are collected in 1-liter amber glass bottles with no preservation added to the samples. Solid samples are collected in 8-ounce amber glass containers with no preservation needed. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may be required for the analysis of laboratory QC samples.

4.2 All sample extracts are stored at 4°C ± 2°C until analyzed.

4.3 Sample extract holding time for PCB analysis by the method is 40 days from date of extraction to date of analysis. The holding time for sample extraction is covered in the corresponding extraction SOP.

4.4 The sample extracts are transferred from Organic Prep Laboratory with all appropriate sample prep information in 2ml auto-sampler vials with Teflon lined crimp cap. All vials should have a meniscus to mark the level of the extracts.

4.5. Samples, sample extracts and standards must be stored separately.

5. Interferences and Potential Problems

5.1 Sources of interference in this method can be grouped into three broad categories: (1) contaminated solvents, reagents, or sample processing hardware; (2) contaminated GC carrier gas, parts, column surfaces or detector surfaces; and (3) the presence of co-eluting compounds in the sample matrix to which the ECD will respond. Interferences co-extracted from the samples will vary considerably from waste to waste. While general clean-up techniques are referenced or provided as part of this method, unique samples may require additional clean-up approaches to achieve the desired degree of discrimination and quantitation.

5.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination. These materials may be removed prior to analysis using Gel Permeation Clean-up (Method 3640). Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and batch analyzing the solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. The solvent rinse should be followed by detergent wash with hot water and rinsed with tap water. Drain the glassware and dry overnight. Store dry glassware in a clean environment. Prior to use, rinse the glassware in methanol and methylene chloride respectively.

5.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCB aroclors. Sulfur contamination may be expected with sediment samples. Method 3660 is suggested for removal of sulfur.

5.5 Waxes, lipids, and other high molecular weight materials may interfere with the analysis of PCB aroclors and can be removed by Gel-Permeation Clean-up (GPC) (Method 3640). Co-eluting chlorophenols can be removed by florisil clean-up (Method 3620).

6. Equipment and Apparatus

6.1 Gas Chromatograph/Electron Capture Detector

There are 5 GC/ECD in the laboratory. Three of the instruments have similar configurations and are labeled as E1, E2 and E3. The other two are GC/μ-ECD are labeled as E4 and E5. GC E1, E2 and E3 – Hewlett Packard (HP) Model 5890 Series II with electron Pressure Control. GC E4 and E5 - HP Model 6890 Series II with u-Electron Pressure Control. All five GC's are temperature programmable instruments with single injection port and dual electron capture detectors, and are equipped with HP Model 7673A Auto-sampler. A single gooseneck splitless injection liner is used in the injection port and connected to a piece (up to a 5m long) of uncoated megabore column (0.53mm id) which serves as a guard column. A guard column is connected to the tee split. Each of two columns is connected to the split tee also. HP 3365 Chemstation software is used in conjunction with EnviroQuant software to handle data acquisition and processing. All GC's are interfaced to the network workstation. LIMS software is used for reports.

6.1.1 HP Vectra PC.

6.1.2 HP 3365, EnviroQuant and Target software.

6.1.3 HP Model 7673A auto-sampler.

6.1.4 Three pairs of chromatographic columns are intensively used in the laboratory:

Pair 1 DB-1701 column (J & W Scientific) 30 m x 0.53mm id (1.0 μm film thickness) and DB-608 column (J & W Scientific) a 30 m x 0.53mm id (0.83 μm film thickness)

Pair 2 RTX-CLPesticides (Restek) 30 m x 0.53 mm id (0.5 um film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.53 mm id (0.42 um film thickness)

Pair 3 RTX-CLPesticides (Restek) 30 m x 0.32 mm id (0.5 um film thickness) and RTX-CLPesticides II (Restek) 30 m x 0.32 mm id(0.42 um film thickness).

6.1.5 Gooseneck splitless injection liner, Cat #20799 from Restek or equivalent

6.1.6 Uncoated megabore guard column (0.53 mm id, up to 5 m long). Cat # 10028 from Restek or equivalent.

6.1.7 Universal "Y" Press-tight tee split Cat # 20406 from Restek or equivalent.

6.2 Glassware:

6.2.1 Class "A" volumetric flasks:
10 ml, 25 ml, 50 ml, 100 ml, and 250 ml.

6.2.2 Syringes:
10 µl, 25 µl, 50 µl, 100 µl, 500 µl, 1 ml, 2.5 ml (accuracy to ± 1% per vendor's specification).

7. Reagents and Standards.

7.1 n-Hexane: Fisher Optima grade.

7.2 The standards used in the method are discussed below. Please note that standards from other vendors could be used as long as the standards are of high purity (>96%) and traceable to reference materials. The laboratory will at all time archive or have on order one complete set of un-opened ampulated standards.

7.3 The list of primary (ampulated) calibration standards are obtained from Restek:

- Aroclor 1016 and 1260 (Cat. # 32039) at 1000ug/mL.
- Aroclor 1221 (Cat.# 32007) at 1000ug/mL.
- Aroclor 1232 (Cat. # 32008) at 1000ug/mL.
- Aroclor 1242 (Cat. # 32009) at 1000ug/mL.
- Aroclor 1248 (Cat. # 32010) at 1000ug/mL.
- Aroclor 1254 (Cat. # 32011) at 1000ug/mL.
- Tetrachloro-m-xylene (TCX) (Cat.# 32027) at 200 ug/mL.
- Decachlorobiphenyl (DCB), (Cat.# 32029) at 200 ug/mL

7.4 The list of second source standards are obtained from Supelco:

- Aroclor 1016 (Cat. # 4-8097) at 1000ug/mL.
- Aroclor 1221 (Cat. # 4-8098) at 1000ug/mL.
- Aroclor 1232 (Cat. # 4-4805) at 1000ug/mL.
- Aroclor 1242 (Cat. # 4-4806) at 1000ug/mL.
- Aroclor 1248 (Cat. # 4-4807) at 1000ug/mL.
- Aroclor 1254 (Cat. # 4-4808) at 1000ug/mL.
- Aroclor 1260 (Cat. # 4-4809) at 1000ug/mL.

7.5 All of the above primary standards are logged into the Primary Pesticide Standard Logbook and labeled as **PP*yymmddX***

Where: PP = Pesticides/PCB primary standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in alphabetical order.

The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. All primary standards are stored according to manufacturer's recommendation. All vials containing primary standards must be labeled according to the current version of SOP No. 80.0001 Standard Preparation, Equivalency and Traceability.

7.6 Intermediate Standards.

All intermediate standards are labeled **PI*yymmddX*** where:

PI = Pesticide Intermediate Standard.
yymmdd = date the intermediate standard is prepared.
X = the order that the intermediate standard is prepared on that date in alphabetical order.

Smaller or larger volumes may be used and the final volume adjusted to keep the concentration the same.

All intermediate standards are stored in amber glass bottles under refrigeration at 4°C or below. All standards are stored separately from samples and sample extracts

All intermediate standard solutions must be prepared every six months, or sooner, if the solution has degraded or concentrated.

All of the appropriate standard preparations are recorded in the Pesticide/PCB Intermediate Standard Preparation logbook.

Analyst must allow all intermediate standard solutions to equilibrate to room temperature before use.

7.6.1 Intermediate Pesticide/PCB Surrogate Mix:

The intermediate stock standard is made by diluting 0.5mL of stock TCX and 1.0 mL of stock DCB and diluting to 50 mL with hexane. Intermediate Pesticide/PCB Surrogate Mix contains TCX at 2 ug/mL and DCB at 4ug/mL.

7.6.2 Intermediate AR1660 (AR1016+AR1260) Stock Solution:

The two Aroclors –Aroclor1016 and Aroclor 1260 have different chromatographic patterns and can be combined for analysis. When the two Aroclors are combined, they are labeled as Aroclor 1660.

The mixture of the two Aroclors is prepared by diluting 1mL of primary Aroclor standard at 1000ug/mL (AR 1016 + AR1260) Mix into 100mLs of hexane.

Concentration of the intermediate AR1660 standard solution is10ug/mL

7.6.3 Aroclors 1221, 1232, 1242, 1248 and 1254 Intermediate Stock Solutions:

Intermediate stock solutions of each of the aroclors are prepared by diluting 1mL of the primary standards (at 1000ug/mL concentration) to 100mLs using hexane. There will be one intermediate solution for each of the aroclors listed above, each at a concentration of 10µg/mL.

Please note that the following preparation procedures pertain to the use of the primary stock standards listed in **Sections 7.3** and **7.4**. Different preparation schemes are needed if different stocks are used.

7.7 Working Standards for Aroclor 1221, 1232, 1242,1248, 1254 and1660.

The working 5 levels calibration standards for each of the individual Aroclors is prepared as follows:

Level 5: 8mL of the intermediate stock Aroclor prepared **in Section 7.6.2**. and 2mL of the intermediate surrogate standard prepared in **Section 7.6.1**.are diluted to 50mL using hexane.

Note: The preparation volumes could be adjusted to make smaller or larger volume of standards as long as the final working concentrations are the same.

Level 4: 25mL of level 5 are diluted to 50mL using hexane.

Level 3: 25mL of level 4 are diluted to 50mL in hexane

Level 2: 25mL of level 3 standard is diluted to 50mL using hexane.

Level 1: 25mL of the level 2 standard are diluted to 50mL using hexane.

7.7.1 The 5 levels of Aroclor 1660 standards contain the following (concentration in ug/mL):

	Aroclor 1016	Aroclor 1260	TCMX	DCB
Level 1	0.1	0.1	0.005	0.01
Level 2	0.2	0.2	0.01	0.02
Level 3	0.4	0.4	0.02	0.04
Level 4	0.8	0.8	0.04	0.08
Level 5	1.6	1.6	0.08	0.16

7.7.2 The working 5 level of each of Aroclors contain the following (concentration in ug/ml):

	Aroclor	TCMX	DCB
Level 1	0.1	0.005	0.01
Level 2	0.2	0.01	0.02
Level 3	0.4	0.02	0.04
Level 4	0.8	0.04	0.08
Level 5	1.6	0.08	0.16

7.7.3 Second source standards:

The second source standards are obtained from Supelco. All Aroclors are individually prepared at 0.4 ug/mL.

Please note that surrogate standards may or may not be added to the second source standards.

All of the above working standards are logged into the Pesticide Working Standard Logbook and labeled as PW*yymmddX*

where: PW = Pesticides/PCB working standard
yymmdd = date the standard is prepared
X = the order the standard is prepared on that date, in alphabetical order

All working standards are stored in amber containers under refrigeration at 4°C or below. The standards are stored in separate location from the samples and/or extracts to minimize cross contamination.

The expiration date for the standards is six months from the date of preparation or whenever the primary standard expires, whichever comes first.

8. Procedure

8.1 Preparation – The methods in USEPA SW-846 for sample extraction are as follows:

Method 3510 (SOP# 50.0022) extracts aqueous samples for water-insoluble and slight water-soluble organics. The samples are serially extracted with methylene chloride using a separatory funnel.

Method 3520 (SOP# 50.0025) extracts aqueous samples for water-insoluble and slightly water-soluble organics. The samples are placed in continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0019) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using 1:1 (v/v) methylene chloride/acetone in a soxhlet extraction.

Method 3550 (SOP# 50.0016) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble organics. The samples are mixed with anhydrous sodium sulfate to form a free-flowing mixture, then extracted using ultrasonic extraction in 1:1 (v/v) methylene chloride/acetone.

Method 3570 (SOP# 50.0100) is the extraction of Pesticides in soil, sediment, tissues, biota and any sample considered solid. A 2-20gram sample is solvent extracted first with acetone, and then with hexane by either a manual shake approach or via rotation or spinning of the sample.

In addition, clean-up procedures are employed to remove co-eluting interferences. The lab uses GPC clean-up (Method 3640) and sulfur clean-up (Method 3660).

8.2 Instrument conditions

The GC operating conditions are as follows:

Carrier Gas	Helium (99.999%)
Column Flow	5-10 mL/min., independent of temperature
Make-up Gas	5% methane/95% argon (P-5)
Make-up Gas Flow	80-100 mL/min.

Injector	Split-less
Injector Temperature	200°C
Initial GC Temperature	120-170°C
Initial GC Hold	1 min.
Temperature Ramp	6-8°C/min
Final Temperature	265-300°C
Final GC Hold	2-15 min.
Detector Temperature	300°C

8.2.1 Optimize GC operating conditions for analytes separation and sensitivity. Once optimized the same conditions must be used for analysis of all standards, samples, blanks and MS/, MSD.

The auto-sampler makes 2µl injection for the analysis of all standards and sample extracts.

8.2.2 Preventative Maintenance.

The injector septum is replaced every time the instrument is set up to perform a sequence of analyses when a leak develops, or when initial and/or continuing calibrations fail to meet the method requirements.

The injection liner is replaced every time the instrument is set up to perform a sequence of analyses, when a leak develops or when initial and/or continuing calibrations fail to meet the method requirements. The gold seal will be replaced and the columns will be trimmed every time before a new calibration is run.

The column will be replaced if standard chromatograms show excessive peak tailing or initial and/or continuing calibrations repeatedly fail to meet the method requirements.

8.2.3 Major instrument maintenance must be documented in the LIMS maintenance logbook, regular (daily) maintenance can be documented in Instrument Run Logbook.

8.2.4 Initial calibration is performed by analyzing 5 level standards of the Aroclor 1660, and at least one level (midpoint) of the following:

1 Aroclor 1221
2 Aroclor 1232
3. Aroclor 1242
4. Aroclor 1248
5. Aroclor 1254
6. Aroclor 1660

All analytical runs need to be documented in the appropriate Instrument Run Log. In addition to listing the data file, the associated working standard ID and standard name should be noted.

The chromatograms for each of the target Aroclor standards are presented in **figures 1 - 7** for both the RTXCLPest and RTXCLPest II columns.

8.2.5 From the initial calibration, the following parameters are determined:

8.2.5.1 Retention time and retention time window:

The retention time window is established by evaluating the retention time of the Aroclor 1260 standards (at 1ug/mL) that were analyzed over a 72-hour interval. Please note that the laboratory does not need to perform retention time window studies for the other Aroclors.

From the standard analysis, three peaks are selected for each of Aroclor 1016 and Aroclor 1260. The retention time windows for the six peaks are determined as ± 3 times the standard deviation from the 72-hour interval. If the standard deviation is zero, a default standard deviation of 0.01 minutes will be used, per SW-846 8000C, Section 11.6.3.

The retention time windows are established by centering the ±3 standard deviation around the retention time of the six peaks in the mid level standard that was analyzed as part of the 5 level initial calibration.

It is Mitkem's experience that the GC retention times operating under EPC conditions is very tight. Using the above approach in determining retention time windows at times has resulted in extremely narrow retention time windows. This may result in false negatives due to slight retention time shifts, especially when sample extracts are subjected to co-eluting interferences. In such cases, the SW-846 default standard deviations will be used.

For the surrogate standards, the retention time window is established using the same approach as the multi-component PCBs. The above retention time window criteria also apply.

8.2.5.2 Mitkem chooses to use external quantitation for instrument calibration and sample quantitation. The following equation is used to calculate the calibration factor (CF):

$$CF = \frac{\text{Peak area (or height) of standard}}{\text{Mass injected (ng)}}$$

The above equation is used to determine the calibration factor for both the single component and multi-component standards. Please note that either peak area or peak height can be used for the calculation.

The lab chooses to use peak height for single component analytes such as the surrogate compounds and for the multi-component PCB.

In calibrating for the Aroclors, the laboratory selects three peaks that are characteristic of each of the Aroclors. The analyst will choose peaks in each Aroclor standards that are at least 25% of the height of the largest Aroclor peak. Also, it is preferable that one of the three peaks selected is unique to that Aroclor.

8.2.5.3 Linearity - linearity is used to evaluate the dynamic range of the calibration factor for each of the single component surrogate standards. Linearity, as measured by the % Relative Standard Deviation (RSD) is calculated using:

$$\% \text{ RSD} = \frac{SD_{CF}}{CF_{av}} \times 100$$

Where: $SD_{CF} = \sqrt{\sum (CF_i - CF_{av})^2 / (n-1)}$
CF_{av} = average calibration factor
CF_i = calibration factor
n = total number of values = 5

Please note that there are three sets of calibration factors for each of the Aroclors.

8.2.5.4 <u>%RSD</u> Acceptance criteria for linearity:

The acceptance limit for linearity check is that the % RSD for the <u>individual Aroclor calibration factors must</u> be less than or equal to 20%. If this condition is met, the instrument is determined to be linear within the calibration range.

Given the large number of <u>individual compound</u> calibration factors, it is likely that some Aroclors may exceed the 20%

acceptance limit. For Aroclors that exceeded the 20% limit, the linearity check is still acceptable if the following least square regression conditions are met:

8.2.5.5 Alternate Least Square Regression criteria for Linearity:

The analyst may employ a regression equation for the Aroclor(s) that do(es) not pass using the earlier approach. The regression will produce the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where y = instrument response (peak area or height)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is not included as the Sixth calibration point and that the above equation is not forced through the origin. The linear regression is deemed acceptable if the correlation coefficient $r \geq 0.995$ ($r^2 \geq .0.990$)

8.2.6 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. The calibration is performed by analyzing standard Aroclor 1660 standard prepared in **Section 8.1.8**. The acceptance criteria are as follows:

The calculated value of the analyte in the ICV should be 80 – 120% of the expected value. If the ICV does not meet the criteria, see corrective action guidelines in **Attachment 1** of this SOP. Results of this evaluation should be documented in the Instrument run log.

If the above criteria are not met, the analyst must evaluate the integrity of the primary and confirmation standards. If needed, re-preparation and re-analysis of the initial calibration is required.

8.2.7 Initial calibration acceptance criteria must be met before any sample, blanks or LCS are to be analyzed.

8.2.8 Corrective Action for Initial Calibration – see **Attachment 1** for corrective action guidelines and documentation.

8.2.9 Original initial calibration raw data must be archived in the company organics analysis calibration (OCAL) database. See Mitkem SOP # 10.0009 (Application Xtender) for scanned archive information.

8.3 Continuing Calibration Verification and Sample Analysis – Method 8082 requires the analysis of Aroclors 1016/1260 as the continuing calibration verification (CCV) every time samples are to be analyzed to ensure that the GC/ECD system continues to meet instrument sensitivity and linearity requirements. Mitkem may also analyze Aroclors 1242, 1248 and 1254 as part of the calibration verification

8.3.1 Frequency of Continuing Calibration (CCV)- The CCV must be performed if the instrument has been idle for more than 12 hours. A continuing calibration standard must also be injected at intervals of not less than once every twenty field samples (the method recommends the frequency to be once every ten samples, to minimize reanalyses due to unacceptable CCVs) and at the end of the analysis sequence.

DoD requires CCV standards must be injected at intervals of not less than once every ten field samples.

8.4.1 The continuing calibration is performed using the Level 3 (0.4 ug/ml) Aroclor 1660 standard (as well as Aroclors 1242, 1248 and 1254 where appropriate). While the method suggests alternating the use of high and low calibration mixtures, Mitkem performs the continuing calibration using the mid-point standard. When the additional Aroclors 1242, 1248 and 1254 are analyzed, they will also be evaluated using the same approach as Aroclors 1016/1260.

8.3.2 Evaluation of the Calibration Verification:

8.3.2.1 *% Difference:* Calculate the % difference between the continuing calibration CF and those from the most recent initial calibration.

The % difference is determined as follow:

$$\% \text{ Difference} = \frac{CF_c - CF_i}{CF_i} \times 100$$

In which:

CF_c = calibration factor from continuing calibration
CF_i = mean calibration factor from the most recent initial calibration

8.3.2.2 *% Drift:* Use % drift when using linear regression calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

In which: $Conc_c$ = concentration obtained from continuing calibration
$Conc_t$ = theoretical concentration of standard

8.3.2.3 *% Difference (or % Drift)* between the continuing calibration factor and that of the initial calibration should be no greater than ± 20% at least on one column. If this criterion is met then the calibration has been verified and sample analysis can proceed. The analyst should mark whether the CCV passed on one or both columns in the instrument logbook. Checkmarks or the term "OK" are acceptable entries .

8.3.2.4 Comparison of the retention time window: the retention time of each aroclor and surrogate peak in the calibration standards should fall within the retention time window established from the initial calibration. Due to the need to perform routine column maintenance (clipping off a small length of the guard and/or analytical columns), the retention time might be shortened. In such cases, either the RT window is adjusted to agree with the calibration verification retention times or new retention times are established using the calibration verification standard.

8.3.3 Corrective Action for opening CCV- The %D (or % Drift) between the individual compound calibration verification factors and those from the initial calibration should be no greater than ±20% on at least one column. If this criterion is exceeded for any peak, the calibration has not been verified.

If the continuing calibration does not meet the criteria, a second injection of the calibration verification is allowed. If the second injection fails, the analytical sequence is stopped. Corrective actions must be performed. This may include preparing new standards, performing inlet maintenance and/or GC column maintenance. If the response of the analyte is still not within the above criteria, then a new initial calibration must be performed.

8.3.4 Corrective Action for closing CCV: When a calibration verification standard fails to meet the QC criteria on both columns, all samples that were injected after the last compliant standard must be evaluated to ensure the data is valid. The analyte(s) which fail(s) the 20%D must be addressed if the analyte affected is one that was detected in project sample(s). The analyst needs to note all QC issues in the instrument run

log and on all associated project data review checklists. This information needs to be addressed in the associated project(s) narrative. See **Attachment 1** for corrective action guidelines and documentation.

8.3.4.1 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**), and the analyte **was not** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, the group of samples is deemed acceptable and does not require re-analysis.

8.3.4.2 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on both columns, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards, then re-injection of all samples are required to ensure accurate quantitation.

8.3.4.3 If the non-compliant standard analyzed **after** a group of samples exhibits an elevated response for an analyte that is greater than 20% D (**positive bias**) on one column, and the analyte **was** detected in the group of samples analyzed between the compliant and non-compliant bracketing standards,and the associated samples are detects, the reported values must come from the compliant column, even if this is the higher result.

8.3.4.4 If the non-compliant standard analyzed **after** a group of samples exhibits a lower response for an analyte that is greater than 20% D (**negative bias**), then re-injection of all samples are required to ensure accurate quantitation.

Sample injections may continue as long as the calibration verification standards meet the instrument QC requirements.

All standard analyses are to be documented in the appropriate Instrument Run Log with associated working standard IDs.

8.3.5 The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Target software. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample. Both retention time and pattern are important when determining PCBs in a sample.

It is important that if Aroclors are detected in the samples, the analysis of these sample extracts should be accompanied by the appropriate Aroclor calibration verification standard as part of the analytical sequence. If this is not done, the sample extracts have to be re-injected with the appropriate Aroclor standards to ensure the PCB pattern is similar.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. In these instances, the analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclors.

8.3.6 The concentration of the target compounds and surrogates are calculated separately for each of the three Aroclor peaks for both columns using the following equation.

For aqueous samples, concentration *ug/L* = $\frac{(Ax)\ (Vt)\ (DF)}{(CF)\ (Vo)\ (Vi)}$

For soil samples, concentration *ug/kg* = $\frac{(Ax)\ (Vt)\ (DF)}{(CF)\ (Ws)\ (Vi)\ (S)}$

in which: Ax = area/height of the peak for the compound to be determined

CF = calibration factor
Vo = volume of water extracted, in mL
Ws = weight of soil sample, in gram
Vi = volume of extract injected, in µL
Vt = volume of extract, in µL
DF = dilution factor
S = solid content, expressed in decimal

For the multi-component Aroclors, the analyte concentration is calculated by taking the average of the concentration determined for each of the three peaks selected for that analyte.

8.3.7 Manual integration will be performed if needed and documented according to the current revision of SOP Number 110.0008. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. The analyst will further initial and date the manual integration with the proper reason code per SOP No.110.0008.

8.3.8 The concentrations of the surrogate analytes are calculated and reported separately for each of the two columns. The acceptance limits for the recoveries are discussed in **Section 10**.

8.3.9 The analyte recoveries of the LCS and matrix spike compounds are determined and reported. The acceptance limits for the recoveries are discussed in **Section 10**.

8.4 Analytical sequences are summarized as follows:

Order	Sequence	Comment
1	AR1232L3	
2	AR1221L3	
3	AR1242L1	Level 3 only for AR1232 and AR1221 are run unless this Aroclor is detected in samples.
4	AR1242L2	
5	AR1242L3	
6	AR1242L4	
7	AR1242L5	
8	AR1248L1	
9	AR1248L2	
10	AR1248L3	
11	AR1248L4	
12	AR1248L5	
13	AR1254L1	
14	AR1254L2	
15	AR1254L3	
16	AR1254L4	
17	AR1254L5	
18	AR1660L1	
19	AR1660L2	
20	AR1660L3	
21	AR1660L4	
22	AR1660L5	
22	AR1660SS	Second source ICV

23	BIBLK01	Instrument blank
24 25 26	Blank LCS Sample extracts	
27 28 29 30	AR1242M01 AR1248M01 AR1254M01 **AR1660M01**	calibration verification
31	BIBLK02	
32	Sample extracts	
33 34 35 36	AR1242M02 AR1248M02 AR1254M02 **AR1660M02**	
37	BIBLK03	
38…	Sample extracts…	Sequence continues as the same as above as long as calibration verifications meet the acceptance criteria.

9. Data Reduction and Calculations

9.1 Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight for solid samples.

Results are reported to two significant figures using the USEPA guidelines in rounding up or down. For solid samples, results are reported in dry weight unless otherwise specified.

For 8082 analyses, PCB aroclors are reported if the determined concentration is above the project reporting limits on both columns. No J flagged results are reported.

9.2 Soil concentrations are calculated using dry weight basis. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids.

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
WW = Sample weight (g) before drying

9.3 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

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

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.4 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100\%$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of the instrumentation and have performed a proficiency test with acceptable accuracy and precision results.

10.2 Method blanks. A method blank is an aliquot of a clean reference matrix (reagent water for water samples, or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. A Method blank is prepared and analyzed with every batch of 20 samples or less. It is used to determine the level of contamination associated with the analytical processing and analysis of the samples. The Method blank MUST be analyzed on the same instrument as the associated samples.

See **Attachment 1** for additional corrective action.

10.2.1 The recovery of the surrogates must be within the calculated acceptance limits discussed in **Section 10.6**.

10.2.2 The concentration of target compounds in the method blank must be ≤ to ½ CRDL or RL.

10.2.3 Corrective action for method blank contamination involves determining the source of the contamination and possibly re-extracting the entire batch.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a volume or weight of a clean reference matrix (organic-free water or Ottawa Sand) that is spiked with Aroclors 1016 and 1260 and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 The recovery of the surrogates must be within the calculated acceptance limits discussed **Section 10.6**.

10.3.2 The LCS recovery is evaluated against the established in-house limits. Any analyte/analytes not meeting the criteria require corrective action. See **Attachment 1**. For DoD projects, see **Attachment 2**, DoD Tables D-14 and 15.

10.3.3 Advisory RPD limits are set at 30% when duplicate LCSs are performed.

Any sample(s) that is/are associated with a non-compliant LCS may require re-extraction and re-analysis. See **Attachment 1** for corrective action guidelines.

10.4 Duplicate Matrix Spikes –Matrix spikes and matrix spike duplicates are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix at a frequency of one set per 20 samples.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and one duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform a matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above.

10.4.2 Control limits for Method 8082 projects are the same as discussed in **Sections 10.3.2 and 10.3.3.** DoD requires 30% RPD for all matrices. See **Attachment 2**, DoD Table B-2.

Depending on sample matrix, chromatographic based analysis such as Method 8082 is susceptible to co-eluting interferences resulted in high biased values. Thus, the control limits for the duplicate matrix spikes should be used as guidelines. The determination of outliers should be addressed in the project narrative to advise the data users of potential matrix related interferences.

10.5 Re-analysis at dilution - Any target compounds that are determined above the instrument calibration range will require reanalysis at dilution. A notation for the need to perform a dilution next to the sample run in the instrument logbook is needed to document the results. An entry such as " rerun at 4x " is acceptable.

The dilution is performed by taking an aliquot of the extract and diluting it to a pre-determined volume using hexane. The analyst should not over-dilute the extract. The analytes that trigger the need for dilution should be determined in the diluted analysis at a concentration above that of the midpoint calibration standard.

10.5.1 When reporting diluted results the following guidelines should be followed. If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis may be reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the DL suffix appended to both the client and sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and all compounds detected in the more diluted (DL) analysis are qualified with the "D" flag.

10.5.2 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the calibration range of the instrument, the initial analysis may be performed at dilution. If this analysis is acceptable (compounds at or above the mid-point calibration) then a less dilute analysis is not required. The sample ID is not changed by adding a DL suffix but the 'initial analysis at dilution' is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

10.5.3 If the initial analysis fails QC criteria, it may be reported if specified by the project or client. If only the dilution is reported, the DL suffix is not added and the dilution is noted on the data review checklist submitted with the data for review as above.

10.6 Surrogate recoveries - The recovery of each surrogate compound in all samples, blanks, MS/MSD and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.6.1 Acceptance criteria for surrogate recovery.

For 8082 projects the percent recovery of each of the surrogate compounds in method blanks and LCS must be within the in-house acceptance criteria window. The current in-house acceptance criteria for each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS are listed under the option SPEC for the Omega LIMS Testcode.

In-house limits may be lab generated or from a government /method based source such as DoD QSM. Please note that the in-house limit will be verified on an annual basis. Where lab generated limits are derived, these will be updated annually.

Surrogate spikes in matrix specific samples that fail to meet the in-house limits would indicate potential matrix effect. In general, high recovery of surrogates due to co-eluting interferences is acceptable. Low recovery of surrogates would indicate potential matrix related problems or one related to the extraction process. The analyst should confirm the sample volume extracted and the final extract volume to verify the calculations. If applicable, the analyst should consider additional cleanup procedures and re-analysis. If no appropriate cleanup procedure could be identified or re-analysis after additional cleanups failed to achieve the limit, this should be noted on the review checklist and also addressed in the narrative.

If surrogate recoveries do not meet recovery criteria see **Attachment 1** for corrective action

10.7 Annually, MDL studies are conducted to establish the limit of detection applicable to this method. Please refer to the SOP No. 80.0005 for more detail. An MDL verification at approximately 2XMDL will be analyzed after the study. MDL verification may be analyzed quarterly in lieu of the annual study.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, is reviewed for technical accuracy and completeness by the analyst. The analyst initiates a project data review checklist and documents all comments regarding analysis there. Sample preparation logs, notebooks, and instrument logs are reviewed and signed by the laboratory supervisor. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 All raw data is peer reviewed at the computer/Target level by another analyst or the lab supervisor prior to final form generation. Analysts generate all hard copy raw data and upload electronic data files to the OMEGA LIMS for reporting. Raw Data, including all support data (such as data review checklist, run-logs, work-order sheets, SDG summaries…) are brought to the data reporting department for assembly. After assembly, all data are reviewed by senior personnel (data reviewers) for quality control and completeness dependent on project specific requirements.

12. Corrective Action Procedures

Corrective actions to be implemented in the event QC results are outside of the acceptance range are covered in **Sections 8, 9, and 10**. See **Attachment 1** for routine corrective action guidelines and documentation.

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedures SOP No. 80.0007.

13. Health and Safety

The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan.

In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible. Spent vials and ampules are disposed of into a red metal drum in the Semivolatile Laboratory. See SOP No. 30.0024, Sample and Waste Disposal for more detail.

Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention; Waste Management; Definitions and Acronyms

See Sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. U.S. Environmental Protection Agency. Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Method 8082A, SW-846 Test Methods for Evaluating Solid Wastes, Final Update IV, Feb 2007.
2. "Methods Compendium for Inorganic and Organic Methods," United States Army Corps of Engineers, Appendix I, 2001.
3. "Shell for Chemical Analytical Requirements," United States Army Corps of Engineers, Appendix H, 1997 including addendum dated 1 February 2001.
4. Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

Attachments:

1. **Table 1**: List of Target Analytes.
2. **Table 2**: List of Acronyms.
3. **Figure 1**: Aroclor 1660 Standard Chromatogram and Quantitation Report.
4. **Figure 2**: Aroclor 1221 Standard Chromatogram and Quantitation Report.

5. **Figure 3**: Aroclor 1232 Standard Chromatogram and Quantitation Report
6. **Figure 4**: Aroclor 1242 Standard Chromatogram and Quantitation Report
7. **Figure 5**: Aroclor 1248 Standard Chromatogram and Quantitation Report
8. **Figure 6**: Aroclor 1254 Standard Chromatogram and Quantitation Report
9. **Attachment 1**: Corrective Action Examples and Documentation Tables
10. **Attachment 2:** DoD QC Requirements: SW 846 DoD-B, Tables B-2, D-14, D-15.

Table 1

List of Target Analytes for Method 8082

Aroclor	CAS Number
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5

Table 2

List of Acronyms

PCB	Polychlorinated Biphenyl
TCLP	Toxicity Characteristic of the Leaching Procedure
SPLP	Synthetic Precipitate Leaching Procedure
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate
GC	Gas chromatograph
ECD	Electron capture detector
GPC	Gel Permeation chromatography
CF	Calibration factor
RSD	Relative standard deviation
RT	Retention Time
EPC	Electron pressure controller
USACE	US Army Corp. of Engineers
AFCEE	Air Force Center of Environmental Excellence
CLP	US EPA Contract Laboratory Program
DoD	Department of Defense

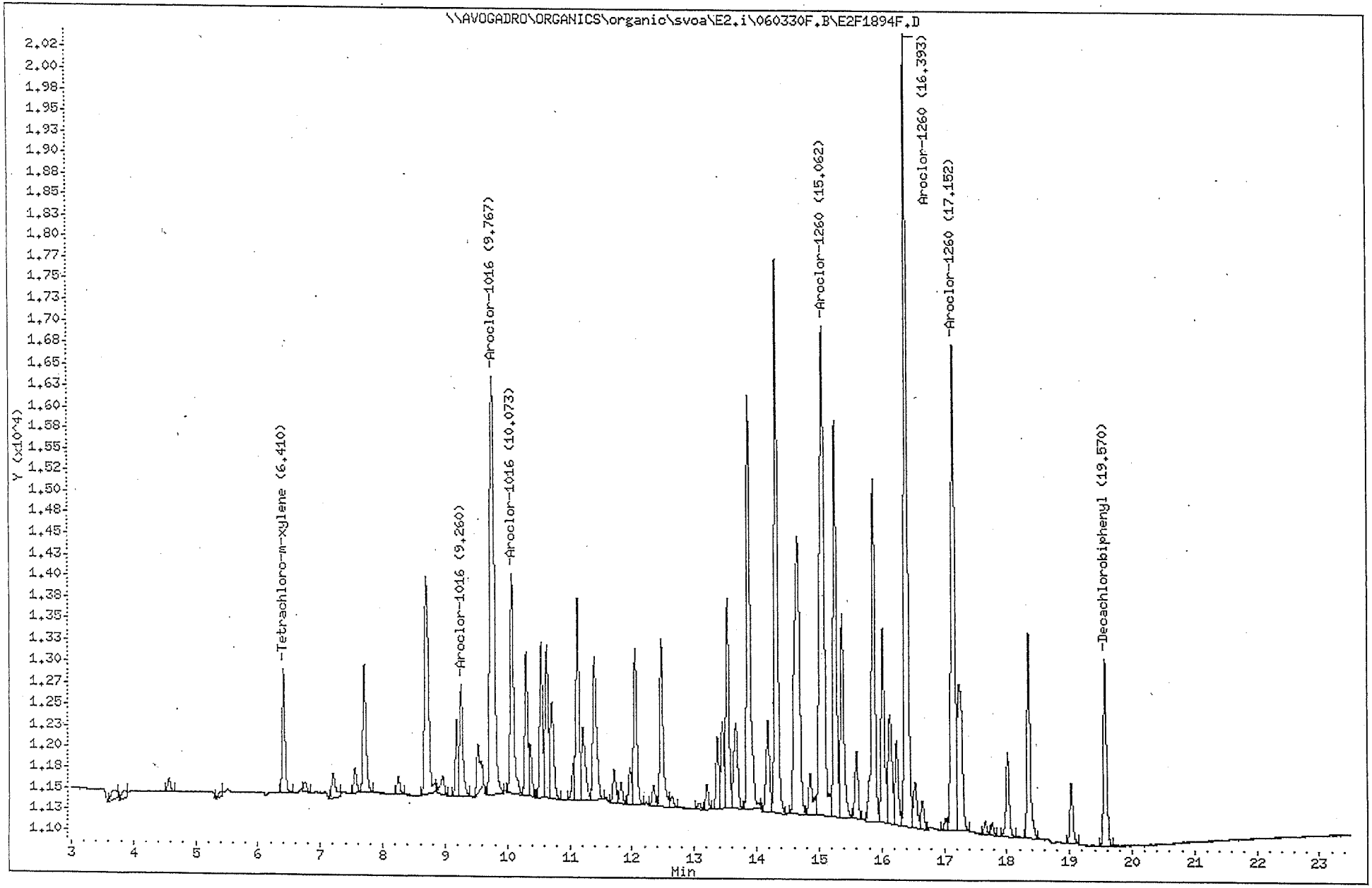
Figure 1

Aroclor 1660 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D
Date : 31-MAR-2006 02:31
Client ID: AR1660FL1
Sample Info: AR1660FL1,AR1660FL1,,ar1660.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

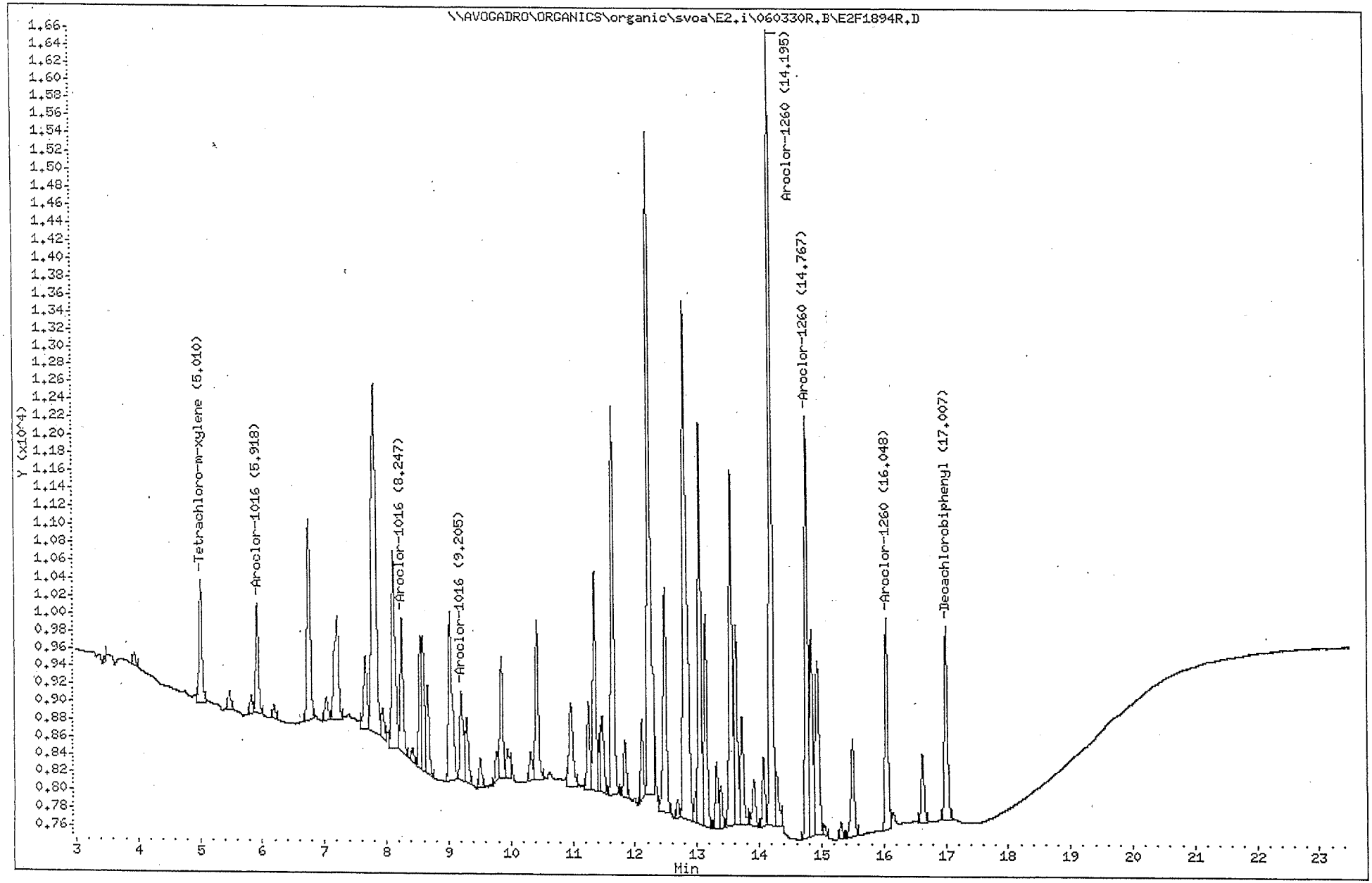
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D
Date : 31-MAR-2006 02:31
Client ID: AR1660FL1
Sample Info: AR1660FL1,AR1660FL1,,ar1660.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1894F.D
Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1894F.D
Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
Inj Date : 31-MAR-2006 02:31
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 18 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1660.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1471	0.00200	0.0022			(a)
4 Aroclor-1016					CAS #: 12674-11-2			
9.26	9.26	0.000	1314	0.10000	0.12	80.00- 120.00	100.00	(a)
9.77	9.77	0.000	4921	0.10000	0.13	327.41- 367.41	374.51	
10.1	10.1	0.000	2590	0.10000	0.12	166.80- 206.80	197.11	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2221	0.00200	0.0026			(a)
8 Aroclor-1260					CAS #: 11096-82-5			
15.1	15.1	0.000	5765	0.10000	0.13	80.00- 120.00	100.00	(a)

3/31/06

Data File: E2F1894F.D
Report Date: 31-Mar-2006 07:55

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
8 Aroclor-1260 (continued)							
16.4	16.4	0.000	9365	0.10000	0.12	151.74- 191.74	162.45
17.2	17.2	0.000	5721	0.10000	0.12	86.33- 126.33	99.24
		Average of Peak Amounts =			0.12		

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: E2F1894R.D
Report Date: 31-Mar-2006 07:55

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1894R.D
Lab Smp Id: AR1660FL1 Client Smp ID: AR1660FL1
Inj Date : 31-MAR-2006 02:31
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1660FL1,AR1660FL1,,ar1660.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:55 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 18 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1660.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1405	0.00200	0.0025			(a)
6 Aroclor-1016					CAS #: 12674-11-2			
5.92	5.91	0.010	1254	0.10000	0.12	80.00- 120.00	100.00	(a)
8.25	8.24	0.010	1526	0.10000	0.12	112.38- 152.38	121.69	
9.21	9.20	0.010	1023	0.10000	0.12	72.72- 112.72	81.58	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2198	0.00200				(a)
8 Aroclor-1260					CAS #: 11096-82-5			
14.2	14.2	0.000	8966	0.10000	0.13	80.00- 120.00	100.00	(a)

3/31/06

Data File: E2F1894R.D
Report Date: 31-Mar-2006 07:55

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
8 Aroclor-1260 (continued)							
14.8	14.8	0.000	4745	0.10000	0.13	32.61- 72.61	52.92
16.0	16.0	0.000	2397	0.10000	0.12	8.08- 48.08	26.73
		Average of Peak Amounts =			0.13		

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

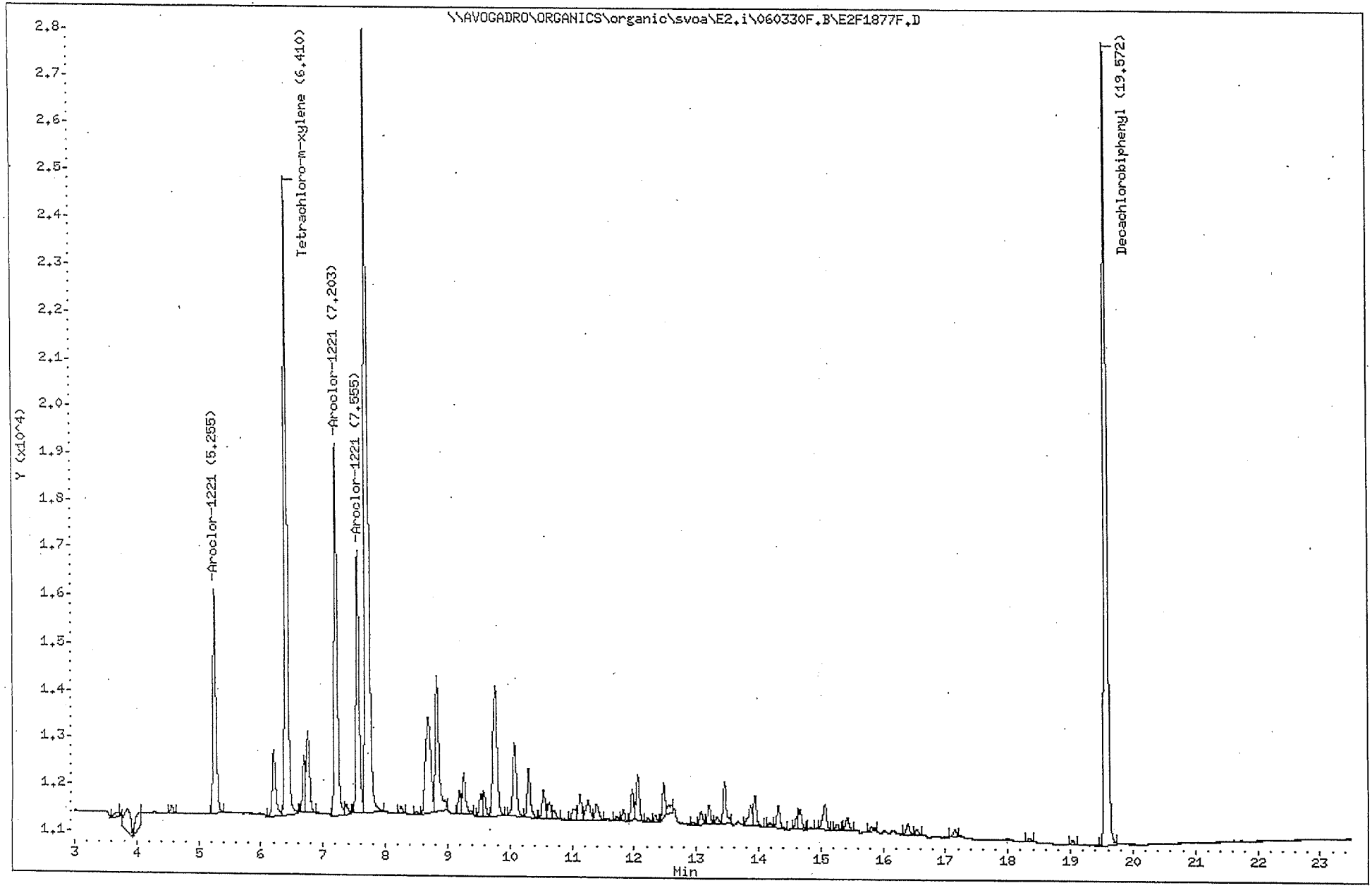
Figure 2

Aroclor 1221 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D
Date : 30-MAR-2006 18:58
Client ID: AR1221FL3
Sample Info: AR1221FL3,AR1221FL3,,ar1221.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

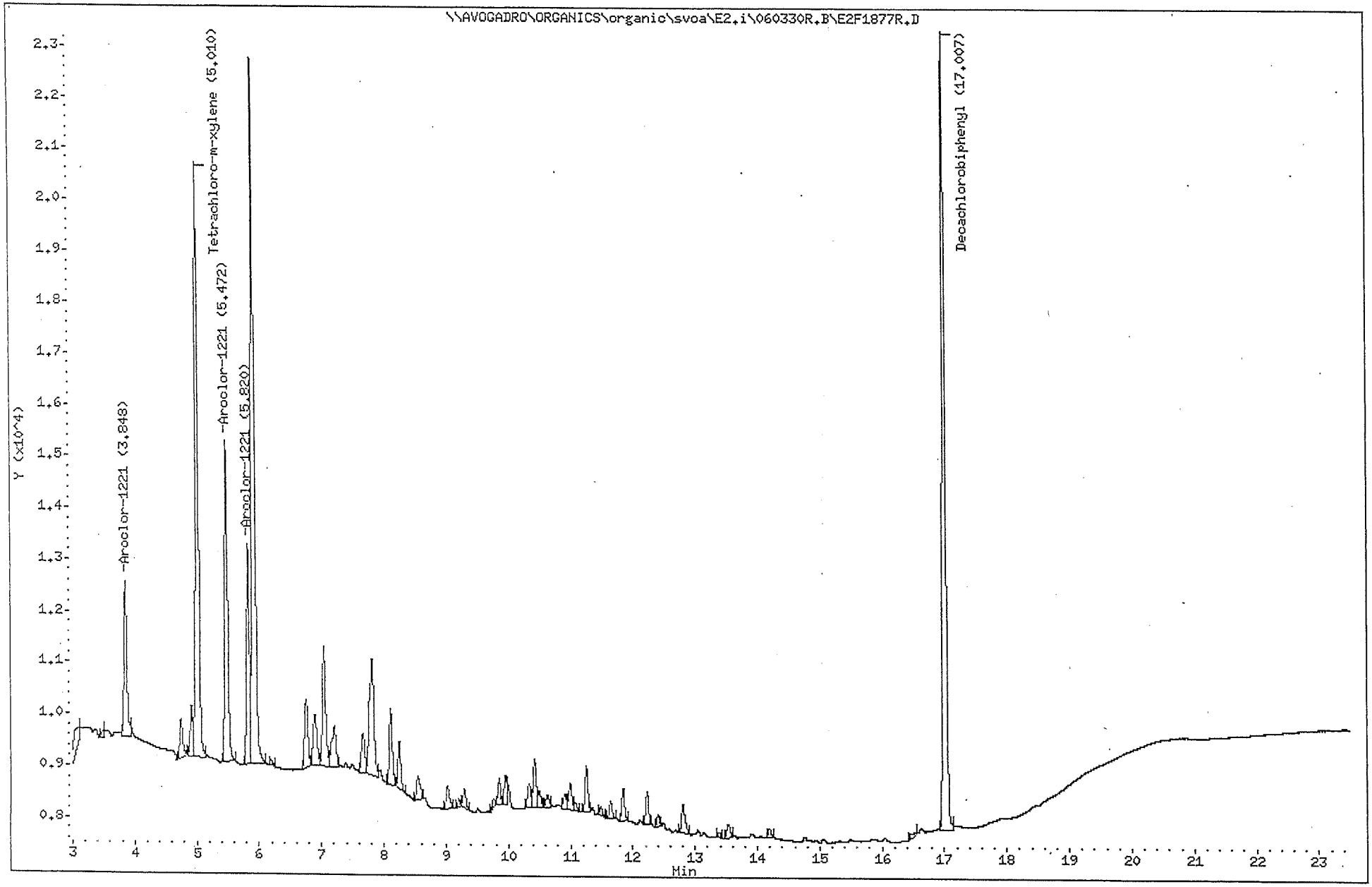
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1877R.D
Date : 30-MAR-2006 18:58
Client ID: AR1221FL3
Sample Info: AR1221FL3,AR1221FL3,,ar1221.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1877F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1877F.D
Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
Inj Date : 30-MAR-2006 18:58
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 1 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1221.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
6.41	6.41	0.000	13493	0.02000	0.020		(a)
2 Aroclor-1221					CAS #: 11104-28-2		
5.26	5.26	0.000	4734	1.00000	1.0	80.00- 120.00	100.00
7.20	7.20	0.000	7871	1.00000	1.0	146.27- 186.27	166.27
7.56	7.56	0.000	5525	1.00000	1.0	96.71- 136.71	116.71
Average of Peak Amounts =					1		
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
19.6	19.6	0.000	16954	0.02000	0.020		(a)

3/31/06

Data File: E2F1877F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1877R.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1877R.D
Lab Smp Id: AR1221FL3 Client Smp ID: AR1221FL3
Inj Date : 30-MAR-2006 18:58
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1221FL3,AR1221FL3,,ar1221.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 1 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1221.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
5.01	5.01	0.000	11534	0.02000	0.020		(a)
2 Aroclor-1221					CAS #: 11104-28-2		
3.85	3.85	0.000	3031	1.00000	1.0	80.00- 120.00	100.00
5.47	5.47	0.000	6227	1.00000	1.0	185.44- 225.44	205.44
5.82	5.82	0.000	4291	1.00000	1.0	121.57- 161.57	141.57
Average of Peak Amounts =					1		
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
17.0	17.0	0.000	15520	0.02000	0.022		(a)

3/31/06 ✓

Data File: E2F1877R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

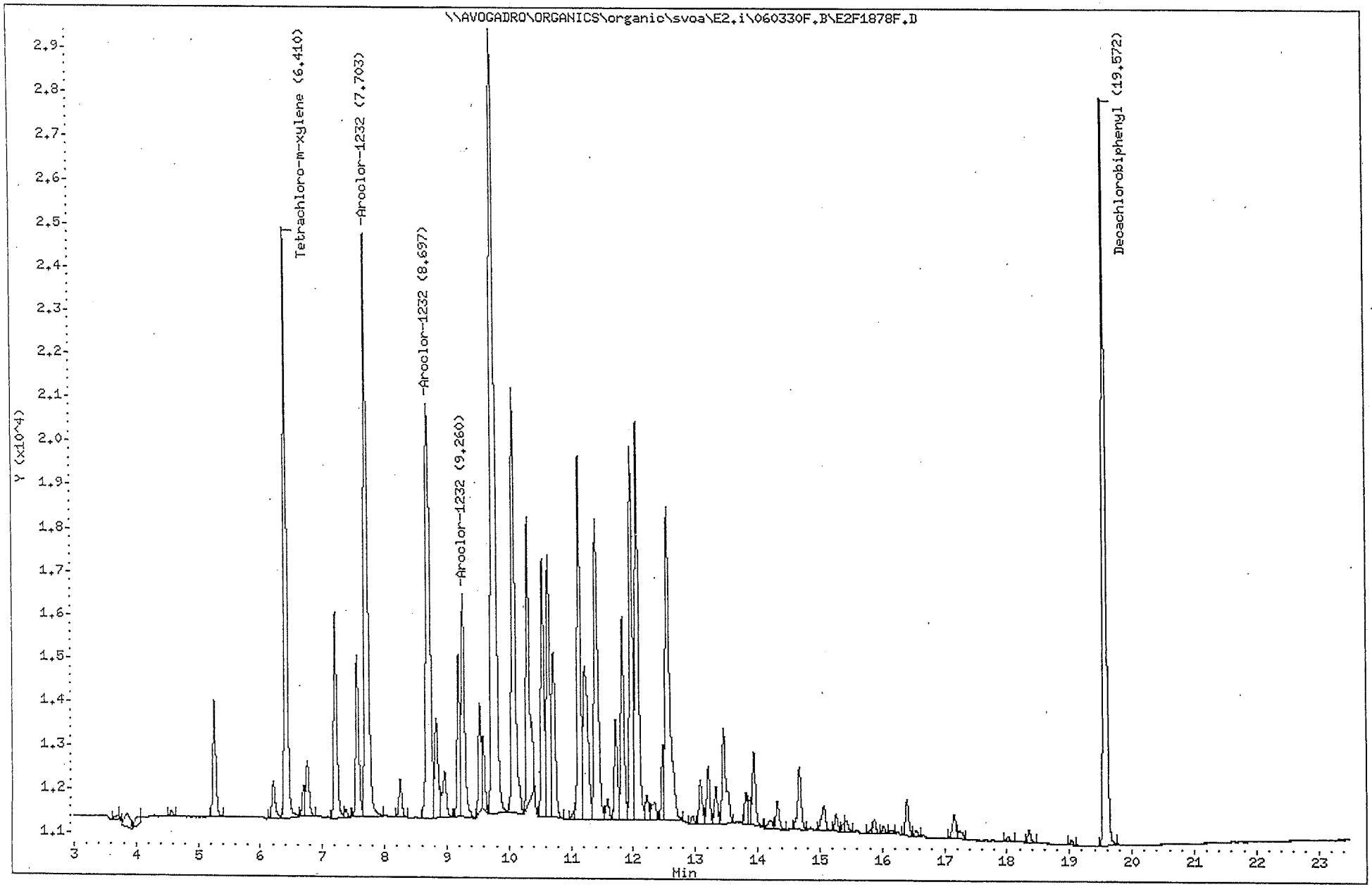
Figure 3

Aroclor 1232 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D
Date : 30-MAR-2006 19:24
Client ID: AR1232FL3
Sample Info: AR1232FL3,AR1232FL3,,ar1232.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

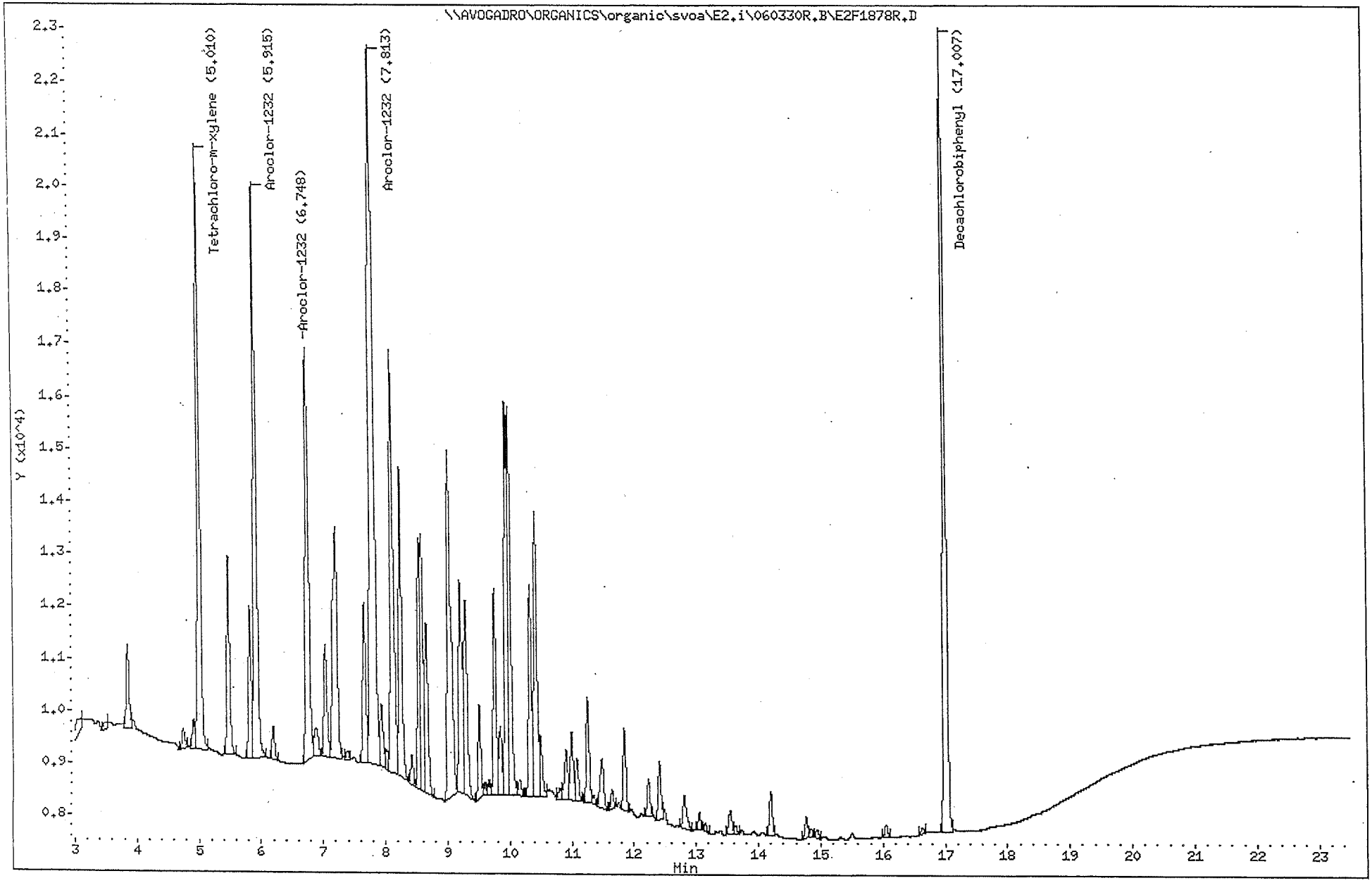
Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D
Date : 30-MAR-2006 19:24
Client ID: AR1232FL3
Sample Info: AR1232FL3,AR1232FL3,,ar1232.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1878F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1878F.D
Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
Inj Date : 30-MAR-2006 19:24
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 2 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1232.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	13570	0.02000	0.020			(a)
3 Aroclor-1232					CAS #: 11141-16-5			
7.70	7.70	0.000	13387	1.00000	1.0	80.00- 120.00	100.00	
8.70	8.70	0.000	9491	1.00000	1.0	50.90- 90.90	70.90	
9.26	9.26	0.000	5098	1.00000	1.0	18.08- 58.08	38.08	
Average of Peak Amounts =					1			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	17061	0.02000	0.020			(a)

3/31/06

Data File: E2F1878F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1878R.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1878R.D
Lab Smp Id: AR1232FL3 Client Smp ID: AR1232FL3
Inj Date : 30-MAR-2006 19:24
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1232FL3,AR1232FL3,,ar1232.sub,,
Misc Info : 1,3,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 2 Calibration Sample, Level: 3
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1232.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	11550	0.02000	0.020			(a)
3 Aroclor-1232					CAS #: 11141-16-5			
5.92	5.92	0.000	10974	1.00000	1.0	80.00- 120.00	100.00	
6.75	6.75	0.000	7851	1.00000	1.0	51.54- 91.54	71.54	
7.81	7.81	0.000	13684	1.00000	1.0	104.69- 144.69	124.69	
Average of Peak Amounts =					1			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	15283	0.02000	0.022			(a)

3/31/06

Data File: E2F1878R.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

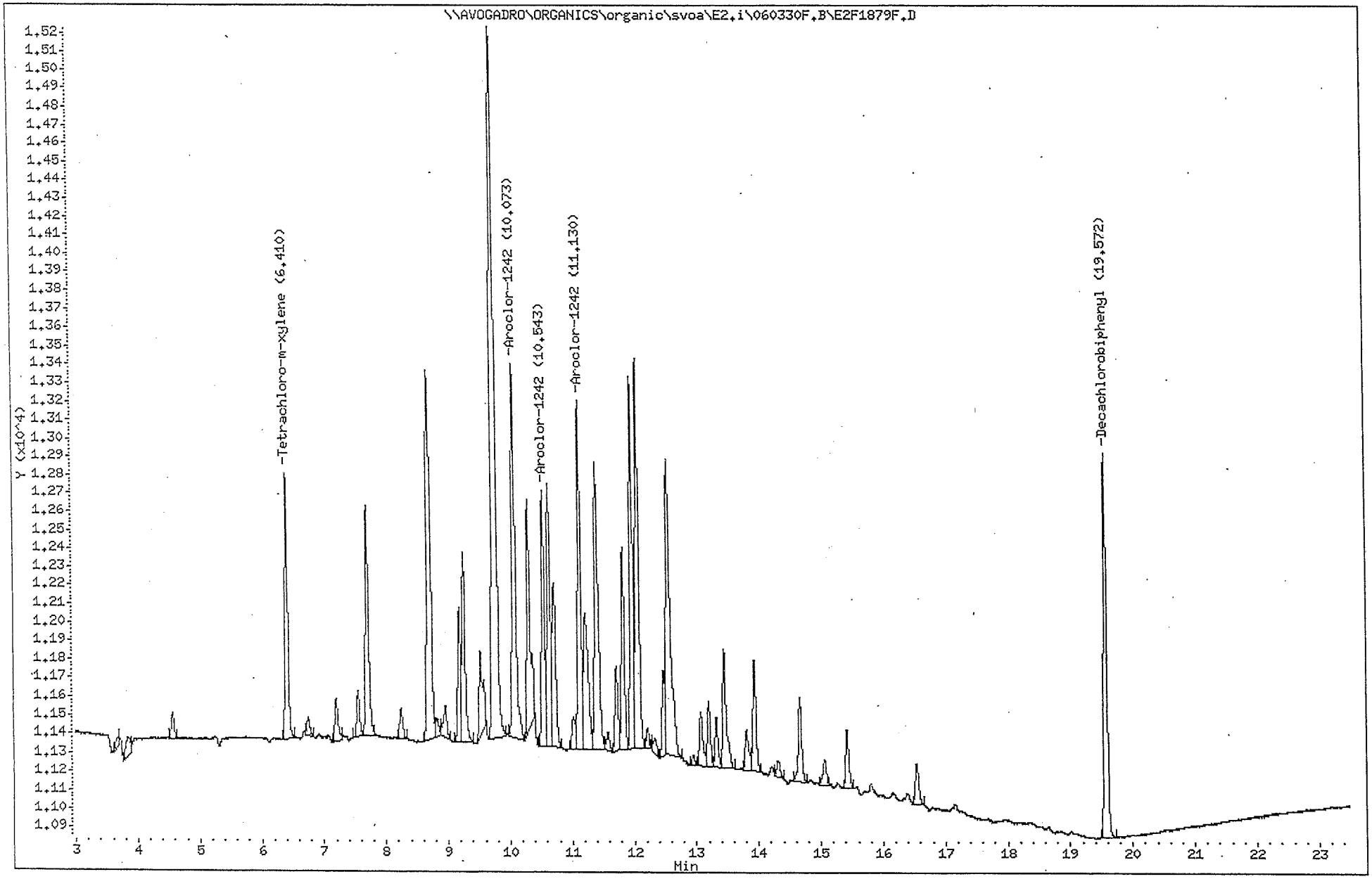
Figure 4

Aroclor 1242 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D
Date : 30-MAR-2006 19:51
Client ID: AR1242FL1
Sample Info: AR1242FL1,AR1242FL1,,ar1242.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

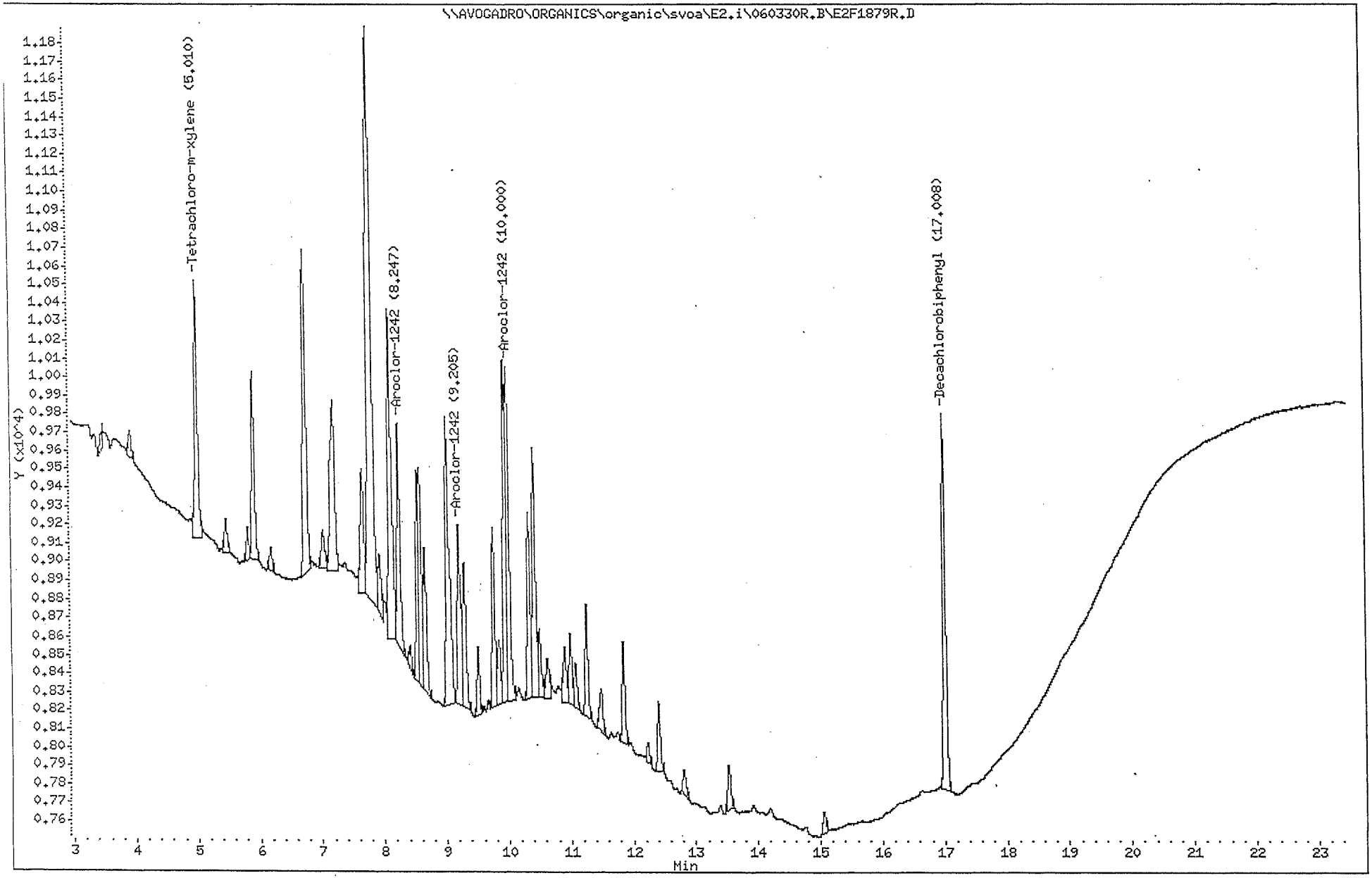
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D
Date : 30-MAR-2006 19:51
Client ID: AR1242FL1
Sample Info: AR1242FL1,AR1242FL1,,ar1242.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1879F.D
Report Date: 31-Mar-2006 07:51

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1879F.D
Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
Inj Date : 30-MAR-2006 19:51
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:51 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 3 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1242.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1442	0.00200	0.0021			(a)
5 Aroclor-1242					CAS #: 53469-21-9			
10.1	10.1	0.000	2024	0.10000	0.12	80.00- 120.00	100.00	(a)
10.5	10.5	0.000	1380	0.10000	0.12	44.06- 84.06	68.18	
11.1	11.1	0.000	1886	0.10000	0.12	71.10- 111.10	93.18	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2073	0.00200	0.0024			(a)

3/31/06

Data File: E2F1879F.D
Report Date: 31-Mar-2006 07:51

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1879R.D
Report Date: 31-Mar-2006 07:52

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1879R.D
Lab Smp Id: AR1242FL1 Client Smp ID: AR1242FL1
Inj Date : 30-MAR-2006 19:51
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1242FL1,AR1242FL1,,ar1242.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:52 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 3 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1242.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1392	0.00200	0.0024			(a)
4 Aroclor-1242					CAS #: 53469-21-9			
8.25	8.24	0.010	1190	0.10000	0.12	80.00- 120.00	100.00	(a)
9.21	9.20	0.010	967	0.10000	0.12	62.62- 102.62	81.26	
10.0	10.0	0.000	1808	0.10000	0.13	115.66- 155.66	151.93	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2030	0.00200				(a)

3/31/06

Data File: E2F1879R.D
Report Date: 31-Mar-2006 07:52

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

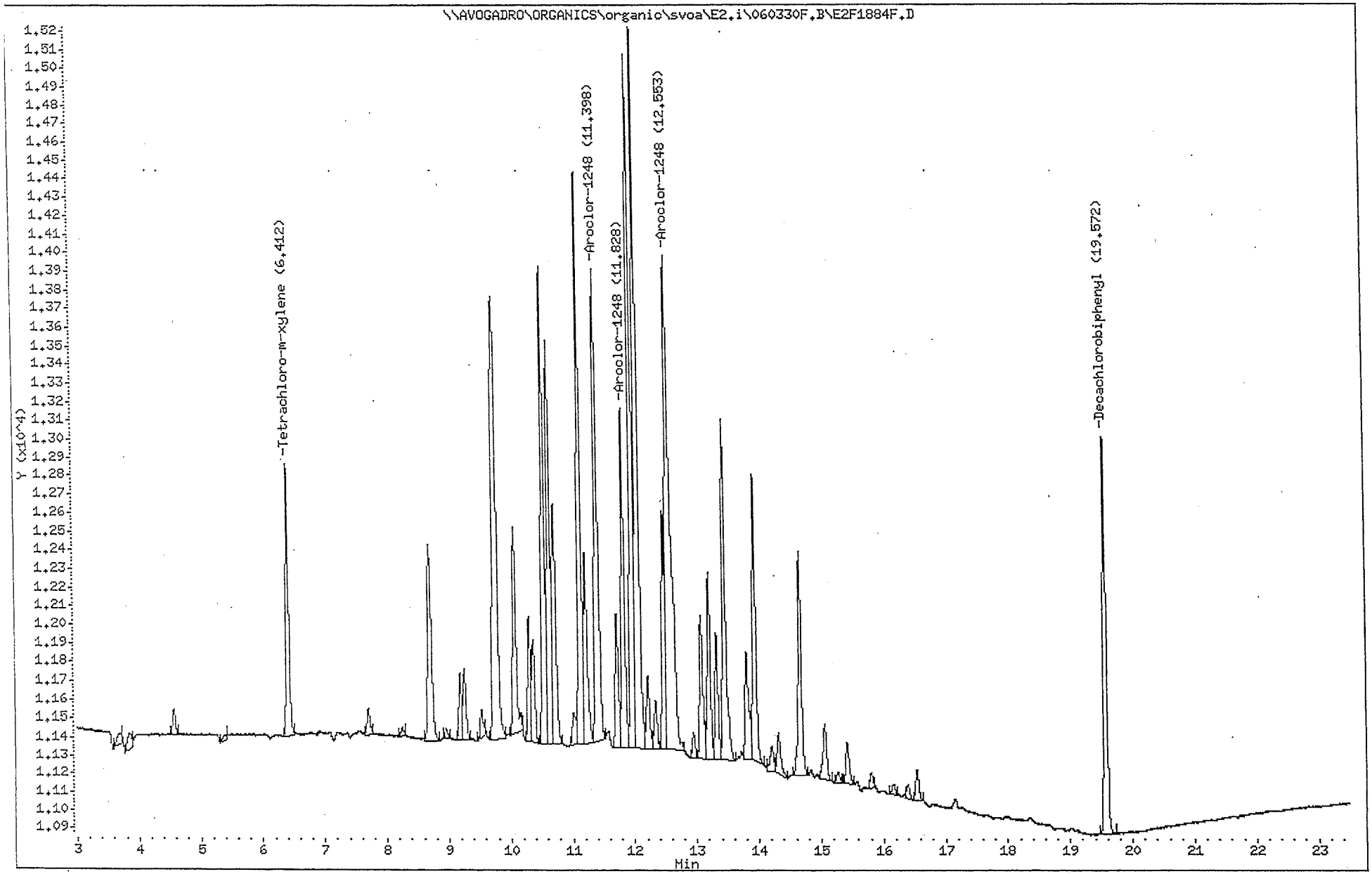
Figure 5

Aroclor 1248 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1884F.D
Date : 30-MAR-2006 22:04
Client ID: AR1248FL1
Sample Info: AR1248FL1,AR1248FL1,,ar1248.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

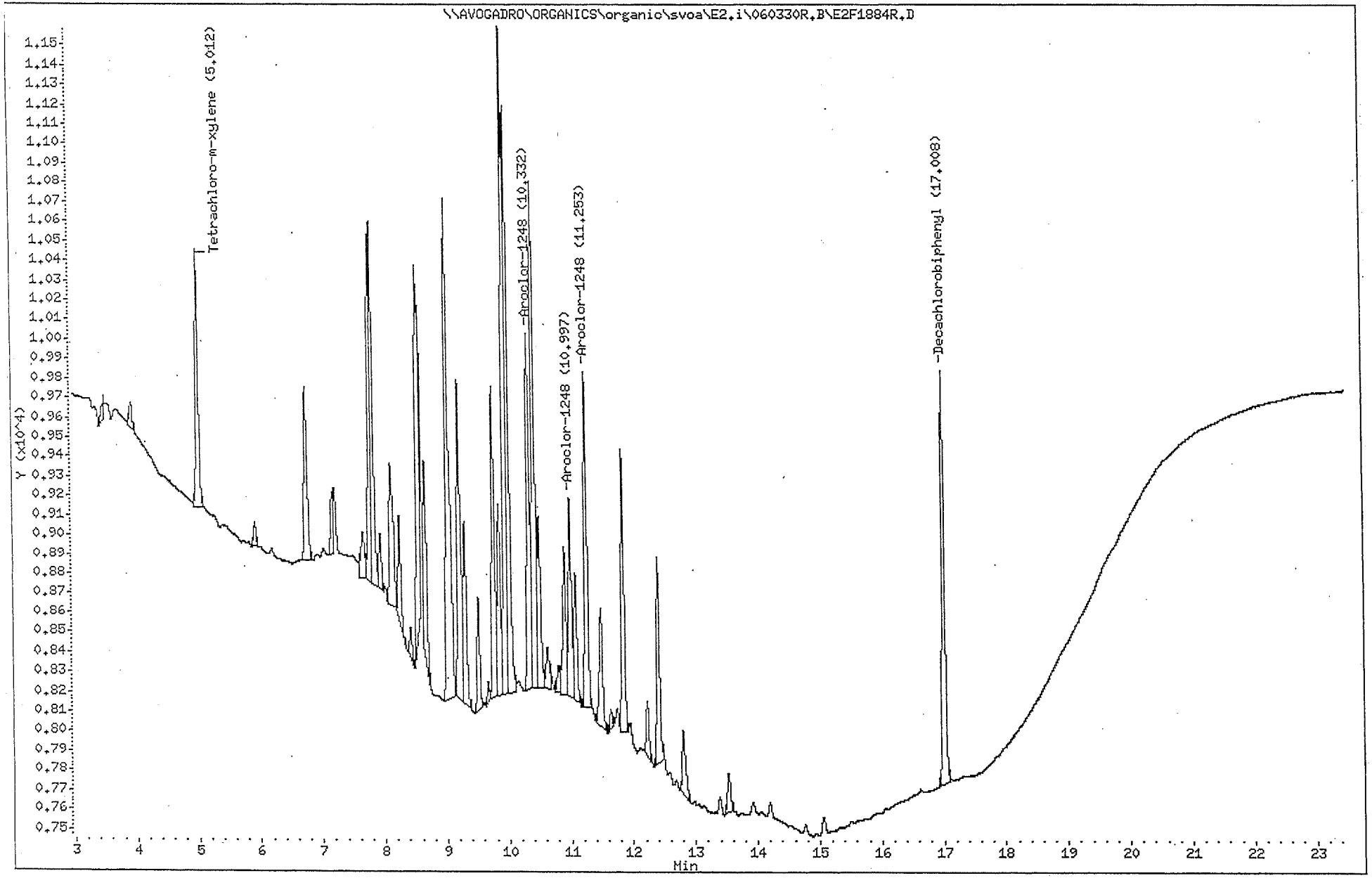
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D
Date : 30-MAR-2006 22:04
Client ID: AR1248FL1
Sample Info: AR1248FL1,AR1248FL1,,ar1248.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1884F.D
Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1884F.D
Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
Inj Date : 30-MAR-2006 22:04
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 8 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1248.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
6.41	6.41	0.000	1454	0.00200	0.0022			(a)
6 Aroclor-1248					CAS #: 12672-29-6			
11.4	11.4	0.000	2529	0.10000	0.12	80.00- 120.00	100.00	(a)
11.8	11.8	0.000	1823	0.10000	0.12	50.48- 90.48	72.08	
12.6	12.6	0.000	2646	0.10000	0.12	89.75- 129.75	104.63	
Average of Peak Amounts =					0.12			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
19.6	19.6	0.000	2141	0.00200	0.0025			(a)

3/31/06

Data File: E2F1884F.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1884R.D
Report Date: 31-Mar-2006 07:53

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1884R.D
Lab Smp Id: AR1248FL1 Client Smp ID: AR1248FL1
Inj Date : 30-MAR-2006 22:04
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1248FL1,AR1248FL1,,ar1248.sub,,
Misc Info : 1,1,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:53 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 8 Calibration Sample, Level: 1
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1248.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	1315	0.00200	0.0023			(a)
5 Aroclor-1248					CAS #: 12672-29-6			
10.3	10.3	0.000	1811	0.10000	0.13	80.00- 120.00	100.00	(a)
11.0	11.0	0.000	1016	0.10000	0.12	37.21- 77.21	56.10	
11.3	11.3	0.000	1715	0.10000	0.13	72.29- 112.29	94.70	
Average of Peak Amounts =					0.13			
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	2112	0.00200				(a)

3/31/06

Data File: E2F1884R.D
Report Date: 31-Mar-2006 07:53

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

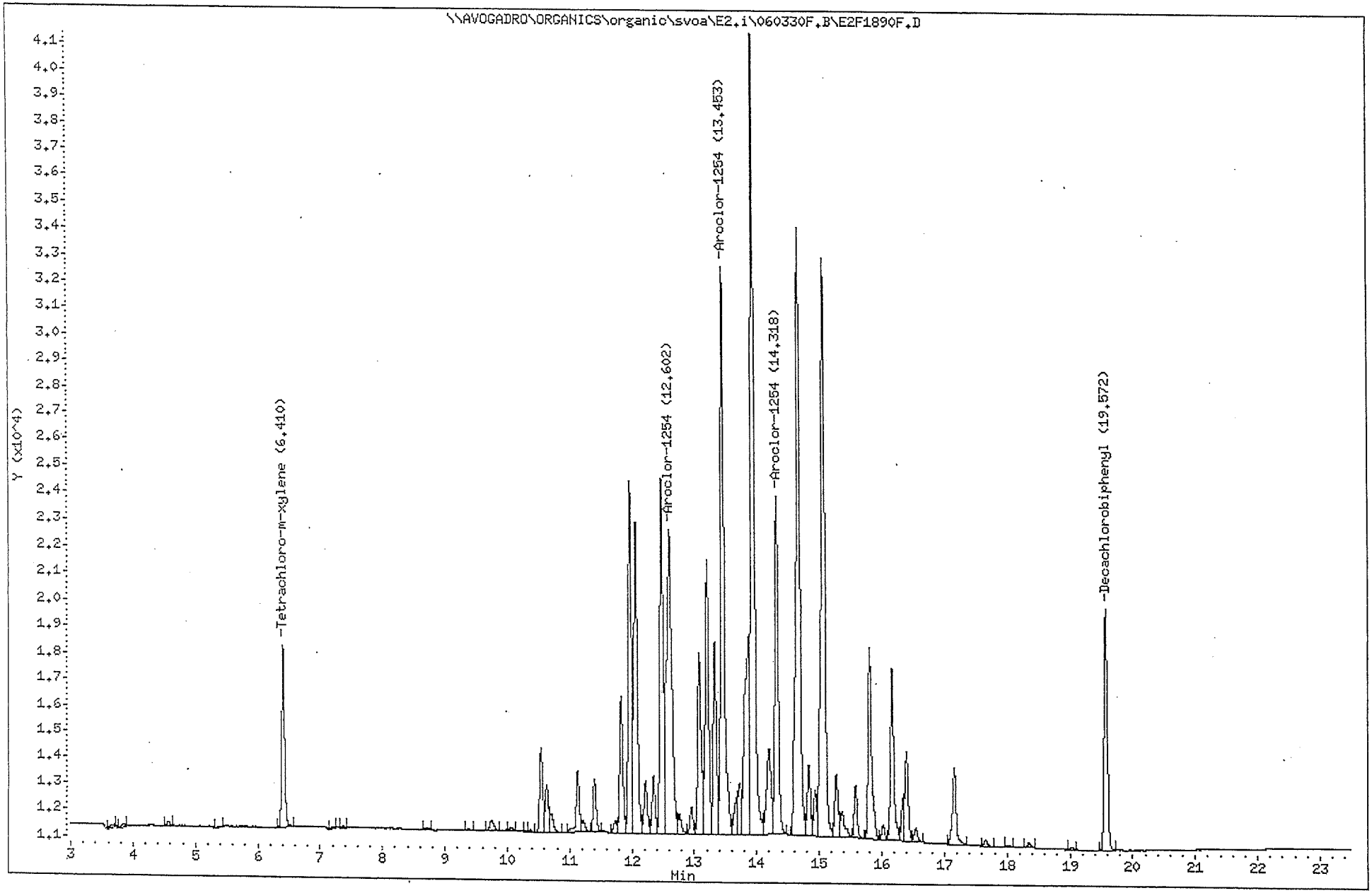
Figure 6

Aroclor 1254 Standard Chromatogram and Quantitation Report

Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D
Date : 31-MAR-2006 00:44
Client ID: AR1254FL2
Sample Info: AR1254FL2,AR1254FL2,,ar1254.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest

Instrument: E2.i

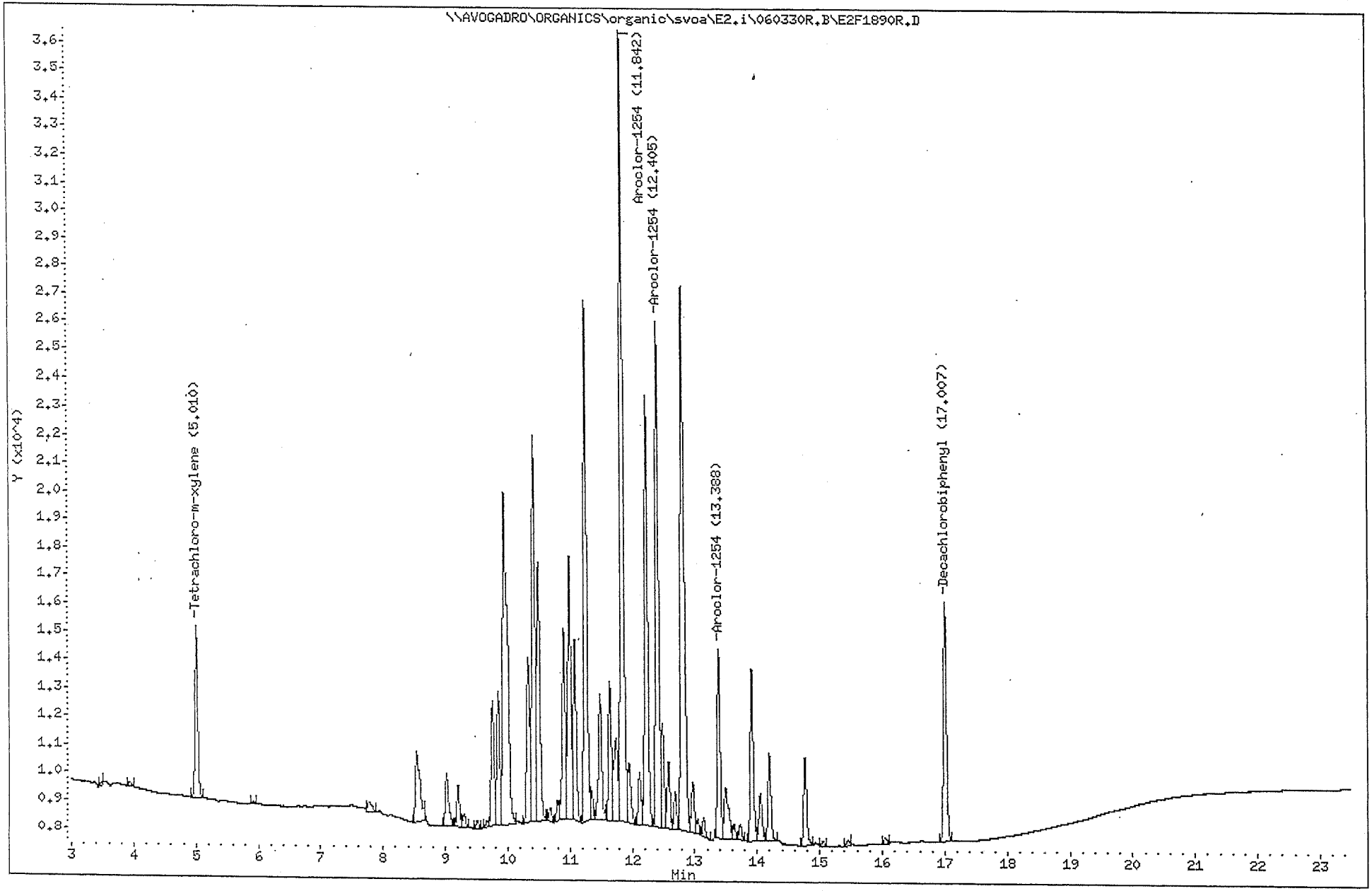
Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1890R.D
Date : 31-MAR-2006 00:44
Client ID: AR1254FL2
Sample Info: AR1254FL2,AR1254FL2,,ar1254.sub,,
Volume Injected (uL): 1.0
Column phase: RtxCLPPest 2

Instrument: E2.i

Operator: SZ SRC: SZ
Column diameter: 0.53



Data File: E2F1890F.D
Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080 PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2F1890F.D
Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
Inj Date : 31-MAR-2006 00:44
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
Misc Info : 1,2,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330F.B\E2_PCB_F.m
Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898F.D
Als bottle: 14 Calibration Sample, Level: 2
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1254.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8		
6.41	6.41	0.000	6923	0.01000	0.010		(a)
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3		
19.6	19.6	0.000	9157	0.01000	0.011		(a)
7 Aroclor-1254					CAS #: 11097-69-1		
12.6	12.6	0.000	11493	0.50000	0.54	80.00- 120.00	100.00(a)
13.5	13.5	0.000	21409	0.50000	0.54	171.37- 211.37	186.28
14.3	14.3	0.000	12764	0.50000	0.54	92.12- 132.12	111.06
Average of Peak Amounts =					0.54		

3/31/06

Data File: E2F1890F.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: E2F1890R.D
Report Date: 31-Mar-2006 07:54

Mitkem Corporation

8080/8081PCB Quantitation Report

Data file : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2F1890R.D
Lab Smp Id: AR1254FL2 Client Smp ID: AR1254FL2
Inj Date : 31-MAR-2006 00:44
Operator : SZ SRC: SZ Inst ID: E2.i
Smp Info : AR1254FL2,AR1254FL2,,ar1254.sub,,
Misc Info : 1,2,,1
Comment :
Method : \\AVOGADRO\ORGANICS\organic\svoa\E2.i\060330R.B\E2_PCB_R.m
Meth Date : 31-Mar-2006 07:54 jgreene Quant Type: ESTD
Cal Date : 31-MAR-2006 04:18 Cal File: E2F1898R.D
Als bottle: 14 Calibration Sample, Level: 2
Dil Factor: 1.00000
Integrator: Falcon Compound Sublist: ar1254.sub
Target Version: 4.03 Sample Matrix: WATER
Processing Host: TARGET1

Concentration Formula: Amt * DF * Uf * Vt/(Vo * Vi)

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	Correction factor
Vt	10000.000	Volume of final extract (uL)
Vo	1000.000	Volume of sample extracted (ml)
Vi	1.000	Volume injected (uL)

RT	EXP RT	DLT RT	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)	TARGET RANGE	RATIO	
$ 1 Tetrachloro-m-xylene					CAS #: 877-09-8			
5.01	5.01	0.000	6063	0.01000	0.011			(a)
$ 10 Decachlorobiphenyl					CAS #: 2051-24-3			
17.0	17.0	0.000	8543	0.01000	0.010			(a)
7 Aroclor-1254					CAS #: 11097-69-1			
11.8	11.8	0.000	28134	0.50000	0.50	80.00- 120.00	100.00	(a)
12.4	12.4	0.000	17965	0.50000	0.49	47.65- 87.65	63.86	
13.4	13.4	0.000	6744	0.50000	0.50	5.17- 45.17	23.97	
Average of Peak Amounts =					0.5			

3/31/06

Data File: E2F1890R.D
Report Date: 31-Mar-2006 07:54

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Attachment 1

Corrective Action Examples and Documentation Tables

Attachment 1

Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria (RSD% >20% or r^2 < 0.990).	1. Check GC conditions such as temperature program, makeup gas flow rate, and carrier gas flow rate. Check if column bleeding occurs and more injection maintenance is needed. After the reasons are found, appropriate maintenance will be done and a new initial calibration will be run. If the new curve still fails, the reasons for failing the curve will be re-evaluated and another a new curve will be rerun after the reevaluation. If still not good, call GC manufacturer.	1. Note in instrument run logbook, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification (ICV) check does not meet QC criteria (D% > ±20%).	2. Check the preparation of ICV standard, and if necessary, make a fresh ICV standard. If it is found that ICV standard is ok, the standards for initial calibration will be checked such as evaporation and the preparation of standards. A new ICV and a new curve will be analyzed after problems are corrected.	2. Note in instrument run logbook, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria (D% > ±20%).	3. Maintenance such as replacing septum and liner for injection port will be done, and then a new set of CCVs and the samples related to the bad CCVs will be re-analyzed. If the CCVs still fail, A full maintenance including replacing septum, liner and gold seal and trimming the column will be done and a new initial calibration will be run. Then the CCV and/or QC and samples re-analyzed.	3. Note in instrument run logbook. If instrument maintenance performed, note in maintenance logbook.
4. Method blank contains target compound above reporting limit.	4. A new aliquot of the blank from prep lab will be reanalyzed to check if contaminants are carried over from previous dirty samples and/or from standards or a bad injection occurs. If the results from re-analysis of the	4. If the re-extraction/re-analysis meets the criteria, the results are reported. Document in the run log. If there is insufficient sample volume for re-extraction/re-analysis, report the initial results. Document in the run log,

	blank are still the same, whole batch of the samples that are related to the blank will be re-extracted and re-analyzed.	document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.
5. Surrogates in the method blank are outside of acceptable range.	5. A new aliquot of the blank from prep lab will be reanalyzed. If the results from re-analysis are the same, the samples that are related to this blank will be re-extracted and re-analyzed.	5. Same as documentation #5.
6. Compound out of acceptance range in laboratory control sample.	6. Investigate source of problem. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP/project requirements, this LCS will be reanalyzed. If the results of reanalysis are the same, the samples that are related to the LCS will be re-extracted and re-analyzed.	6. Flag all compounds out of range on Form 3 of data report, if samples are re-analyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook, comment in data review checklist to be included in project narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer. If re-analysis cannot be performed due to insufficient sample, comment in data review checklist to be included in project narrative, and document as above.
7. Surrogates in samples are out of control limits.	7. 7.1 No actions are needed for the following situations: • One of the surrogates (TCX and DCB) is in control limits, and the other is above the upper limit due to coelution with contaminants. • TCX is in the control limits, and DCB is below the lower limit. This is possibly due to matrix effect. 7.2 Further actions are needed for the following cases: • Both of the surrogates (TCX and DCB) do not meet criteria. • TCX is lower than the lower limits and DCB is	7. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, comment on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. Document in the Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer.

	in the limits. This is most possibly due to evaporation of TCX in the extracts. Actions for 7.2: The samples will be reanalyzed. If the results of re-analysis are the same, the samples will be re-extracted and re-analyzed.	
8. Compound in sample exceeds upper calibration standard concentration.	8. Re-analyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running an instrument using auto-sampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be re-analyzed if sufficient volume exists.	8. Note in instrument run log, comment on data review checklist to be included in project narrative.
9. Instrument blank (GC) contains contamination above QC criteria.	9. Investigate source of problem, decontaminate purge and trap instrument, re-analyze blank and all effected samples.	9. If only re-analysis is reported, note in instrument run log. If both sets of data are to be reported, note in instrument run log, re-extraction request form, preparation logbooks, comment on data review checklist to be included in project narrative.

10. Matrix spike recovery out of QC range.	10.Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in un-spiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, re-analyze spike sample.	10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
11. Duplicate (or MSD) relative percent difference exceeds QC limit.	11. Evaluate problem. If concentration of analyte is close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, re-analyze duplicate sample.	11. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.
12. Retention Time shift.	12. A. No further action is required if the retention times of the CCV and samples shift after regular maintenance such as replacing the septum, liner, gold seal and trimming of the columns. The retention times will be established. B. If the retention times of the CCV shift during analysis, GC maintenance will be performed and the CCV and associated samples and QC will be re-analyzed. C. If the retention times of the CCV do not shift, but the retention times of surrogates in some of the samples do shift, the samples will be re-analyzed. If the retention times still do not meet the criteria, matrix affect is assumed and the results will be reported. This situation will be documented.	12. Document in the run log. If CCV, QC, and samples were re-analyzed, document in the run log. If the retention times shift with respect to re-analysis, document in the run log, in the checklist for the case narrative, and document in the Corrective Action Logbook with a CAR number, have supervisor initial/date.

Attachment 2
Department of Defense QC Requirements

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	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 ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
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 72-hour study.	NA	NA	
Breakdown check (Endrin/ DDT Method 8081 only)	Daily prior to analysis of samples	Degradation ≤ 15% for both Endrin and DDT.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15%.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below (except for Method 8082, which may only use Option 1 or 2): Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	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 and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria	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.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	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 (in Method 8081A exclude toxaphene and technical chlordane, in Method 8015B exclude GRO, DRO, and residual range organics (RRO)).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

TABLE D-15. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 SOLID MATRIX[26]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
4,4'-DDD	81	18	30	135	10	155
4,4'-DDE	97	10	70	125	60	135
4,4'-DDT	92	16	45	140	30	155
Aldrin	93	16	45	140	30	155
alpha-BHC	93	10	60	125	50	135
alpha-Chlordane	92	10	65	120	55	130
beta-BHC	95	11	60	125	50	135
delta-BHC	94	12	55	130	45	145
Dieldrin	96	10	65	125	55	135
Endosulfan I	74	20	15	135	10	155
Endosulfan II	89	17	35	140	20	160
Endosulfan sulfate	99	12	60	135	50	145
Endrin	97	12	60	135	50	145
Endrin aldehyde	92	18	35	145	20	165
Endrin ketone	100	11	65	135	55	145
gamma-BHC	91	11	60	125	50	135
gamma-Chlordane	96	10	65	125	55	135
Heptachlor	96	15	50	140	35	155
Heptachlor epoxide	98	11	65	130	55	140
Methoxychlor	100	14	55	145	45	155

TABLE D-16. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 WATER MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	85	20	25	145
Aroclor 1260	87	19	30	145

TABLE D-17. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 SOLID MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	90	16	40	140
Aroclor 1260	96	12	60	130

[26] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

[27] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

For

Total Petroleum Hydrocarbons by GC-FID Using EPA SW-846 Method 8015B/State Methods

SOP No. 60.0050
Rev 10

	Signature	Date
QA Director:		2/16/07
Lab Director:		2/21/07

MITKEM CORPORATION

STANDARD OPERATING PROCEDURE

for

Extractable Total Petroleum Hydrocarbons by GC-FID Using EPA SW-846 Methods 8015B and State Methods

Rev 10

1. Scope and Application

1.1. This method describes the procedure for analysis of extractable total petroleum hydrocarbons (ETPH) in soil, sediment and aqueous samples (i.e. surface and ground water). The conditions are designed to measure the C_9 to C_{36} range of hydrocarbons. This range represents the major components of a number of widely used petroleum products such as kerosene, jet and diesel fuels, No. 2 to No. 6 fuel oils and motor oil. This method is not used for quantitation of gasoline contamination because the major components of gasoline are not retained in the sample extraction and concentration procedure. This method is not intended for the analysis of potable water.

1.2. Some state or government agencies require various options included in this method. Where appropriate, these options are noted in separate boxes. This SOP describes Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM) protocols for analysis of Army, Navy and Air Force projects. Various state methods, such as Connecticut ETPH and Maine DRO are also included within the scope of this SOP.

2. Personnel Qualifications and Responsibilities

2.1. Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with this SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that this SOP is accurate and up-to-date, and that it is implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure. The Data Reviewer evaluates all laboratory reports for reasonableness of the results and signs the reports. The **QA Director** monitors all aspects of the quality control program to insure the quality of the data generated passes an assessment of data accuracy and precision.

3. Summary of Procedure

3.1. A sample submitted for ETPH/DRO/TPH analysis is extracted with methylene chloride and dried over sodium sulfate. The extract is then concentrated in a Kuderna-Danish apparatus to a final volume of 1.0 ml.

3.2. The 1.0 ml extract is then analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram is collectively integrated within the C_9 through C_{36} aliphatic hydrocarbon range. Average response factors determined using an aliphatic hydrocarbon standard mixture are used to calculate the collective concentrations of petroleum products eluting in the C_9 through C_{36} aliphatic hydrocarbon range.

3.3. This method is suitable for the analysis of waters, soils, wipes, and sediments. It can also be used as an identification procedure for oil and product samples when analyzed diluted in solvent.

4. Sample Preservation, Containers, Handling, and Storage

4.1. Aqueous samples are collected in 1-liter amber glass bottles with Teflon-lined screw caps.

4.2. Soil and sediment samples are collected in 4 oz. (120 ml) amber wide-mouth glass jars with Teflon-lined screw caps.

4.3. Aqueous samples may be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5 ml of 1:1 HCl to a liter sample. The use of alternative acids is permissible. Following collection and addition of acid, the sample must be cooled to 4°C.

> Note: There are other methods for the analysis of petroleum hydrocarbons that allow for longer holding times if the sample is acid preserved. EPA Method 1664 (Oil & Grease and Total Petroleum Hydrocarbons) allows for a 28 day holding time if the sample is preserved to a pH <2. Massachusetts EPH method allows for a 14 day holding time if the sample is acid preserved. The 7-day holding time is a Maine DRO, Connecticut ETPH and generic SW-846 aqueous extractable holding time that applies to a wide range of methods, most of which are not amenable to acid preservation. Petroleum-specific methods allow for longer holding times as long as pH adjustment and refrigeration prevent the microbiological degradation of hydrocarbons. There is likely no loss of hydrocarbons in acid-preserved, refrigerated samples between 7 and 28 days from collection. Aside from any technical or chemical justification, there may be important regulatory significance to the 7 day holding time.

4.4. Soil and sediment samples must be cooled to 4°C immediately after collection.

4.5. A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

4.6. Aqueous samples must be extracted within 7 days of collection, except where noted above; and extracts must be analyzed within 40 days of extraction.

4.7. Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

5. Interferences and Potential Problems

5.1. Washing all glassware with hot soapy water, then rinsing with warm tap water, acetone, and methylene chloride reduces method interferences.

5.2. High purity reagents must be used to minimize interference problems.

5.3. Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it will be followed by the analysis of a system solvent blank to check for cross-contamination.

5.4. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled. A silica gel SPE cleanup procedure may be used to overcome many of these interferences, but some samples may require additional cleanup approaches that are beyond the scope of this SOP.

5.5. Certain organic compounds not associated with the release of petroleum products, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantified as Total Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.

5.6. This procedure will quantify those semivolatile portions of petroleum hydrocarbons amenable to solvent extraction/concentration/GC analysis. Portions of petroleum products beyond the semivolatile range (heavy tars) are not amenable to GC analysis, and will not be detected by this method. Volatile hydrocarbon products such as gasoline will have significant portions of the product lost during the extract concentration phase of the analysis, resulting in false-low results.

6. Equipment and Apparatus

6.1. Gas Chromatograph

6.1.1. Gas Chromatograph: Dual column HP6890 GC with programmable temperature. Chemstation and Enviroquant software are used to store and process data.

6.1.2. Capillary column: 30m x 0.32mm id x 0.25 µm film thickness DB-5MS. Must allow for adequate resolution of alkanes C_9-C_{36} in less than one hour. Must also be capable of resolving pristane from C17 and phytane from C18 in commercial diesel fuel per the State of Maine DRO method.

6.1.3. Flame Ionization Detector (FID).

6.1.4. Auto-sampler: HP 6890 Series Injector, 1ul injection volume.

6.1.5. Microsyringes: 10-µl, 100-µl, 250-µl, 500-µl, 1000-µl.

7. Standards

7.1. Primary Standards

7.1.1. The standards used in this method are discussed below. Please note that standards from other vendors may be used as long as the standards are of high purity (>96%) and traceable to reference materials. This list includes several common hydrocarbon fuel standards. A subset of these fuels is analyzed with each initial calibration to aid in qualitative identification of petroleum products in samples. Other products may be analyzed from time-to-time depending on the needs of a particular project or client. The laboratory should have or have on order one complete set of unopened ampulated standards.

7.1.2. The list of primary (ampulated) standards obtained from Restek:

- MA EPH Aliphatics Standard at 1,000 ug/mL (Cat. # 31459).
- Connecticut Alkane Standard at 1,000 ug/mL (Cat. # 31614).
- #2 Diesel Fuel Composite Standard at 50,000 ug/mL (Cat. # 31258).
- #6 Fuel Oil Standard at 5,000 ug/mL (Cat. # 31218).
- O-Terphenyl Surrogate at 2,000 ug/mL (Cat. # 31066).
- JP-4 Military Jet Fuel at 5,000 ug/mL (Cat. # 31219).
- Kerosene Fuel Standard 5000 ug/mL (Cat. #31094).
- 1-Chlorooctadecane Standard at 10,000 ug/mL (Cat. # 31098).

7.1.3. The list of primary (ampulated) standards obtained from Ultra Scientific:

- EPH Aliphatic Hydrocarbon Standard at 1,000 ug/mL (Cat. # SMA-310).

- Unleaded Gasoline Standard at 5,000 ug/mL (Cat # RGO-608).
- Fuel Oil #4 Solution at 5,000 ug/mL (Cat # RGO-631).
- Fuel Oil #6 Solution at 50,000ug/mL (Cat # RGO-652).
- 5-α Androstane Standard at 2,000 ug/mL (Cat #IST-500).
- SAE 10W30 Motor Oil Standard at 1,000 ug/mL (Cat #RGO-722).
- Jet Fuel A Standard at 5,000 ug/mL (Cat # RGO-671).

7.1.4. The 1-Chlorooctadecane (COD) Internal Standard is purchased at a neat concentration from Sigma. A 4000ug/mL solution is prepared.

7.1.5. 5-α Androstane Surrogate Standard material is purchased at a neat concentration from Sigma. The O-Terphenyl (OTP) Surrogate Standard material is purchased at a neat concentration from Aldrich. These standards are used to prepare the extraction surrogates at 10,000ug/mL.

7.1.6. Other fuel identification standards (Diesel Fuel, Mineral Oil, Coal Tar, Lubricating Oil, Transmission Oil, etc) are purchased from commercial vendors such as fueling stations and automotive supply stores.

7.1.7. All of the standard information is recorded in the Primary Standard Logbook. All vials containing primary standards must be labeled according to the current version of Mitkem SOP 80.0001, Standard Preparation, Equivalency and Traceability.

7.1.8. The expiration date for ampulated standards shall not exceed the manufacturer's expiration date. If no expiration date is given by the manufacturer, the unopened ampules are considered valid for two years. Once opened, all primary standards have a one year expiration date. All expiration dates must be entered on the vial label. All primary standards are stored according to manufacturer's recommendation.

7.2. Working Standards

Using the primary standards listed above, the working standard volumes may be adjusted to make smaller or larger quantities of standards as long as the final working concentrations are the same. All standards made from a primary standard expire on or before the expiration date of the primary standard.

All of the following working standards are logged into the Semivolatile Working Standard Logbook, and labeled as Sw*yymmddX*

where: SW = Semivolatile working standard
yymmdd = date the standard is prepared, in year/month/day format
X = the order the standard is prepared on that date, in alphabetical order

The standards are stored in amber containers under refrigeration at 4°C or below. All standards are stored in a separate location from the samples and/or extracts to minimize cross contamination.

The expiration dates for the working standards are six months from the date of preparation. All standard expiration dates are superceded by the primary standard's expiration date. All expiration dates must be entered on the vial label.

7.3. Stock Standards

7.3.1. The aliphatic hydrocarbon standard consists of the 14 normal alkanes listed below in **Table 1**, and the 5α-Androstane surrogate. Prepare this stock using the Restek brand standards; the resultant concentration for each analyte (aliphatics and surrogate) will be 200 ug/mL. This will be diluted to concentrations of 100, 50, 25 and 2.5 ug/mL to form the calibration curve.

Table 1: The n-alkane standard contains:

Carbon Number	Compound Name
9	n-Nonane
10	n-Decane
12	n-Dodecane
14	n-Tetradecane
16	n-Hexadecane
18	n-Octadecane
19	n-Nonadecane
20	n-Eicosane
Surrogate	5α-Androstane
22	n-Docosane
24	n-Tetracosane
26	n-Hexacosane
28	n-Octacosane
30	n-Triacontane

36	n-Hexatriacontane

7.3.2. The petroleum reference standard consists of a commercially available diesel fuel standard. Prepare this stock by dissolving 0.01g diesel fuel into methylene chloride in a 10ml volumetric flask.

7.4. Surrogate Standard

7.4.1. The surrogate standard is used to monitor the efficiency of sample extraction, chromatographic, and calibration systems.

7.4.2. The surrogate spiking solution is 5α-Androstane and OTP at a concentration of 40 ug/mL in methanol. Each sample, blank, and matrix spike is fortified with 1.0 ml of the surrogate spiking solution.

7.5. Internal Standard

7.5.1. Every 1.0 ml extract is spiked with 10 ul of the internal standard 1-Chlorooctadecane (COD) solution for a concentration of 40 ug/mL.

7.6. Matrix Spike Standard

7.6.1. The matrix spike solution is prepared from a different source as the calibration standards. The spiking solution consists of commercial diesel standard, and is prepared in acetone at a concentration of 5 mg/mL. The samples selected as the matrix spike and the LCS are fortified with 1.0 ml of the matrix spiking solution.

8. Procedure

8.1. Sample Preparation: The following methods are routinely used to extract samples. The associated Mitkem SOP Nos. are listed for reference.

8.1.1. Aqueous: SW 3520 Continuous Liquid/Liquid Extraction, SOP No. 50.0050.
8.1.2. Aqueous: SW 3510 Separatory Funnel Extraction, SOP No. 50.0051.
8.1.3. Soil: SW 3550 Sonication Extraction, SOP No. 50.0052.
8.1.4. Soil: SW 3540 Soxhlet Extraction, SOP No 50.0053.
8.1.5. Soil: SW 3570 Microscale Solvent Extraction, SOP No. 50.0100.
8.1.6. Soil: SW 3545 PFE Extraction, SOP No. 50.0101.

8.2. Gas Chromatography

8.2.1. Gas Chromatographic Conditions:

- Set oven temperature to 55°C for 2 minutes, then ramp 15°C/min up to 300°C and hold for 5 minutes.
- Sample/auto-sampler injection is 1.0 µl.
- Carrier gas is helium or hydrogen.
- Carrier Gas Flow: 3.0 or 4.0 ml/min.
- Air: 400 ml /min.
- Hydrogen: 40 ml /min.
- Makeup gas flow: 40 ml /min.
- FID temperature: 320°C.
- Injection port temperature: 280°C.
- GC operated in pulsed splitless mode.
- Column Pulsed splitless presssure: 20 psi for 0.50 min and a purge time of 0.55 min.
- Linear velocity: approximately 50 cm/sec.

8.2.2. GC Maintenance:

Maintenance is required if indicated by calibration verifications being outside of the QC limits. Maintenance is commonly indicated if the response of the C36 peak is significantly lower than the response of the C32 peak. As this analysis is able to extract high boiling point, non-volatile hydrocarbons and other compounds which are not able to be chromatographed, these compunds condense in the injection port and on the head of the GC column. The major effect of these compounds is to reduce the response of the later-eluting hydrocarbons, as indicated by the C36 response.

- Capillary columns: Clean and deactivate the glass injection port insert or replace with a cleaned and deactivated insert.
- Break off the first few inches, up to one foot, of the injection port side of the column.
- Remove the column and solvent back flush according to the manufacturer's instructions.
- Bake out the column at 320°C. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.
- Record all maintenance in the associated instrument injection logbook. When more intensive maintenance is required it must be documented in the LIMS instrument maintenance log.

8.3. Retention Time Windows

8.3.1. Before establishing retention time windows, make sure the GC is within optimum operating conditions. Make three injections of the Aliphatic Hydrocarbon standard mixture 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.

8.3.2. Calculate the standard deviation of the three absolute retention times for each individual component in the Aliphatic Hydrocarbon Standard, the extraction surrogate, and the internal standard (where applicable).

8.3.3. Plus or minus three times the standard deviations of the absolute retention times for each standard should be used to define the retention time window. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

8.3.4. In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.

8.3.5. The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. These data must be retained be the laboratory.

8.4. Calibration:

8.4.1. Calibrate the instrument using the internal standard procedure. The suggested internal standard for this method is 1-Chlorooctadecane (COD).

8.4.1.1. Using the stock standard from **section 7.3.1** (concentration 400 ug/mL per component), prepare 5 dilutions at 200, 100, 50, 25 and 2.5 ug/mL. The lowest calibration standard must be <u>at or below</u> the Reporting Limit. The other concentrations must correspond to the expected range of concentrations found in real world samples or should define the working range of the detector.

8.4.1.2. Inject each calibration standard using the same technique that will be applied to the samples (e.g., 1 µl injection). Tabulate the peak height or peak area responses against the concentration of each individual compound and internal standard. Calculate response factors (RF) for each individual compound using *Equation 1* (**See Section 10**).

If the RF value over the working range is constant (≤ 20% RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations.

8.4.1.3. A collective response factor must also be established for the entire C_9 to C_{36} hydrocarbon range. To calculate the collective RF, tabulate the summation of the peak areas of all components (all 14 normal alkanes) against the total mass injected using *Equation 2* (**See Section 10**). The collective RF is the average of the responses for the C_9 to C_{36} individual compounds.

8.4.1.4. When a different hydrocarbon range is being quantified, the response factor will change to reflect that range. For the Maine DRO analysis, the response factor includes only the C_{10} to C_{28} range hydrocarbons.

Note: The area of the extraction surrogates must **not be** included in the area of the Aliphatic Hydrocarbon range.

8.4.1.5. The initial calibration is followed by the same normal alkane standard at midlevel concentration from a second, independent source, to confirm that the calibration is valid (within $\pm$20%). Additionally the initial calibration should be followed by common fuel identification standards such as diesel, #4 fuel, #6 fuel and motor oil to ensure that the reference chromatograms for those standards reflect current operating conditions.

8.4.1.6. At a minimum, the RF must be verified on each working day, after every 20 samples per column, or after 12 hours, whichever is more frequent, by the injection of a mid-level calibration 50 ug/mL standard to verify instrument performance and linearity. If the relative percent difference (RPD) for TPH varies from the predicted response by more than ±20%, as calculated using *Equation 3* (See Section 10), a new calibration curve must be prepared.

The **Maine DRO** method requires the GC to be able to resolve pristane from C_{17} and phytane from C_{18} in a typical diesel fuel chromatogram. A diesel fuel standard at 5 mg/mL should be included after each continuing calibration when Maine DRO samples are analyzed. The Maine DRO method requires that the resolution be 60% at the end of each sequence containing Maine samples. If this resolution cannot be demonstrated, the samples should be rerun. If this is not possible, the data must be qualified with the lack of resolution noted in the report narrative.

8.5. GC Analysis:

8.5.1. Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with blanks and QC samples. The sequence ends when the set of sample extracts has been injected or when qualitative and/or quantitative QC criteria are exceeded.

8.5.2. Extracts are introduced into the gas chromatograph by direct injection.

8.5.3. Inject 1 µl of the sample extract using the solvent flush technique.

8.5.4. Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in **Section 8.3**.

8.5.5. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation on a second GC column or by GC/MS may be necessary.

8.5.6. Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

8.5.7. The ETPH range of interest is determined by the collective integration of all peak elutions between specified range "marker" compounds.

For CT-ETPH and ordinary TPH analysis, the hydrocarbon range of interest is defined as all resolved and unresolved peaks eluting between C_9 and C_{36} inclusive.

For the State of Maine and other Diesel Range Organic analyses, the hydrocarbon range of interest is defined as all resolved and unresolved peaks eluting between C_{10} and C_{28} inclusive.

8.5.8. When quantifying on a peak area basis by internal calibration, collective peak area integration must be <u>from baseline</u> (i.e., must include the unresolved complex mixture "hump" areas). For the integration of individual target analytes, surrogate compounds, and internal standards, a valley-to-valley approach should typically be used.

8.6. Calculations

8.6.1. Internal Standard Calibration:

The concentration of each analyte and hydrocarbon range in a sample may be determined by calculating the amount of analyte or hydrocarbon range injected, from the peak response, based upon the analyte/internal-standard response ratio.

8.6.1.1. Aqueous samples:

8.6.1.1.1. The general equation to determine the concentration of a specific analyte or hydrocarbon range in aqueous samples is provided in *Equation 4* (**See Section 10**).

8.6.1.2. Non-aqueous samples:

8.6.1.2.1. The general equation to determine the concentration of a specific analyte or hydrocarbon range in soil or sediment samples is provided in *Equation 5* (**See Section 10**).

8.6.2. External Calibration Method:

In most circumstances where the internal standard cannot be resolved from the analytes of interest, samples are diluted and re-analyzed. This increases the relative proportion of internal standard to the unknown petroleum product so the internal standard peak can be more easily identified. There are circumstances where the external quantification approach may be used. This typically occurs when analyzing #6 Fuel. The Response Factor (*RF*) for external standard quantification is determined using *Equation 6* (**See Section 10**).

When analyzing samples known to contain #6 fuel, a #6 fuel curve is run at 0.5, 5, 10, 20 and 40 mg/mL. The mean RF of these 5 levels is used to quantify samples. A #6 fuel LCS at Level 3 concentration may also be used for this analysis.

%RSD is calculated using *Equations 2 and 3* (**See Section 10**).

8.7. Data Upload to LIMS

Once the QC and samples have completed running they are quantified by uploading them to the LIMS System. On-column concentrations are obtained in the EnviroQuant software. These on-column concentrations are uploaded and adjusted by preparation, percent moisture and dilution factors within the LIMS to generate a final reportable result. Refer to Mitkem SOP No. 20.0004 Data Entry to LIMS, for further details.

9. Quality Assurance/Quality Control

See **Attachments 2 and 3** for method specific QA requirements and general corrective action procedures.

9.1. The laboratory is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results meet the performance standards for the method. When results of sample spikes indicate a typical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

9.2. A system solvent blank must be run after a sample suspected of being highly contaminated to determine if sample carryover has occurred.

9.3. At a minimum, for each analytical batch (up to 20 samples), a Method Blank (MB) and Laboratory Control Sample (LCS) must be run. A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) should be analyzed as well. If insufficient sample to perform MS/MSD analysis is provided, a duplicate LCS (LCSD) is analyzed. A Calibration Check Standard must be run after every 20 samples. The method blank and spiked samples must be carried though all stages of the sample preparation and measurement process.

9.3.1. A typical sequence would proceed as follows:

(1) Continuing Calibration Verification (CCV)
(2) Laboratory Method Blank
(3) Laboratory Control Sample
(4) Samples
(5) Matrix Spike/Duplicate
(6) Continuing Calibration Verification (CCV)
(7) Diesel standard at 5 mg/mL to demonstrate resolution (for Maine DRO)

9.3.2. Control charts can be viewed in the LIMS which plot both LCS recoveries and surrogate standard recoveries as a function of time. At a minimum, when surrogate recovery from a clean soil sample, blank, or QC sample is out of control check the calculations to locate possible errors; check the fortifying solution for degradation and changes in instrument performance. If the cause cannot be determined, reanalyze the sample. Surrogate recoveries from clean water samples are often relatively low, with the

method blank often having the lowest recovery for the entire batch. The extraction efficiency of the surrogate is lower in DI water than in samples containing minerals. Several methods offer the option of adding salt to improve extraction efficiency for aqueous samples.

9.3.3. In the case when the sample or MS/MSD contains petroleum product and surrogate recovery is outside the control limits, evaluate the data for validity. Resolved or unresolved hydrocarbons eluting in the same range of the surrogate can interfere with the surrogate recovery. Re-extraction or reanalysis may not be required if calibration verifications indicate proper instrument performance, but it is important to contact the Supervisor or Operations Manager to confirm this.

9.4. Minimum Instrument QC

9.4.1. The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest. See **Section 8.4.** regarding Maine DRO resolution requirements.

9.4.1.1. The n-nonane (n-C_9) peak must be adequately resolved from the solvent front of the chromatographic run.

9.4.1.2. The surrogate and internal standards must be adequately resolved from any individual components in the Aliphatic Hydrocarbon standard.

9.4.1.3. All peaks of interest from the Aliphatic Hydrocarbon standard must be adequately resolved to baseline.

9.4.1.4. Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See **Section 8.3**)

9.4.1.5. Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the percent relative standard deviation (%RSD) over the working range of the curve is less than or equal to 20%.

9.4.1.6. The linear range of the aliphatic hydrocarbon standard should be established based upon peak height. The upper limit of the linear range is the peak height of the highest calibration standard. Samples containing peaks greater than the height of the highest calibration standard must be reanalyzed at dilution. Samples where the internal standard cannot be resolved must also be diluted, or quantified using the external standard procedure.

9.5. Initial and Periodic Method QC Demonstrations

The procedures specified in **Section 9.3.1 and 9.3.2** must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to significant changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method or operational problems.

9.5.1. Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate acidified reagent water blanks and four clean sand blanks spiked with diesel fuel or the n-alkane mix at approximately 5 ppm. These are batched in LIMS as IPANDA1 through IPANDA4.

9.5.1.1. Analyze each replicate in accordance with the procedures described in **Section 8.0.**

9.5.1.2. Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte/product, and the precision (as %RSD) of the measurement for each analyte.

9.5.1.2.1. When using the n-alkane mix approach, the mean accuracy of each individual hydrocarbon, excluding C_{36}, expressed as a percentage of the true value, must be between 40% and 140 %. Poorer recoveries may be experienced for the C_{36} standard. Additionally, C9 may have recoveries down to 30% due to losses during sample concentration. For each analyte, the %RSD must be less than or equal to 25%.

9.5.1.2.2. When using the diesel fuel spike mix approach, the mean accuracy of the total diesel product, expressed as a percentage of the true value, must be between 60% and 140 %, and the %RSD must be less than or equal to 25%.

9.5.1.3. If desired, the Accuracy and Precision evaluation may be combined with the MDL evaluation specified in **section 9.3.2**.

9.5.2. Method Detection Limits for Diesel Range Organics

Analyze a minimum of seven replicate reagent water blanks and seven clean sand blanks which have been fortified with diesel fuel at 3 to 5 times

the estimated or calculated Detection Limit, or at the reporting limit. Typical diesel fuel spiking concentrations are 0.1 mg/L for water, and 10 mg/kg for soil. Extract and analyze each replicate according to the procedures in **section 8.0**. Calculate the Method Detection Limit (MDL), Minimum Level (ML) and Reporting Limit (RL) using the procedure described in **section 12.0**. The MDL for TPH analysis does not have any particular significance except as a demonstration of capability. Sample results are never reported below the concentration of the lowest standard in the initial calibration. See Mitkem SOP No. 80.0005 Determination of MDL, for more information.

9.5.2.1. Water MDLs are determined by extracting 7 to 10 replicates of 1-L acidified reagent water blanks spiked with 5α-Androstane/OTP and the petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of approximately 100 ug/mL. Spike a 1-L reagent water blank with 1.0 ml of this spiking solution.

9.5.2.2. Soil MDLs are determined by extracting 7-10 replicates of 10-g sand blanks spiked with 5α-Androstane/OTP and petroleum reference standard. Prepare a petroleum reference standard spiking solution by diluting the petroleum reference stock standard solution with acetone to a concentration of approximately 100 ug/mL. Spike 10g of clean sand blanks with 1.0 ml of this spiking solution.

9.5.2.3. The mean recovery of the summation of all hydrocarbon ranges (including target analytes), expressed as a percentage of the true value of the petroleum reference standard, should be between 40% and 140%.

9.6. On-going Method QC Demonstrations

9.6.1. Each sample, blank, and LCS must be spiked with the surrogate spiking solution. Surrogate recoveries must be within the range determined annually by the QC department (**Section 9.1.5**) or as set in the analytical method. Recoveries outside this range must be noted and discussed on the data report form. Recoveries outside of the acceptance range are common for samples containing interfering peaks, or for clean water samples with low mineral content that exhibit low extraction efficiency. This is especially true for 5α-Androstane in water samples. This surrogate should be evaluated using advisory limits due to its problematic characteristics.

9.6.2. At a minimum, with every batch of 20 samples or less the lab must analyze the following:

9.6.2.1. **Continuing Calibration Verification Standard-** A midlevel calibration standard from the same stock solution used to develop the calibration curve must be analyzed prior to sample analysis to verify the calibration state of the instrument. The CCV will be analyzed at a frequency of every 20 samples or every 12 hours, whichever is sooner. If the relative percent difference (RPD) for the collective TPH response factor or the surrogate varies from the predicted response by more than 20% new calibration curve must be prepared. For Maine DRO analysis, a 5 mg/mL standard must be analyzed at the end of each sequence to demonstrate resolution per **Section 8.2.1.**

CT-ETPH: Requires a 30% D ME DRO: Requires a 20%D MA TPH: Requires a 25%D

9.6.2.2. **Laboratory Method Blank-** A water or soil Laboratory Method Blank is prepared by fortifying a reagent water or clean sand blank with 1.0 ml of the surrogate spiking solution. TPH detected within the retention time window of any analyte or range of interest above the Reporting Limit must be noted on the data report form.

9.6.2.3. **Laboratory Control Sample-** A laboratory control sample is prepared by fortifying a reagent water or clean sand blank with 1.0ml of the surrogate spiking solution and 1.0 ml of the matrix spiking solution.

ME DRO LCS/LCSD recovery limits are set at 60-140, with a 20%RPD. MAEPH LCS/LCSD recovery limits are set at 40-140, with a 25% RPD. CT ETPH LCS/LCSD recovery limits are set at 60-140.

9.6.2.4. **Matrix Spike/Duplicate-** The water or soil MS/MSD is prepared by fortifying an actual water or soil sample with 1.0 ml of surrogate spiking solution and 1.0 ml of matrix spiking solution. The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations. The corrected concentrations of each analyte should be within the same recovery range as the LCS. The %RPD of the duplicates should be less than 50%.

CT ETPH and ME DRO MS/MSD limits are set at 50-150.

9.6.3. If any of the performance standards specified in this section are not met, the problem must be corrected before further samples are analyzed. Any samples run between the last QC samples that meet the criteria and those that are out of control must be rerun. If this is not possible, the issue must be discussed in the report narrative.

10. Data Reduction and Calculations

- *Equation 1*: Individual Compound Response Factor:

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \text{ or no IS } RF = \frac{(A_s)}{(C_s)}$$

where,

A_s = Response for the analyte to be measured.
C_{is} = Concentration of the internal standard, ng/µl.

A_{is} = Response for the internal standard.

C_s = Concentration of the analyte to be measured, ng/µl.

- *Equation 2*: Range Response Factor:

$$Range\ RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)} \text{ or no IS } Range\ RF = \frac{(A_s)}{(C_s)}$$

where,

A_s = Summation of peak areas of component standards.

C_{is} = Concentration of internal standard, ng/µl.

A_{is} = Response for internal standard.

C_s = Total mass concentration of injected standards, ng/µl.

- *Equation 3*: Relative Percent Difference (RPD):

$$RPD = \frac{R_2 - R_1}{(R_1 + R_2)/2} \times 100\%$$

where,

R_1 = Calibration Factor from calibration curve.
R_2 = Calibration Factor from calibration check.

• *Equation 4*: Concentration in Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(V_s)} \text{ or no IS}$$

$$Concentration\ (\mu g / L) = \frac{(A_x)(D)}{(RF)(V_s)}$$

where,

A_x = Response of the analyte, hydrocarbon range/TPH being measured, units may be in area counts or in peak height.

C_{is} = Amount of internal standard added to extract, ng.
D = Dilution factor, if a dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

A_{is} = Response of the internal standard, units same as A_x.

RF = Response factor for analyte or hydrocarbon range/TPH, dimensionless.

V_s = Volume of aqueous sample extracted, ml.

• *Equation 5*: Concentration in Non-Aqueous Samples:

$$Concentration\ (\mu g / Kg) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(W_d)} \text{ or no IS}$$

$$Concentration\ (\mu g / Kg) = \frac{(A_x)(D)}{(RF)(W_d)}$$

where,

W_d = Dry weight of sample extracted, g. (See Equations 7 – 9).

A_x, C_{is}, D, A_{is}, and RF have the same definition as for aqueous samples.

- *Equation 6*: Response Factor (*RF*) for external standard quantification :

$$RF = \frac{A_x}{C_x}$$

where,

A_x = peak area of the Fuel Oil to be measured.
C_x = Concentration of the Fuel Oil to be measured.

- *Equation 7*: % Moisture:

$$\%\ Moisture = \frac{g\ sample - g\ dry\ sample}{g\ sample} \times 100\%$$

- *Equation 8*: % Dry Solids:

$$\%\ Dry\ Solids = 100 - \%\ Moisture$$

- *Equation 9*: Dry Weight:

$$W_d\ (g) = (\%\ Dry\ Solids/100)\,(g\ of\ extracted\ sample)$$

11. Data Validation and Reporting

11.1. Calibration

Using the internal calibration procedure (**Section 8.4.1**) calibrate the GC as follows:

11.1.1. Calculate an RF for the individual normal alkane compounds that comprise the aliphatic hydrocarbon standard.

11.1.2. Calculate an RF for each surrogate.

11.1.3. Calculate a collective RF for the total mass concentration of the C_9-C_{36} Aliphatic Hydrocarbons (or diesel range). Tabulate the summation of the peak areas of all component standards against the total mass injected. Do not include the surrogates.

11.2. Sample Analysis

11.2.1. Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for n-C_9 and 0.1 minutes after the RT for n-C_{36} (or C_{10}-C_{28} for DRO).

11.2.2. Determine the peak area count for the sample surrogate standards and internal standard. Subtract these values from the collective area count value within the appropriate hydrocarbon range.

11.2.3. Using the equations contained in Section 10 or linear regression analysis, calculate the collective concentrations of all resolved and unresolved peaks within the range of interest, and individual concentrations of any sample surrogates and internal standard.

11.3. Reporting Limits:

The Reporting Limits (RL) for individual analytes are set to be equal to the lowest calibration standard that is still within the linear range of the calibration curve, assuming 100% extraction of the sample matrix. The reporting limit for the entire range is based upon the summation of the reporting limit for each individual analyte.

11.4. Data Review:

All data calculations must be reviewed 100% by the analyst or a peer. A final full technical review is done by the Lab Manager or another senior chemist, according to Mitkem's data review and validation procedures.

12. Corrective Action Procedures

All out-of-control situations must immediately be reported to the department supervisor or the QA Director. Out-of-control situations must be recorded in the LIMS Corrective Action Log and documented the situation returned to control as described in the SOP No. 80.0007 Corrective Action Procedures.

13. Health and Safety

The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. All Lab Analysts must refer to the Material Safety Data Sheets (MSDS) prior to handling any unfamiliar reagents.

14. Waste Management, Pollution Prevention, Definitions and Acronyms

Please see Section 20.0 of Mitkem's current Quality Assurance Plan.

15. References

SW-846 Method 8015-Modified for Diesel Range Organics, Revision 2, December 1996.

Maine Health and Environmental Testing Laboratory. Modified ~~GRO~~ DRO Method for Determining Diesel Range Organics, Method ~~4.2.17~~ 4.1.25, September 6th 1995. JOC 9/25/07

Connecticut Extractable Total Petroleum Hydrocarbons , Environmental Research Institute, University of Connecticut, March, 1999.

Department of Defense, Quality Systems Manual for Environmental Laboratories. Final Version 3, January 2006.

Attachments:

1. **Attachment 1:** TPH Standard Chromatograms.
2. **Attachment 2:** Method Specific QA Requirements.
3. **Attachment 3**: Corrective Action and Documentation For Method TPH (GC/FID).

Attachment 1

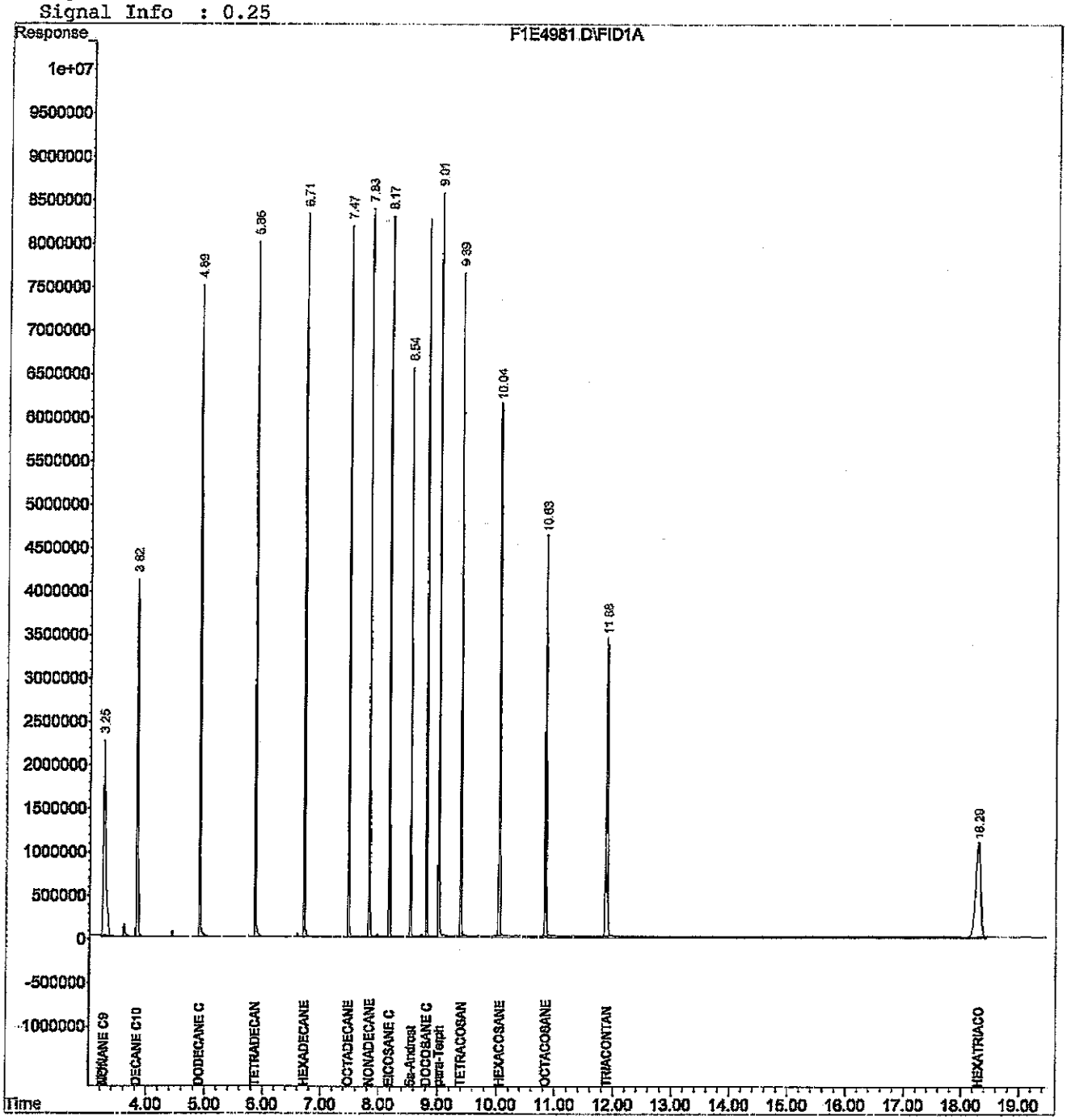
TPH Standard Chromatograms

Quantitation Report

Data File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4981.D Vial: 76
Acq On : 12-29-05 12:21:43 Operator: JG
Sample : ,FSTD0501C,,FSTD0501C,,TPH-F Inst : F1
Misc : Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 29 12:52 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Thu Dec 29 06:03:27 2005
Response via : Multiple Level Calibration
DataAcq Meth : ETPH-YD1.M

Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25



Quantitation Report (QT Reviewed)

Data File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4981.D Vial: 76
Acq On : 12-29-05 12:21:43 Operator: JG
Sample : ,FSTD0501C,,FSTD0501C,,TPH-F Inst : F1
Misc : Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 29 12:52 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Thu Dec 29 06:03:27 2005
Response via : Initial Calibration
DataAcq Meth : ETPH-YD1.M

Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25

Compound	R.T.	Response	Conc Units
Internal Standards			
1) I 5a-Androstane	8.54	52384338	40.000 ug/ml
System Monitoring Compounds			
2) S para-Terphenyl	9.01	69155378	51.518 ug/ml
Spiked Amount 50.000		Recovery =	103.04%
Target Compounds			
3) NONANE C9	3.25	59077662	49.794 ug/ml
4) DECANE C10	3.82	59797824	49.412 ug/ml
5) DODECANE C12	4.89	60459305	49.395 ug/ml
6) TETRADECANE C14	5.85	61522705	50.085 ug/ml
7) HEXADECANE C 16	6.71	62784557	50.290 ug/ml
8) OCTADECANE C18	7.47	64315368	50.797 ug/ml
9) NONADECANE C19	7.83	64618192	50.916 ug/ml
10) EICOSANE C20	8.17	64858959	50.810 ug/ml
11) DOCOSANE C22	8.80	65494137	51.287 ug/ml
12) TETRACOSANE C24	9.39	65790216	51.254 ug/ml
13) HEXACOSANE C26	10.04	66304517	51.598 ug/ml
14) OCTACOSANE C28	10.83	66335257	52.411 ug/ml
15) TRIACONTANE C30	11.88	66232763	53.348 ug/ml
16) HEXATRIACONTANE C36	18.29	62958503	58.657 ug/ml
17) H TPH	3.22	996491756	804.766 ug/ml

12/29/05 N

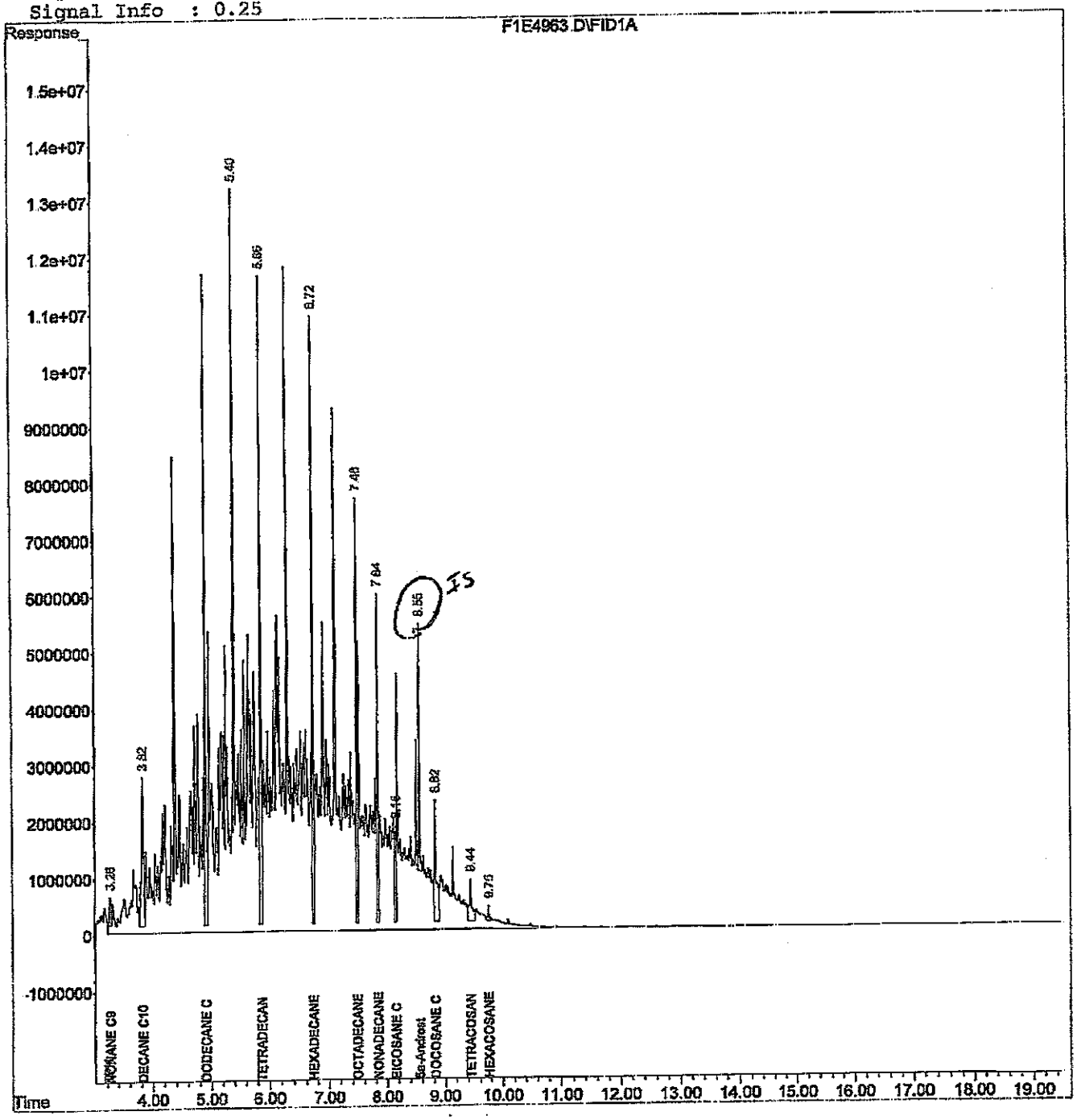
(f)=RT Delta > 1/2 Window (m)=manual int.

Quantitation Report

Data File : O:\ORGANIC\SVOA\F1.I\051228F.B\F1E4963.D Vial: 99
Acq On : 12-28-05 15:19:04 Operator: JG
Sample : ,DIESEL,,DIESEL,,TPH-F Inst : F1
Misc : 100uL~10mlMECL Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 28 15:45 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Wed Dec 28 14:45:10 2005
Response via : Multiple Level Calibration
DataAcq Meth : ETPH-YD1.M

Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25

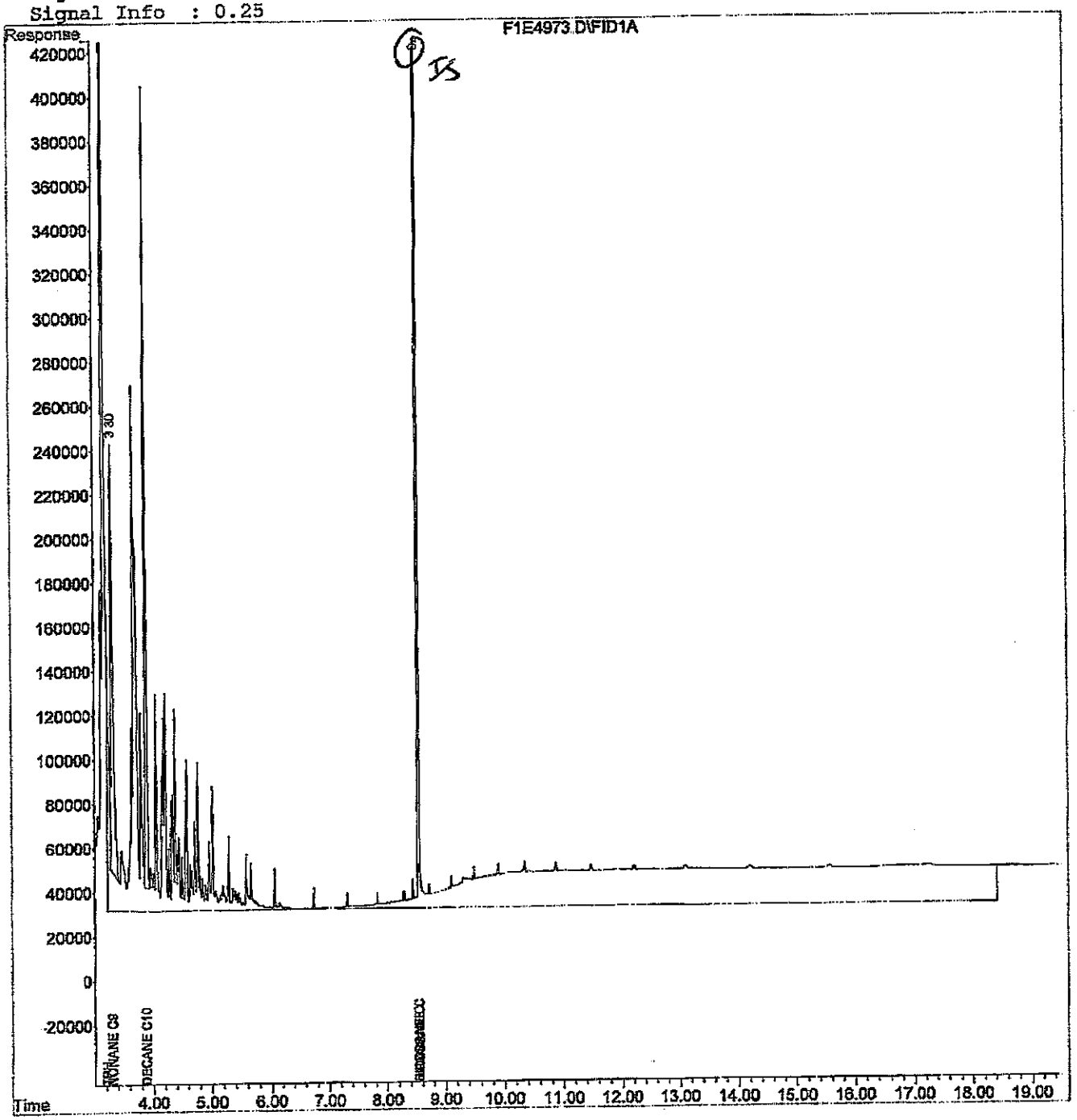


Quantitation Report

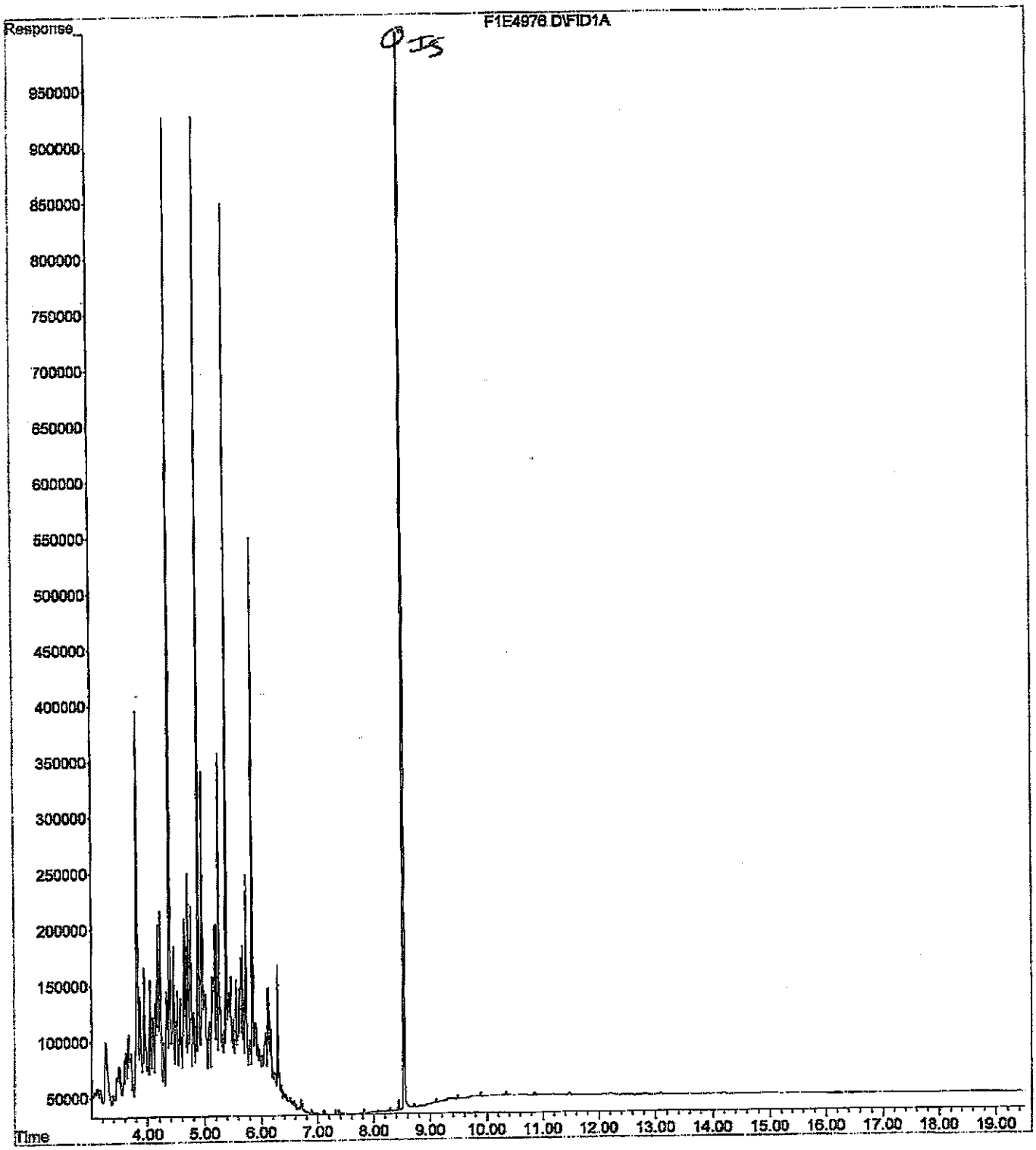
Data File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4973.D Vial: 6
Acq On : 12-29-05 8:30:40 Operator: JG
Sample : ,GASOLINE,,GASOLINE,,TPH-F Inst : F1
Misc : 200ug/mL Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 29 9:02 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Thu Dec 29 06:03:27 2005
Response via : Multiple Level Calibration
DataAcq Meth : ETPH-YD1.M

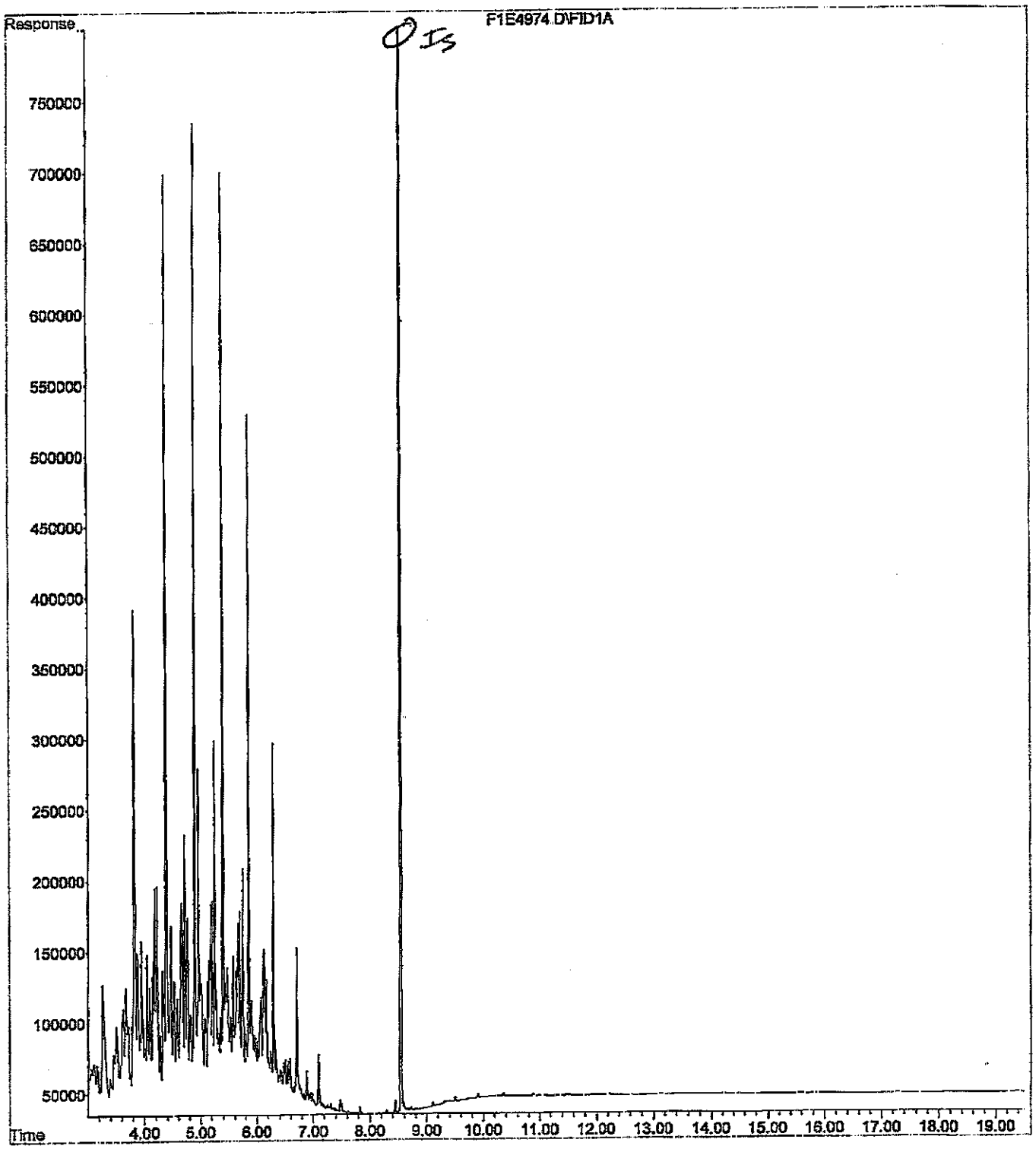
Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25



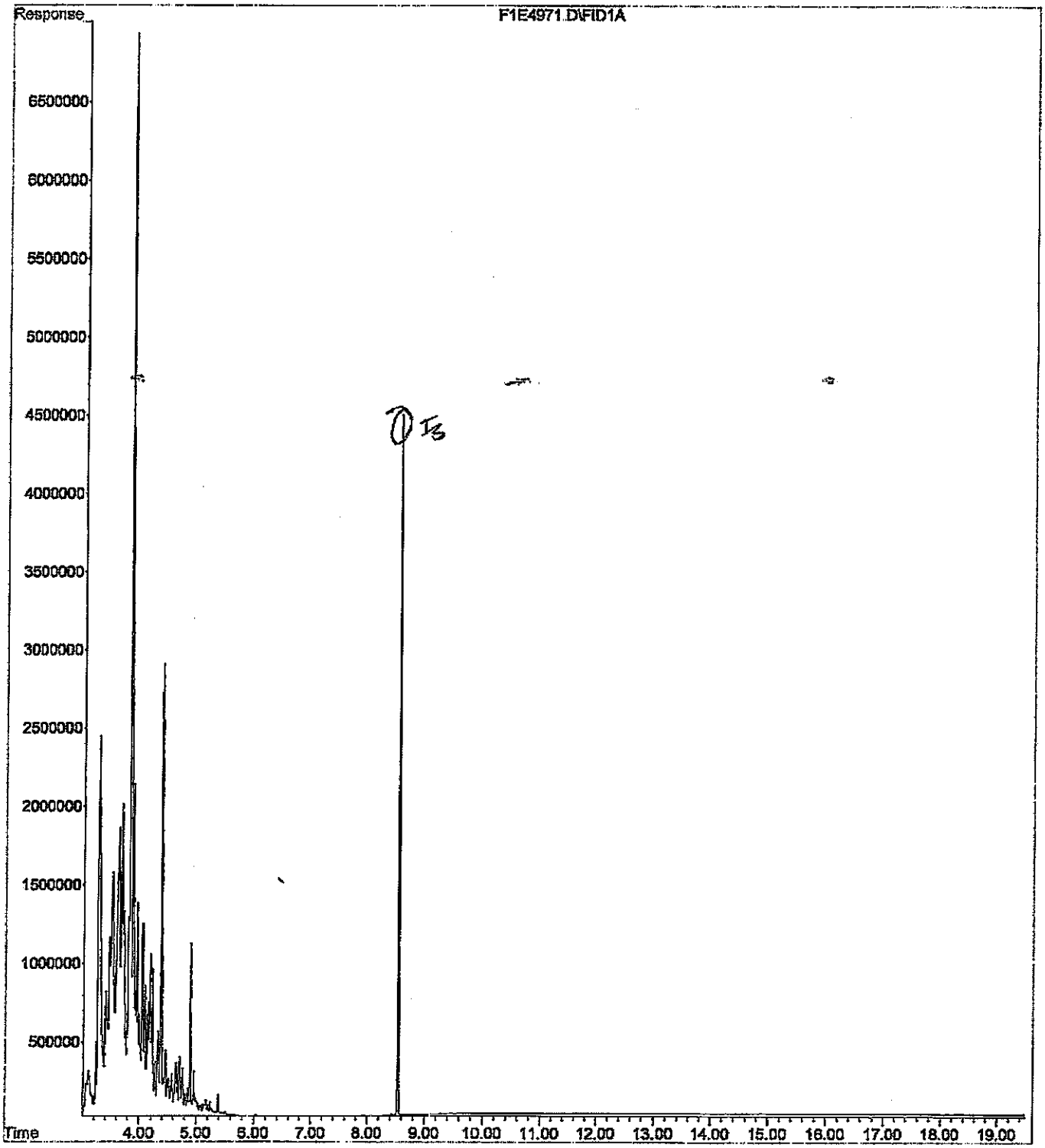
File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4976.D
Operator : JG
Acquired : 12-29-05 9:58:10 PM using AcqMethod ETPH-YD1.M
Instrument : F1
Sample Name: ,JP5-FUEL,,JP5-FUEL,,TPH-F
Misc Info : 200ug/mL
Vial Number: 8



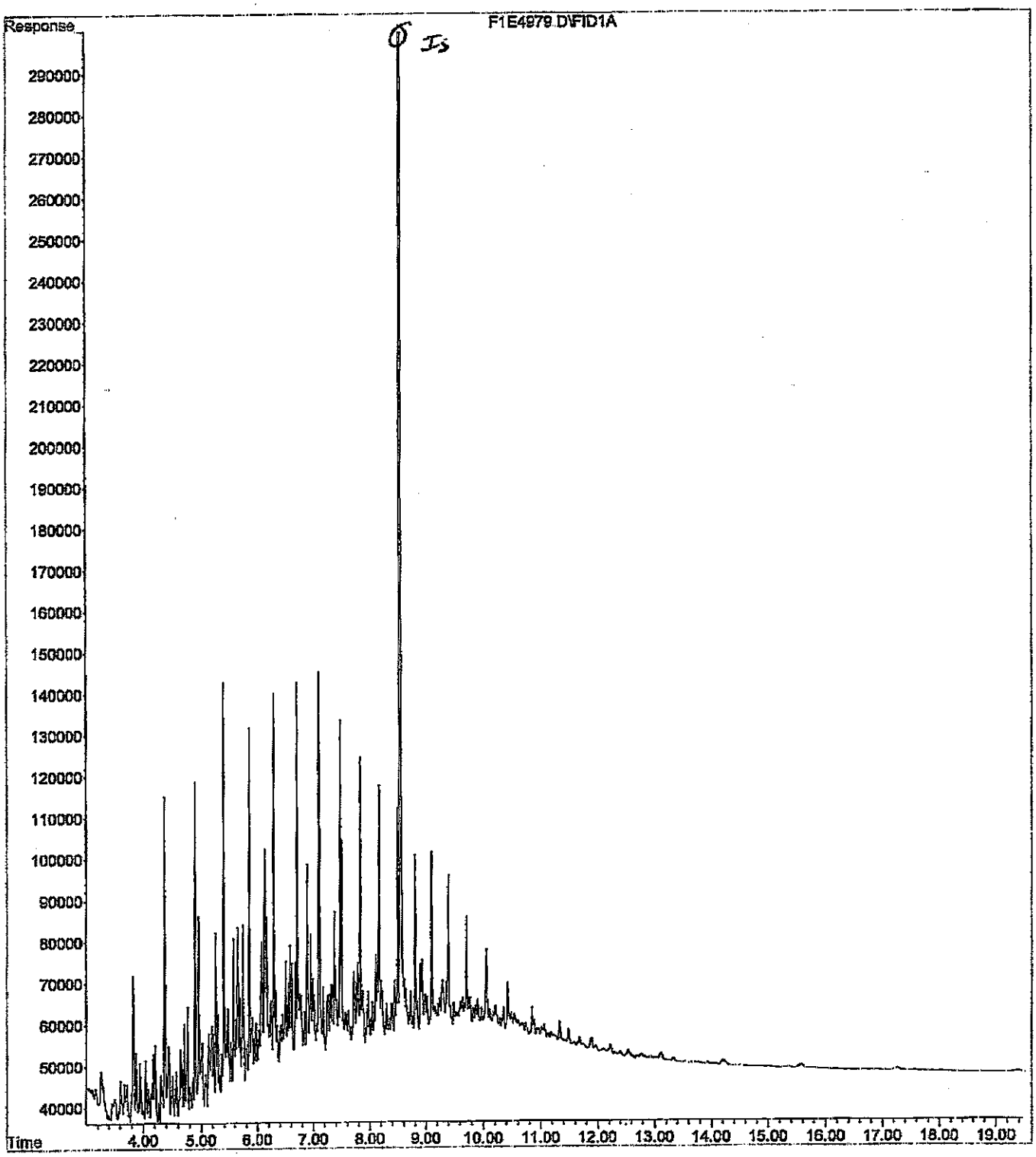
File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4974.D
Operator : JG
Acquired : 12-29-05 8:59:49 PM using AcqMethod ETPH-YD1.M
Instrument : F1
Sample Name: ,JET FUEL A,,JET FUEL A,,TPH-F
Misc Info : 200ug/mL
Vial Number: 5



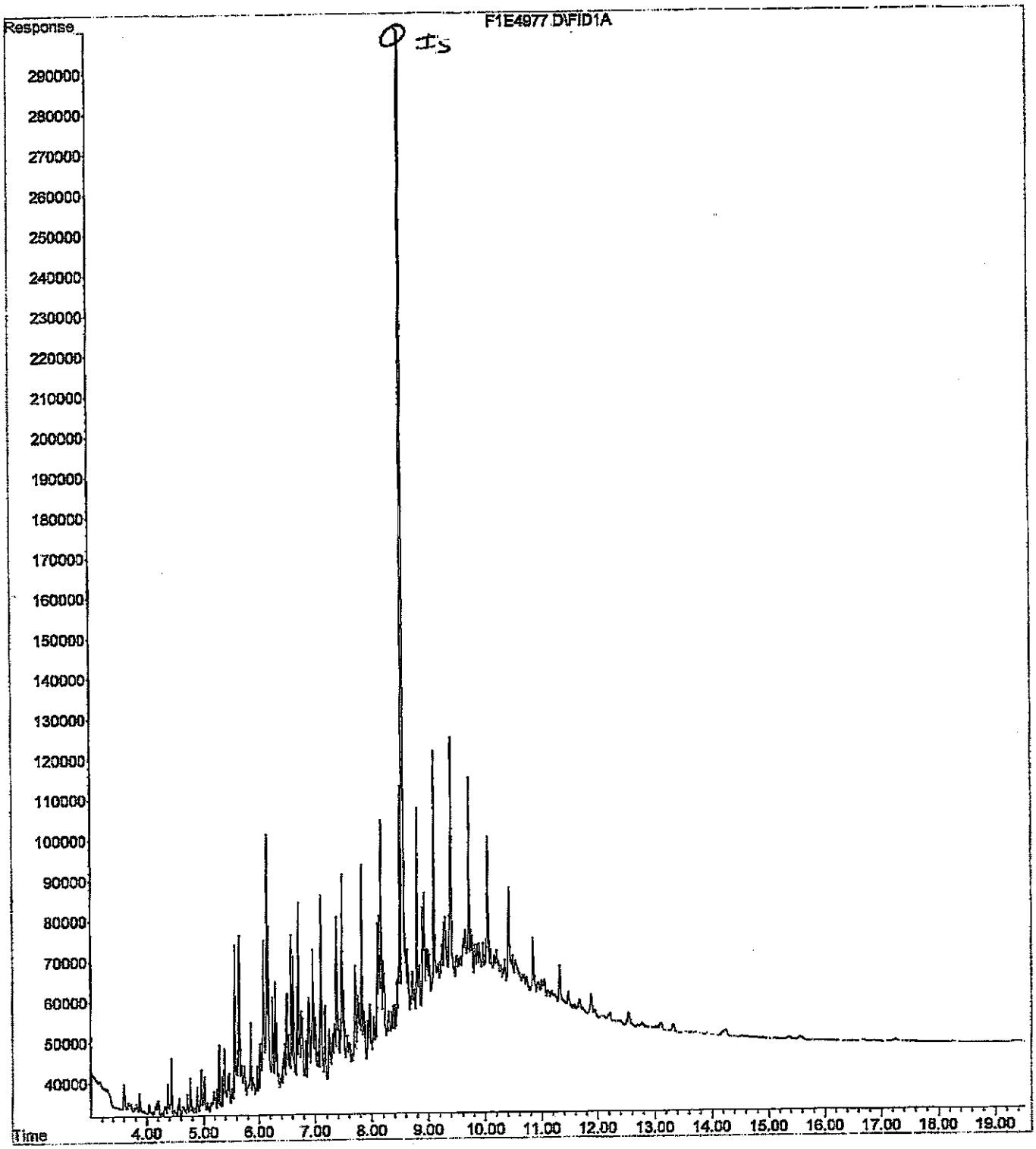
File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4971.D
Operator : JG
Acquired : 12-29-05 7:32:40 PM using AcqMethod ETPH-YD1.M
Instrument : F1
Sample Name: ,MINERAL,,MINERAL,,TPH-F Mineral Spirits
Misc Info : 100uL-10mLMECL,10XDL
Vial Number: 4



File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4979.D
Operator : JG
Acquired : 12-29-05 11:24:03 PM using AcqMethod ETPH-YD1.M
Instrument : F1
Sample Name: ,FUEL OIL #4,,FUEL OIL #4,,TPH-F
Misc Info : 200ug/mL
Vial Number: 11



File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4977.D
Operator : JG
Acquired : 12-29-05 10:26:53 PM using AcqMethod ETPH-YD1.M
Instrument : F1
Sample Name: ,FUEL OIL #6,,FUEL OIL #6,,TPH-F
Misc Info : 250ug/mL
Vial Number: 9

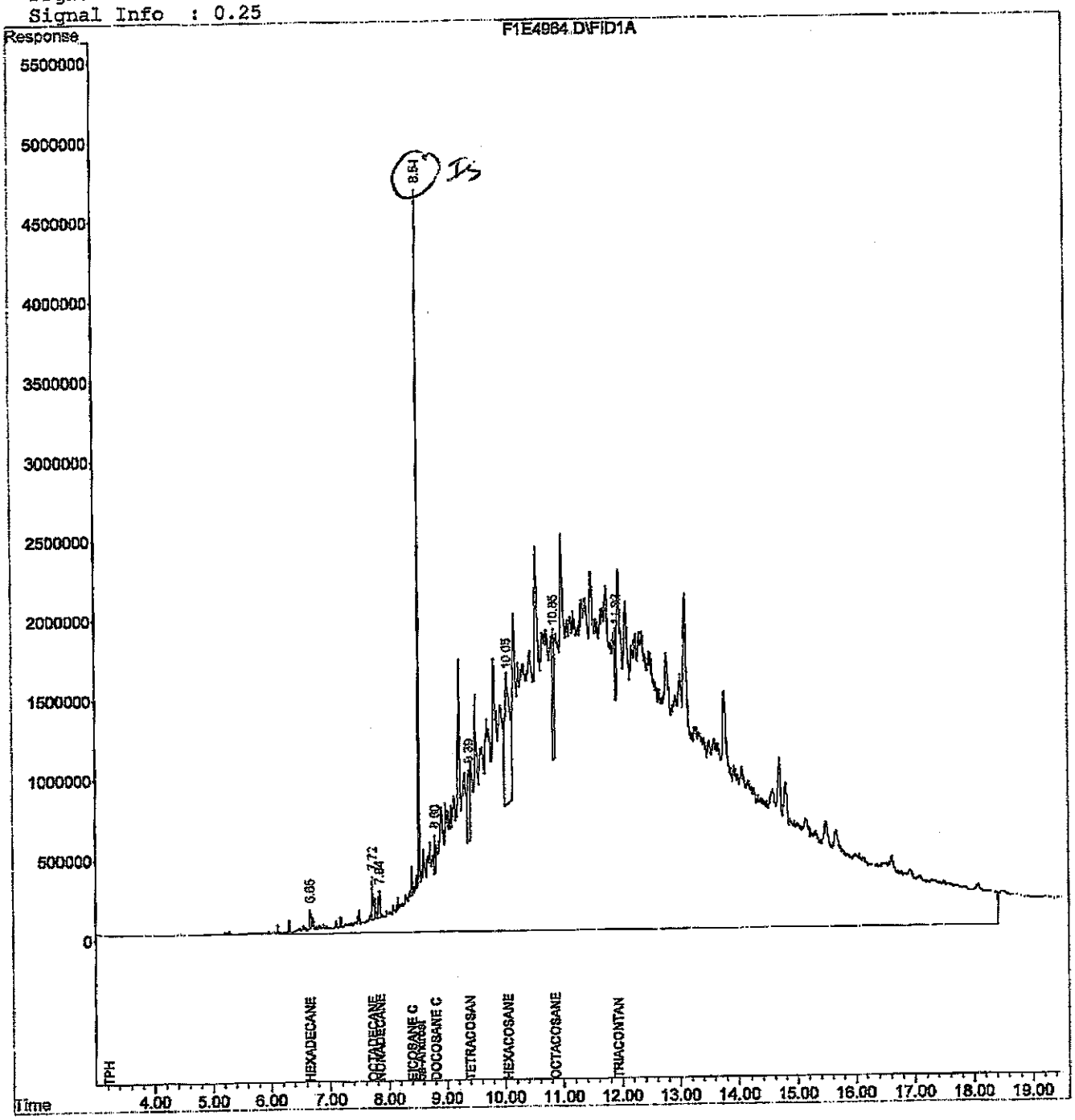


Quantitation Report

Data File : O:\ORGANIC\SVOA\F1.I\051228F.B\F1E4964.D Vial: 98
Acq On : 12-28-05 15:48:14 Operator: JG
Sample : ,MOTOR OIL,,MOTOR OIL,,TPH-F Inst : F1
Misc : 100uL~10mLMECL Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 28 16:12 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Wed Dec 28 14:45:10 2005
Response via : Multiple Level Calibration
DataAcq Meth : ETPH-YD1.M

Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25

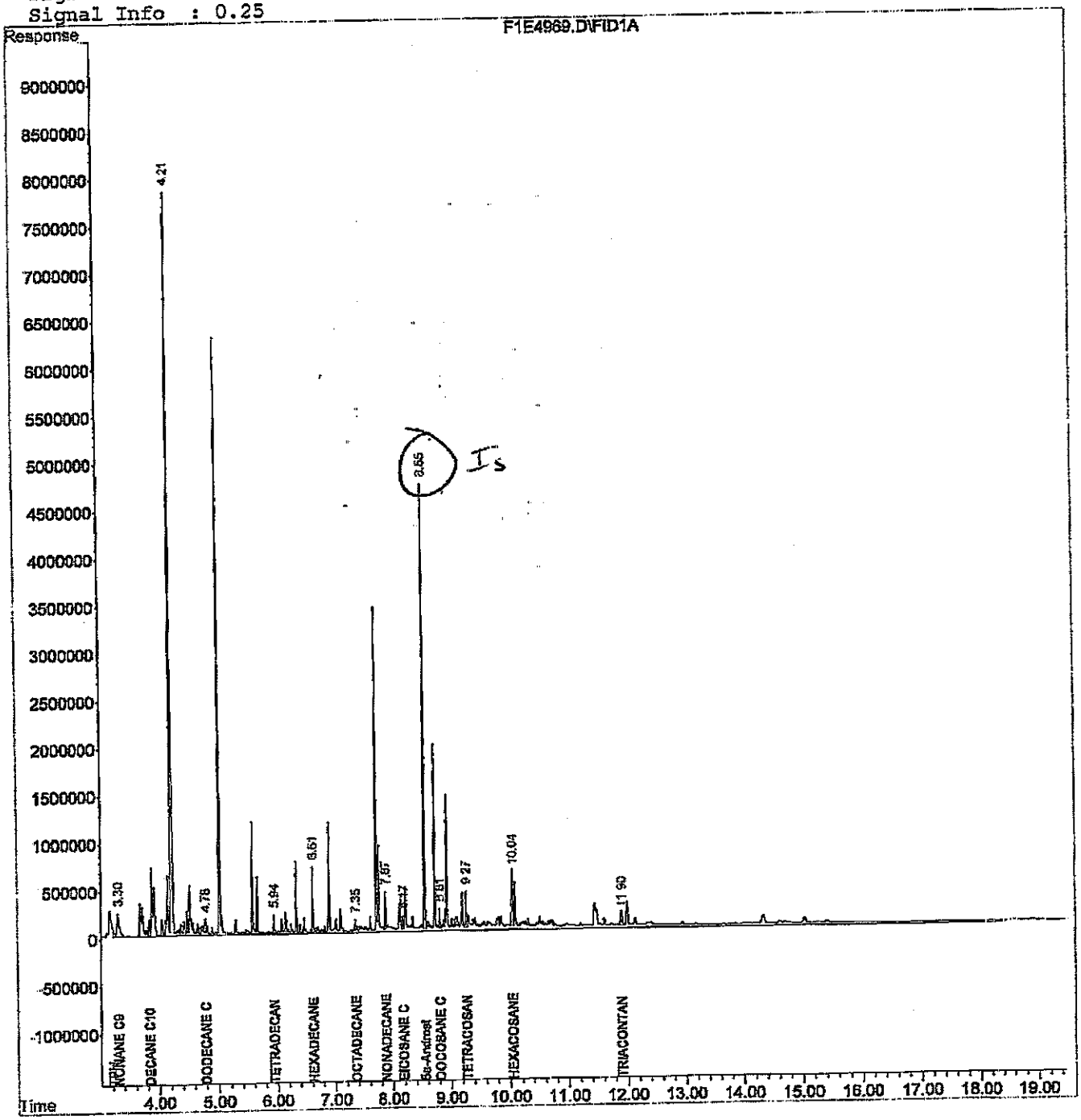


Quantitation Report

Data File : O:\ORGANIC\SVOA\F1.I\051229F.B\F1E4969.D Vial: 1
Acq On : 12-29-05 6:35:07 Operator: JG
Sample : ,COAL TAR,,COAL TAR,,TPH-F Inst : F1
Misc : 100uL~10mLMECL,10XDL Multiplr: 1.00
IntFile : AUTOINT1.E
Quant Time: Dec 29 7:07 2005 Quant Results File: TPH1204F.RES

Quant Method : O:\ORGANIC\S...\TPH1204F.M (Chemstation Integrator)
Title : TPH, ETPH, DRO, Fuel ID,
Last Update : Thu Dec 29 06:03:27 2005
Response via : Multiple Level Calibration
DataAcq Meth : ETPH-YD1.M

Volume Inj. : 1
Signal Phase : DB-5MS
Signal Info : 0.25



Attachment 2

QA Criteria and Method Quality Objectives for Method SW8015M for DoD

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	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 ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
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 72-hour study.	NA	NA	
Breakdown check (Endrin/ DDT Method 8081 only)	Daily prior to analysis of samples	Degradation ≤ 15% for both Endrin and DDT.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15%.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below (except for Method 8082, which may only use Option 1 or 2): Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1232). In addition, a mid-level or lower standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	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 and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	NA	NA	
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria are not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification. For method 8015, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis CCV: After every 10 field samples and at the end of the analysis sequence	All analytes within ± 20% of expected value from the ICAL	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. See section 5.5.10 and box 55. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification.	ICV: Flagging criteria are not appropriate. CCV: Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification, if reanalysis is not possible.	If %D for an individual analyte is > 20%, no samples may be analyzed until the problem has been corrected.
Method blank	One per preparatory batch	No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected ≥ RL.	Correct problem, then see criteria in box D-5; if required, reprep then reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch	
Laboratory control sample (LCS) containing all analytes required to be reported, including surrogates	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix DoD-D)	If corrective action fails apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch	
Matrix spike (MS)	One MS per preparatory batch per matrix (see box D-11)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	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 J-flag 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	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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015, 8021, 8070, 8081, 8082, 8141, 8151, 8310, AND 8330) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria	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.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	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 (in Method 8081A exclude toxaphene and technical chlordane, in Method 8015B exclude GRO, DRO, and residual range organics (RRO)).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J-flag if RPD > 40% or Q-flag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and document in the case narrative.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

Attachment 3

Corrective Action and Documentation for Method TPH (GC-FID)

ITEM #	OCCURRENCE	ACTION	DOCUMENTATION
1	RSD% of target compounds such as 5-Alpha androstane and diesel and other petroleum products for initial calibrations exceeds 20%.	• Full maintenance will be done and a new curve will be run. • If RSD% for this new curve is still not ok, five levels fresh standards will be prepared and run. • If still not good, check chromatograph if column bleeding occurs. If column bleeding does happen, the old column will be replaced with a new one. • If still not good, make a service call.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned. • Any maintenance will go to LIMS maintenance logbook.
2	D% for initial calibration verification (ICV) exceeds 20%.	• Check if ICV standard is right one. • Check the preparation of ICV standard. • Check preparations for five level initial calibration standards if the ICV standard is ok. • Re-analyze if necessary.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
3	D% for continuing calibration verification (CCV) exceeds 20%.	• If D% is out but positive, and target compounds are lower than the reporting limits, no further action is required. • If D% is out and also negative, following further actions are needed: • Maintenance will be done including replacing septum, liner, gold seal, and trimming the column (full maintenance). • Check if CCV standard is evaporated, and if it does, make a fresh CCV standard. • If still not good, a new curve will be analyzed.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned. • Any maintenance will go to

			maintenance logbook.
4	Method blank contains petroleum products that are higher than reporting limits.	• This method blank will be re-analyzed to check if contamination is carried over. • Check if the blank and the other sample are switched. • After the blank contamination is confirmed, the samples that are related to this blank will be re-extracted and re-analyzed.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
5	Diesel or other spiked petroleum products in LCS are out of control limits.	• Check if it is double spiked. If it is, no further action is needed. • No further action is needed if LCS recovery is positive and the samples that are related to this LCS are lower than the reporting limits. • LCS will be re-analyzed, and if the result is still not ok, re-extraction and re-analysis for the samples that are related to this LCS will be done.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
6	Surrogate recovery in method blank or LCS is out of control limits.	• No further action is needed if surrogate recovery is higher than the upper limit and there are no target hydrocarbons detected in the related samples. • The method blank or LCS will be re-analyzed if surrogate recovery is lower than the lower value of control limits. If the results of re-analysis are still the same, the samples that are related to this blank or LCS will be re-extracted and re-analyzed. • The method blank or LCS will be re-analyzed if surrogate recovery is higher than the upper value of the control limits and the samples that are related to this blank or LCS are detected for target hydrocarbon products. If the result of re-analysis is the same, the batch will be re-extracted and reanalyzed.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.

7	Surrogate recoveries in samples are out of control limits.	• No further action is needed if surrogate recovery is higher than the upper limit and there are no target hydrocarbons detected in the samples. • The extracts will be re-analyzed if surrogates are out of limits and target petroleum products are detected in these samples. If the re-analysis results are the same, the affected samples will be re-extracted and re-analyzed.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Log and a CAR# assigned.
8	Matrix spike (MS) recovery is out of QC range.	• If duplicate spike (MSD) shows the same effect, it is generally matrix effect, and no further action is needed except documentation. However, frequent failures will require investigation to check if concentration of spiking standard is correct.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Logbook and a CAR# assigned.
9	Relative Percent Difference (RPD%) for Matrix spike (MS) and Matrix Spike Duplicate (MSD) recoveries is out of QC range.	• This limit is advisory. However, failures will require investigation such as checking if concentration of spiking standard is correct and spiking volume is consistent. Documentation is required.	• This will be noted in the instrument run logbook. • The information will be noted on the package checklist for documentation in the project narrative. • This will be recorded in the LIMS Corrective Action Logbook and a CAR# assigned.

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

Rev. 5

	Signature	Date
QA Director:		12/20/07
Lab Director:		12/20/07

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL, INC.

STANDARD OPERATING PROCEDURE

for

Determination of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by Selective Ion Monitoring (SIM) using Modified Method SW-846 8270

1. Scope and Application

This Standard Operating Procedure (SOP) describes the analysis of Semivolatile Polycyclic Aromatic Hydrocarbons (PAHs) in aqueous and solid sample using gas chromatography/mass spectrometry (GC/MS) and Selective Ion Monitoring (SIM). The SOP follows the analyses as discussed in current USEPA SW-846 Final Update IV , Method 8270D.

The semivolatile Polycyclic Aromatic Hydrocarbons that can be analyzed using this SOP are listed in **Table 1**. Additional SW-846 8270 semivolatile compounds can be analyzed by this method.

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol. **Supervisors** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors** review the logbooks and data generated from this procedure and approve all reported results. The **Project Manager** evaluates laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

3.1 The samples are extracted using appropriate sample extraction methods (see SOPs for sample extraction) and, if necessary, sample clean-up procedures.

3.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer connected to the gas chromatograph.

3.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by coelution of the ions that are selected at the correct retention times. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a minimum of a five-point calibration curve.

3.4 A list of acronyms used in this SOP is included in **Table 2**.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. In some instances, clients will provide their own containers. For semivolatile organic compound analysis by the SIM method, water samples are collected in 1-liter amber glass bottles. Solid samples are collected in 8-ounce amber glass containers. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples.

4.2 Sample extracts are transferred to the semi-volatile organics analysis lab with appropriate sample preparation information. Extracts are stored at 4° ± 2°C, protected from light, in sealed vials (screw-cap or crimp-cap) equipped with un-pierced PTFE-lined septa and stored in a separate location from the analytical standards.

4.3 The holding time of the extracts for semivolatile organic compounds analysis by the SIM method is 40 days from date of sample extraction. The sample preparation holding times are covered in the corresponding extraction procedures SOPs.

5. Interferences and Potential Problems

5.1 Evaluate the raw GC/MS data to verify that interferences were not introduced during the extraction and/or clean up of the samples.

5.2 The SIM method is not appropriate for multi-component analytes (Aroclor, toxaphene, chlordane, etc.). Refer to Methods 8081 and 8082.

5.3 This method is not appropriate for some of the routine SW8270 compounds. Semivolatile PAHs are routinely analyzed by the SIM method.

6. Equipment and Apparatus

6.1 Equipment:

6.1.1 There are five GC/MS in the semivolatile organic analysis lab. They are S1, S2, S3, S4 and S5. S1 and S2 have similar configurations, including Hewlett Packard (HP) Model 5890 gas chromatograph interfaced to HP Model 5972A mass spectrometer (GC/MS). S3 and S4 are composed of HP 6890 GC System (Model: G2614A) and Agilent 5973 MSD. S5 is a Hewlett Packard (HP) Model 7890A gas chromatograph with a front and rear injection port. The front injector is interfaced to HP Model 5975C mass spectrometer (GC/MS). The rear injector is interfaced with a flame ionization detector (FID). HP EnviroQuant Software is used to handle data acquisition. Target software by ThruPut is used for data reduction and report generation.

6.1.1.1 The HP GC is fitted with an electron pressure controller (EPC) to allow constant carrier gas flow during the temperature ramp.

6.1.1.2 A 30m x 0.25mm id (0.5 µm film thickness) DB-5MS fused-silica capillary column (J & W) is used for the analyses. Equivalent columns including HP-5MS have been used with similar performance.

6.1.1.3 A HP Model 6890 autosampler is used for sample injection on S1 and S2. An Agilent Autoinjection Tower 7683 is for S3 and S4. S5 is equipped with a CTC Analytical Leap autosampler capable of dual injections.

6.1.2 Instrument operating conditions are as follows:

<u>General Gas Chromatography Conditions</u>

Carrier Gas	Helium (99.999%)
Column Flow	about 1 mL/minute

Injector Temperature	290°C
Transfer Line Temperature	280°C
Injection Volume	1 µL

GC Temperature program

Ramp	Rate	Initial Temp	Hold Time	Run Time
1		45°C	2.0 min	
2	15°C/min	225°C	0 min	
3	25°C/min	310°C	12.25min	29.65min

General Mass Spectrometry Conditions

Mass Range	SIM scans specific to the masses for the individual compounds and surrogate.
Scan Speed	at least 1 scan per second
Ionization Mode	70 eV positive ion

GC/MS program for DFTPP tune analysis:

DFTPP

GC Temperature program for analyzing DFTPP

Ramp	Rate	Initial Temp	Hold Time	Run Time
1		150°C	1.0 min	
2	13 °C/min	300°C	2.0 min	14.54 min

GC/MS Program for Standards, Blanks, LCS, MS/MSD and sample analysis:

BNA_SIM_W or BNA_SIM_S, PAH_SIM_W or PAH_SIM_S

6.1.3 The MS program method scans for specific SIM masses in different time groups.

Group 1 scan should begin after the solvent delay and continue until the elution of 2-methylnapthalene.

Group 2 scan should start right after Group 1 and continue until after fluorene.

Group 3 scan should begin right after Group 2 and continue until after anthracene.

Group 4 scan should begin right after Group 3 and continue until after chrysene.

Group 5 should begin right after Group 5 and continue until after benzo(g,h,i)perylene.

6.1.4 A primary and secondary ion for each analyte and surrogate will be collected. For some compounds an additional secondary ion will be collected to assist with identification. **Table 3** lists the primary and confirmation ions for target and surrogate analytes.

6.2 Preventative Maintenance - GC/MSs are maintained according to the manufacturer's recommendation. The lab analyst performs preventive maintenance as discussed below.

6.2.1 On a daily basis whenever analyses are to be performed, replace the GC septum and clean the injection liner. Also clip up to 6" of the column.

6.2.2 If needed, the analytical column is replaced; this is usually indicated by the tailing of the polar compounds such as pentachlorophenol/benzidine and/or initial and continuing calibration verifications that repeatedly fail to meet method requirements.

6.2.3 If the system constantly drifts out of DFTPP tune and/or the initial and continuing calibration verifications repeatedly fail to meet method requirements, the ion source will need to be cleaned.

6.2.4 There are two filaments in the mass spectrometer. If both filaments are blown, the method sample will be vented to replace both filaments.

NOTE: After major maintenance such as the scenarios described in **sections 6.2.2 through 6.2.4**, an Initial Calibration (ICAL) is analyzed. Document the date of the ICAL in the resolution field in the LIMS Maintenance Logbook.

6.2.5 The rough-pump oil will be replaced at least once a year. Check the oil level periodically and add oil if needed. Document this maintenance as above.

6.2.6 Once a year, all GC/MS systems may undergo extensive maintenance by a skilled technician. When this occurs, collect all associated paperwork and enter relevant information in the LIMS

maintenance log. The paperwork can be brought to the data reporting area for PDF inclusion on the server.

6.2.7 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the instrument vendor for telephone-consultation and if needed, schedule on-site repair. This information should be documented as in **Section 6.2.6**. In addition, the resolution field in the LIMS Maintenance Logbook should be filled in fully.

6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD hardware manual.

6.4 Glassware:

6.4.1 Hamilton syringes (10 µl, 25 µl, 100 µl, 500 µl, 1000 µl). Syringe accuracy is certified to ±1% by the manufacturer.

6.4.2 Vials – 2ml glass with PTFE-lined screw-cap or crimp-cap tops.

7. Reagents

7.1 Organic solvents – J.T. Baker ultra analyzed grade methylene chloride for standard preparation

7.2 The standards used for this SOP are discussed below. ***Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*** These standards are obtained as ampulated mixture.

7.3 The laboratory will at all times archive or have on order one complete set of unopened ampulated standards (to include internal standards, surrogate standards and target analyte standards).

7.4 All **Primary** standards received from vendors are logged into the SVOA Primary Standard Logbook. The standards are labeled *SPyymmddX*,

where: SP = Semivolatile Primary Standard
yymmdd = date the standard is received
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.4.1 Tune Standard: the tuning standard contains DFTPP, 4,4'-DDT, Pentachlorophenol and benzidine. It is purchased from Restek(Cat. No.31615) at 1000 ug/mL.

7.4.2 Internal Standard: the internal standard is obtained from Cambridge Isotope as neat compounds. A 2000ug/mL solution of all compounds (1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthalene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12) is prepared and transferred into 1mL aliquoted vials. An ampule is opened and then emptied into a mini-inert vial equipped with an on/off valve.

7.4.3 Primary Calibration Standard: the primary calibration standards are obtained from Restek :

- 8270 MEGA Mix (Restek, Cat. No. 31850) at 500-1000ug/mL

7.4.4 Second Source Standard: the second source standards are obtained from NSI:

- TCL BNA LCS Spike 100ug/mL (Cat No. WL-408-25)

7.4.5 Surrogate Standard: Benzo(e)pyrene d-12 standard is obtained from Cambridge (Cat.No.DLM-257-S) at 200 ug/mL.

7.5 All **Intermediate** standards are logged into the SVOA Intermediate Standard Logbook. The standards are labeled *SIyymmddX*,

where:

SI = Semivolatile IntermediateStandard
yymmdd = date the standard is prepared
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.5.1 An intermediate 8270 standard is prepared by making a 10 times dilution of the primary standards in **Section 7.4.3**. An additional intermediate calibration standard at 10ug/mL is prepared by adding the following two standards, and diluting to 4 mL with methylene chloride.

8270 Intermediate (200 ppm)	200 uL
Benzo(e)pyrene-d12 (200 ppm)	200 uL

7.5.1.1 The Initial Calibration standards are prepared as follows:

Levels of	Volume (uL)	Volume (uL)

Initial Calibration (ug/mL)	Intermediate Standard	Methylene Chloride
10	1000	0
5.0	500	500
1 (L3)	100	900
0.5	50	950
0.1	10	990

7.5.2 The ICV is prepared in a similar manner as the Midpoint (L3) of the ICAL. Serial dilutions of the second source standards in **Section 7.4.4** are made until the result is a final 1ug/mL standard.

7.6 All **Working** standards are logged into the SVOA Working Standard Logbook The standards are labeled *SWyymmddX*,

where: SW = Semivolatile Working Standard
yymmdd = date the standard is prepared.
X = the order the standard is logged into the logbook on that date, in increasing alphabetical order.

7.6.1 The working tune standard at 50 ug/mL is prepared by adding 50uL of the stock standard to a final volume of 1 mL with methylene chloride

7.6.2 The internal standard working standard at 500 ug/mL is prepared by adding 250uL of the primary standard (2000 ug/mL) and diluting it to 1 mL with methylene chloride.

Please note that the volumes can be adjusted to make larger or smaller volume of the standards.

The standards are protected from light and stored in the freezer (F7) at -10°C to -20° C. The standards are stored away from sample extracts to minimize cross contamination.

Unopened ampulated standards' expiration dates are based on manufacturer's expiration dates. If no manufacturer's expiration date is provided the ampulated standards may be retained unopened for up to two years. Once an ampulated standard is opened it may be retained for one year from the date it was opened. Intermediate and Working Standard expiration dates are up to 6 months after they are prepared.

NOTE: All standards prepared from a primary standard expire on or before the primary standard's expiration date.

8. Procedure

8.1 The SW-846 methods for sample extraction are as follows:

Method 3510 (SOP# 50.0051) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are serially extracted with methylene chloride using a separatory funnel. The final volume for the extracts is 0.5mL to achieve the lower reporting limit.

Method 3520 (SOP# 50.0050) extracts aqueous samples for water-insoluble and slightly water-soluble PAHs. The samples are placed in a continuous liquid-liquid extractor and extracted with methylene chloride for 18 hours.

Method 3540 (SOP# 50.0053) extracts waste, sludge, sediment and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate, placed in an extraction thimble or between plugs of glass wool, and extracted using 1:1 v/v methylene chloride/acetone in a Soxhlet extractor.

Method 3550 (SOP# 50.0052) extracts waste, sludge, and soil samples for water-insoluble and slightly water-soluble PAHs. The samples are mixed with anhydrous sodium sulfate to form a free-flowing powder, then extracted by ultrasonic extraction using 1:1 v/v methylene chloride/acetone.

8.2 Tuning:

The tune standard is prepared at 50 µg/mL. The GC/MS must be tuned to meet decafluorotriphenylphosphine (DFTPP) criteria every 12 hours when standards, blanks, LCS and/or samples are to be analyzed.

All of the analysis information is to be recorded in the Semivolatile Instrument Logbook. The logbook is issued by the QA officer and will be archived upon its completion.

8.2.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.1.1.4** to perform the tune analysis.

8.2.2 Acceptance criteria for tune - The DFTPP tune will be analyzed using scan for m/z of 35 to 500 amu, based on the full scan. The mass spectrum of DFTPP must be acquired in the following manner for analysis: three scans (the peak apex scan and the scans

immediately preceding and following the apex) are acquired and averaged; if needed, background subtraction will be performed and must be accomplished using no more than 20 scans prior to the elution of DFTPP. It is important that the analyst does not selectively add or subtract scans to generate the tune.

For SW846 projects, tune can also be obtained using one of the following procedures (1) use one scan at the peak apex, (2) scan immediately before or after the apex, (3) use the average across the entire peak

A typical mass spectrum and mass spectral listing of the tune in listed in **Figure 1**.

The acceptance criteria are as follows:

Mass	Ion Abundance
51	10 - 80% of Base Peak
68	< 2.0% of mass 69
70	< 2.0% of mass 69
127	10 - 80% of Base Peak
197	< 2.0% of mass 198
198	Base peak, or > 50% of mass 442
199	5.0 - 9.0% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present, < 24% of mass 442
442	Base Peak , or > 50% of mass 198
443	15 - 24% of mass 442

Once the mass spectrometer passes the DFTPP tune, all subsequent standards, samples, and blanks associated with the tune must be analyzed using identical mass spectrometer instrument conditions.

8.3 Initial Calibration - Initial calibration is performed after the instrument passes the tune, % breakdown requirements and column performance check. Initial calibration is required after major instrument maintenance including source cleaning and/or changing column. Initial calibration will also be performed if continuing calibration analyses do not meet QA/QC criteria.

Five calibration standard solutions are required for all target and surrogate compounds. Standard concentrations of 10, 5.0, 1.0, 0.5, and 0.1ng/µL are required for the surrogates and all compounds.

8.3.1 Calculation for Initial Calibration:

A typical chromatogram of a 2.0ng standard is shown in **Figure 2** attached with the quantitation report.

From the multi-level level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A_x = area of the selected ion for the target compound to be measured
A_{is} = area of the selected ion for the associated internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

The mean relative response factor is determined by averaging the 5 level values.

The % relative standard deviation (%RSD) of the RRF is also calculated using:

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

where: Standard Deviation = $\sqrt{\sum (Xi - X)^2 / (n-1)}$

where: Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values = 5

8.3.2 Initial calibration acceptance criteria for SW-846 is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of –50% to +100% of the mean area response of the internal standard in all of the calibration level.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the

mean retention time over the initial calibration range for each internal standard.

- The RSD for all target analytes and surrogate compound must be < 20%. The Target software will flag any compound whose RSD is greater than 20%. If the RSD of any target analytes and/or surrogate compounds is less than 20%, then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments

(1) The method allows for a maximum of 10% of the target analytes and/or surrogate compound to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier RSDs have a maximum of 50%.

(2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

$$y = mx + b$$

Where y = instrument response (peak area)
m = slope of the line
x = concentration of the calibration standard
b = intercept

It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient **r ≥ 0.995**.

(3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)
a and b = slope of the curve
x = concentration of the calibration standard

c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.3.3 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 1ug/mL standard prepared in **Section 7.5.2.** The acceptance criteria are as follows:

8.3.3.1 For routine SW8270 SIM analyses, the calculated value of the analyte in the ICV must be 70 – 130% of the expected value. DoD **full scan SW8270** analyses have an ICV recovery limit of 75-125%.

8.3.4 Initial calibration acceptance criteria must be met before any sample, blanks or LCS are to be analyzed.

8.3.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action includes preparing fresh standards, source cleaning, changing GC column or injection liners.

8.4 Continuing Calibration - Continuing calibration standards containing all of the target and surrogate compounds at 1 ng on-column injection is performed every time samples are to be analyzed to ensure that the GC/MS system continues to meet instrument sensitivity and linearity requirements.

8.4.1 Frequency of Continuing Calibration - The continuing calibration standard must be performed once every 12 hours. If time remains in the 12-hour time period after meeting the acceptance criteria for the initial calibration, samples may be analyzed. The continuing calibration is also required whenever blanks, LCS, MS/MSD and samples are analyzed.

8.4.2 Continuing calibration acceptance criteria:

- The % D must be ≤ 20% (use % drift if using a regression fit model). A maximum of 20% of the target analytes and/or surrogate compound are allowed to fail the 20% RSD criteria. These allowable outliers should NOT be common compounds or compounds of interest to a specific project that will utilize this initial calibration. In addition, these outlier %Ds have a maximum of 50%.
- No quantitation ion may saturate the detector.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (1ug/mL) of the associated initial calibration.
- The internal standard area counts must be within +100% to –50% from that of the mid-point calibration (1ug/mL) of the associated initial calibration.

8.4.3 Corrective Action for Continuing Calibration - Depending on which compound(s) fail(s) the criteria, corrective action included preparing fresh standards, source cleaning, changing GC column or injection liners. Repeated failure to pass continuing calibration may necessitate performing new initial calibration.

8.4.4 Continuing calibration acceptance criteria must be met before any samples or blanks are to be analyzed for generation of acceptable data.

8.5 Sample Analysis:

Prior to sample analysis, the sample extract and the internal standard are allowed to warm to room temperature to ensure complete dissolution of the high molecular weight internal standards. Ten microliters of the internal standard solution is added to each of the sample extracts at 1.0 mL final volume to ensure 5.0ng on-column amount. Shake the extract slightly to mix. The internal standard volume will be adjusted for smaller extract volume.

8.5.1 Analytical Sequence: The following QC protocol is recommended for analyses:

Initial Batch		Middle/Final Batch	
1.	Tune including Breakdown	1.	Tune
2.	ICal Standard #1	2.	%Breakdown
3.	ICal Standard #2	3.	CCV
4.	ICal Standard #3	4.	Method Blank
5.	ICal Standard #4	5.	LCS

6.	ICal Standard #5	6-13.	Samples (< 8)
7.	ICV	14.	CCV
8.	Method Blank	15.	Method Blank
9.	LCS	16.	MS
10.	LCSD	17.	MSD
11-18.	Samples (< 8)	18-25.	Samples (< 8)
19.	Tune (as required per 12 hr.)	26.	CCV (Final Batch Only)
20.	CCV (as required per 12 hr.)		
21.	Method Blank		
22.	MS		
23.	MSD		
24-31.	Samples (< 8)		

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative Retention Time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Coelution of the primary and confirmation ions.

9.2 Determining the Concentration of Target Compounds - Sample data should be reported in units of µg/L for aqueous samples and µg/Kg dry weight basis for solid samples.

9.2.1 Target Quantitation: Compounds identified are quantitated using the following equations:

For **aqueous** samples, Concentration *ug/L* $= \dfrac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(V_o)\,(V_i)}$

For **solid** samples, Concentration *ug/Kg* $= \dfrac{(A_x)\,(I_s)\,(V_t)\,(Df)}{(A_{is})\,(RRF)\,(W_s)\,(V_i)\,(S)}$

where: A_x = area of the characteristic ion for the compound to be measured

A_{is} = area of the characteristic ion of the associated internal standard
I_s = amount of internal standard added in nanogram
RRF = relative response factor
V_o = volume of water extracted in milliliters = 1,000
V_t = volume of the sample extract in milliliters = 1
Df = dilution factor
W_s = weight of soil extracted in grams
S = % solid

9.3 Rounding Rule – Use the most current EPA rounding rules.

9.4 Acceptance Criteria for Sample Analysis:

- the sample must meet both extraction and analysis holding time.
- the sample has to have a compliant tune, initial calibration and continuing calibration.
- the sample has to have a compliant method blank.
- the sample has to have a compliant LCS.
- the surrogate recovery per this SOP (**Section 10.5**) or client-specified criteria.
- all of the target analyte concentration should be within the calibration range.
- area count of each of the internal standards in the inclusive range of - 50% and +100% of the response of the continuing calibration
- retention time of each of the internal standards must not shift more than ± 0.5 minute from the continuing calibration.
- excluding the solvent front or the aldol condensation peak for solid extract analysis, no ion should saturate the detector

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)

where: SSR = spiked sample result
SR = sample concentration
SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1-D2)}{(D1+D2)/2} \times 100$$

where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results (IPANDA). To ensure the appropriate analyst is performing the analysis, the analyst's initials should be entered in the Enviroquant acquisition software (do not use the default value). The analyst processing and reviewing data will initial the Run Logbook when processing data.

10.2 Method blanks - A method blank is prepared and analyzed with every batch not to exceed 20 samples. Acceptance criteria for the method blank are as follows:

- The recovery of Benzo(e) pyrene-d12 must be within the acceptance limits discussed in **Section 10.5.**
- Method blank concentration for DoD full scan SW8270 projects must be less than one half the reporting limit.

10.2.1 The "B" qualifier is applied to positive sample results on Form 1 or LIMS Level 2 data sheet when the same compound is detected in the method blank.

10.3 Lab Control Sample (LCS) – A Lab Control Sample is a weight or volume of a clean reference matrix (anhydrous sodium sulfate or DI water) that is spiked with target analytes and surrogate spike and carried through the entire analytical procedure. It is used to determine the efficiency of extraction with the analytical processing and analysis of the samples.

10.3.1 Acceptance criteria for LCS:

- General acceptance: compliant Benzo(e)pyrene-d12 recovery

- Recovery of individual compounds within 45-135% with the exception of pentachlorophenol which uses a 5-135% limit. These limits are currently being used per the ACOE guidelines until enough points are collected for both soil and water matrices, at which time in-house derived QC limits may be substituted.

10.3.2 If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged

with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.4 Duplicate Matrix Spikes: Matrix spikes and matrix spike duplicate are performed to evaluate the accuracy and precision associated with the sample batch of similar matrix.

For samples that are known to contain target analytes, the laboratory should perform one matrix spike and duplicate. For clean samples and those without documented history, a duplicate set of matrix spikes is performed. Since the majority of the samples received at Mitkem do not have any documented history, Mitkem will perform matrix spike and matrix spike duplicate.

10.4.1 Acceptance criteria for Duplicate Matrix Spike:

Matrix spike and matrix spike duplicate are used to assess the effect of matrix interferences on the analysis of the target analytes and the recovery should be used as advisory guidelines to answer question posed above. Control limits are the same as discussed in **Section 10.3** but are used as advisory guidelines.

If any compounds are outside of the acceptance limits, their recoveries and/or RPD are qualified with the "*" flag on the recovery MS/MSD summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries:

10.5.1 The recovery of Benzo(e)pyrene-d12 in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.2 Acceptance criteria - The percent recovery of benzo(e) pyrene in blanks, samples, duplicate matrix spikes and LCS must be within the set QC limits of 45-135%. These limits may be updated to in-house derived QC limits by the QA Department on an annual basis.

10.5.3 Corrective Action for Recovery Failures:

10.5.3.1 Any sample which fails the above will be subjected to re-extraction. Any method blank which fails the above will be re-extracted with the associated samples.

10.5.3.2 If re-extraction and re-analysis of the sample demonstrate similar recovery performance, both sets of results will be reported to demonstrate matrix- related problems. Re-extraction is not required if the recovery is out of the above range for both the native sample and its duplicate matrix spikes.

10.5.3.3 All surrogate outliers will be flagged with an "*" on the surrogate recovery report (Form 2) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports.

10.6 Internal Standard Response and Retention Times:

10.6.1 The area count of the characteristic ion of each of the internal standards in the samples, blanks, duplicate matrix spikes and LCS must be within the inclusive range of -50.0% and +100% of the response of the internal standards of the continuing calibration.

10.6.2 The retention shift for each of the internal standards in the samples, blanks and LCS must be within ± 0.5 minute of those obtained from the associated Continuing Calibration.

11. Data Validation and Reporting

11.1 All raw data, including calibrations, QC results, and samples results, are peer reviewed for technical accuracy and completeness. Sample preparation logs, notebooks, and instrument logs are reviewed and signed daily by the supervisor. The laboratory supervisor reviews 100% of the data prior to report generation. The QA Director randomly reviews 10% of the data reported by the laboratory.

11.2 Reports are generated by the reporting group. The data submitted for report preparation is dependent on project requirements and is subjected to further reviewed by the project manager for reasonableness prior to release to the customer.

12. Corrective Action Procedures

Discrepancy reports are generated in the event of an out-of-control situation that cannot be corrected by the analyst. The procedure for submitting a discrepancy

report for the purpose of identifying the appropriate corrective action is covered in Corrective Action Procedure SOP No. 80.0007. Starting in 2006, corrective actions have been recorded in the Omega LIMS system in the Quality Control section/corrective action reports. All employees have access to LIMS and may initiate a corrective action. If help is needed, see the QA Director for assistance.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is archived by the health and safety officer and available to all laboratory personnel. In addition, laboratory personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and goggles when handling these reagents and work in a hood whenever possible.

13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Definitions and Acronyms

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan

15. References

U.S. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8270D, SW-846 Test Methods for Evaluating Solid Wastes, Update IV, Revision 4, February 2007.

Attachments:

1. **Table 1**: PAH Target Analyte List For Method 8270 SIM
2. **Table 2:** List of Acronyms.
3. **Table 3:** Selected Ions for Target Compounds and Surrogate.
4. **Figure 1**: DFTPP Tune and Chromatogram.
5. **Figure 2**: Continuing Calibration Standard Chromatogram and Quantitation Report.

Table 1. PAH Target Analyte List For Method 8270 SIM

Target Analyte	CAS Registry No.
Acenaphthene	83-32-9
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo(a)anthracene	56-55-3
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(g,h,i)perylene	191-24-2
Benzo(a)pyrene	50-32-8
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Indeno(1,2,3-cd)pyrene	193-39-5
2-Methylnaphthalene	91-57-6
Naphthalene	91-20-3
Phenanthrene	85-01-8
Pyene	129-00-0

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Table 2 – List of Acronyms

1,2-DCB	1,2-Dichlorobenzene
1,3-DCB	1,3-Dichlorobenzene
1,4-DCB	1,4-Dichlorobenzene
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
ACOE	Army Corp of Engineers
DoD	Department of Defense
RL	Reporting Limit
MDL	Method detection limit
MQL	Method quantitation limit
PQL	Project Quantitation limit
IS	Internal Standard
SS	Surrogate Standard
DFTPP	Decafluorotriphenylphosphine
LCS	Lab control sample
MS	Matrix spike
MSD	Matrix spike duplicate

Table 3. Selected Ions for PAH Target Compounds and Surrogates

Target Analyte	Primary Quantitation Ion	Confirmation Ion
Acenaphthene	154	153
Acenaphthylene	152	151
Anthracene	178	89
Benzo(a)anthracene	228	114
Benzo(b)fluoranthene	252	126
Benzo(k)fluoranthene	252	126
Benzo(g,h,i)perylene	276	138
Benzo(a)pyrene	252	126
Chrysene	228	114
Dibenz(a,h)anthracene	278	139
Fluoranthene	202	101
Fluorene	166	165
Indeno(1,2,3-cd)pyrene	276	138
2-Methylnaphthalene	142	141
Naphthalene	128	129
Phenanthrene	178	89
Pyrene	202	101
<u>Surrogate:</u>		
Benzo(e)pyrene-d12	264	132

* Note: Additional non-PAH compounds may be analyzed under SIM mode on a client requested basis

Figure 1

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8260.D

Date : 03-JAN-2008 17:38

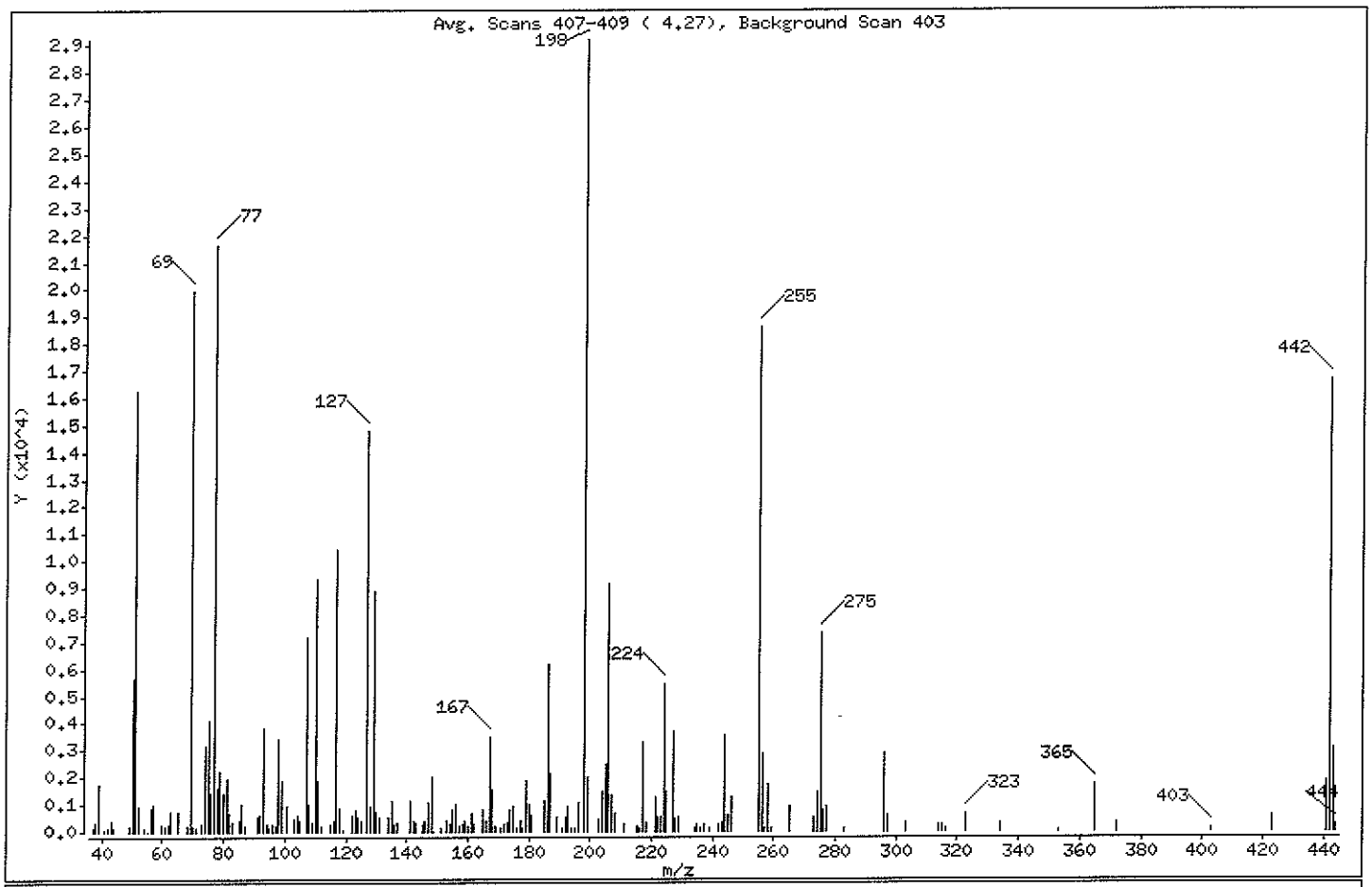
Client ID: DFTPP3S | Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0 | Operator: CLM

Column phase: DB-5MS | Column diameter: 0.25

1 dftpp



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	30.00 - 60.00% of mass 198	55.58
68	Less than 2.00% of mass 69	0.63 (0.93)
69	Mass 69 relative abundance	68.39
70	Less than 2.00% of mass 69	0.77 (1.13)
127	40.00 - 60.00% of mass 198	50.51
197	Less than 1.00% of mass 198	0.00
199	5.00 - 9.00% of mass 198	6.94
275	10.00 - 30.00% of mass 198	24.97
365	Greater than 1.00% of mass 198	5.87
441	Present, but less than mass 443	6.54
442	40.00 - 99.99% of mass 198	57.06
443	17.00 - 23.00% of mass 442	10.47 (18.35)

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8260.D

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S | Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0 | Operator: CLM

Column phase: DB-5MS | Column diameter: 0.25

Data File: S3E8260.D
Spectrum: Avg. Scans 407-409 (4.27), Background Scan 403
Location of Maximum: 198.00
Number of points: 180

m/z	Y	m/z	Y	m/z	Y	m/z	Y
37.00	126	99.00	1876	161.00	652	227.00	3694
38.00	345	101.00	957	162.00	263	228.00	455
39.00	1736	103.00	491	165.00	835	229.00	567
41.00	85	104.00	593	166.00	385	234.00	130
42.00	133	105.00	412	167.00	3471	235.00	236
43.00	431	107.00	7180	168.00	1531	236.00	101
44.00	113	108.00	1004	169.00	233	237.00	268
49.00	223	109.00	304	171.00	118	239.00	101
50.00	5664	110.00	9368	172.00	251	242.00	270
51.00	16247	111.00	1905	173.00	343	243.00	336
52.00	943	112.00	186	174.00	816	244.00	3547
54.00	146	115.00	273	175.00	919	245.00	607
55.00	27	116.00	436	176.00	147	246.00	1277
56.00	903	117.00	10447	177.00	391	255.00	18592
57.00	1024	118.00	885	178.00	126	256.00	2892
60.00	261	119.00	84	179.00	1906	257.00	117
61.00	209	122.00	631	180.00	1025	258.00	1759
62.00	262	123.00	836	181.00	623	259.00	167
63.00	734	124.00	513	185.00	1151	265.00	909
65.00	720	125.00	412	186.00	6167	273.00	518
68.00	185	127.00	14764	187.00	2152	274.00	1483
69.00	19992	128.00	948	189.00	533	275.00	7300
70.00	226	129.00	8882	191.00	115	276.00	837
71.00	112	130.00	752	192.00	530	277.00	958
73.00	286	131.00	565	193.00	920	283.00	132
74.00	3171	134.00	557	194.00	119	296.00	2884
75.00	4083	135.00	1134	195.00	102	297.00	625
76.00	1402	136.00	240	196.00	1047	303.00	317
77.00	21632	137.00	357	198.00	29232	314.00	250
78.00	1627	141.00	1174	199.00	2030	315.00	242
79.00	2243	142.00	425	203.00	490	316.00	101
80.00	1382	143.00	347	204.00	1470	323.00	682
81.00	1940	145.00	282	205.00	2453	334.00	318
82.00	694	146.00	409	206.00	9153	353.00	100
83.00	349	147.00	1046	207.00	1363	365.00	1717

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8260.D

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

Data File: S3E8260.D

Spectrum: Avg. Scans 407-409 (4.27), Background Scan 403

Location of Maximum: 198.00

Number of points: 180

m/z	Y	m/z	Y	m/z	Y	m/z	Y
85.00	412	148.00	2035	208.00	669	372.00	309
86.00	1023	149.00	22	211.00	293	403.00	132
87.00	201	151.00	109	215.00	228	423.00	575
91.00	526	153.00	434	216.00	137	441.00	1911
92.00	620	154.00	271	217.00	3296	442.00	16680
93.00	3798	155.00	776	218.00	309	443.00	3060
94.00	276	156.00	1027	221.00	1285	444.00	237
95.00	103	157.00	178	222.00	515		
96.00	269	158.00	284	223.00	564		
97.00	208	159.00	390	224.00	5411		
98.00	3434	160.00	190	225.00	1486		

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8260.D

Date : 03-JAN-2008 17:38

Client ID: DFTPP3S

Instrument: S3.i

Sample Info: DFTPP3S,DFTPP3S

Volume Injected (uL): 1.0

Operator: CLM

Column phase: DB-5MS

Column diameter: 0.25

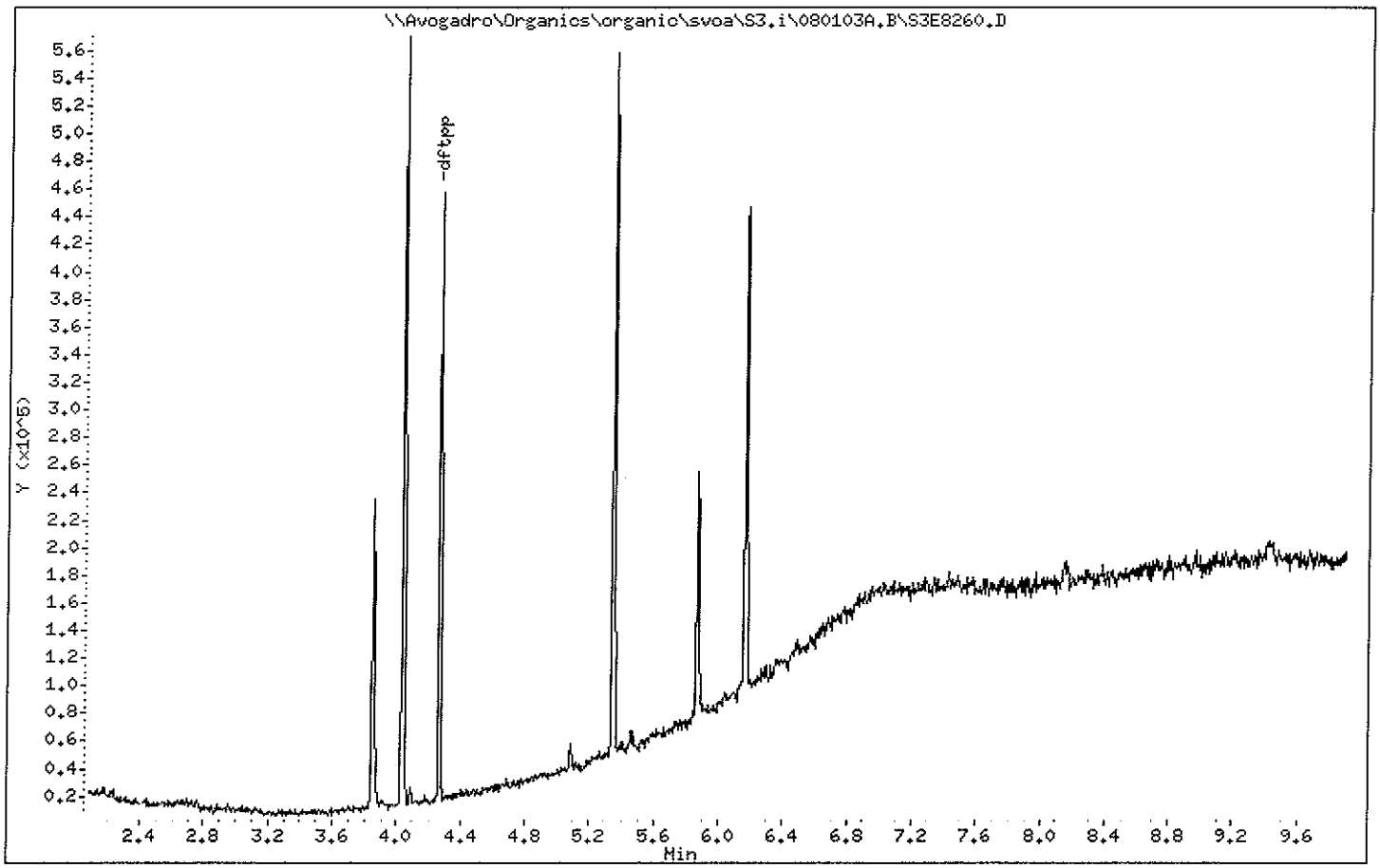
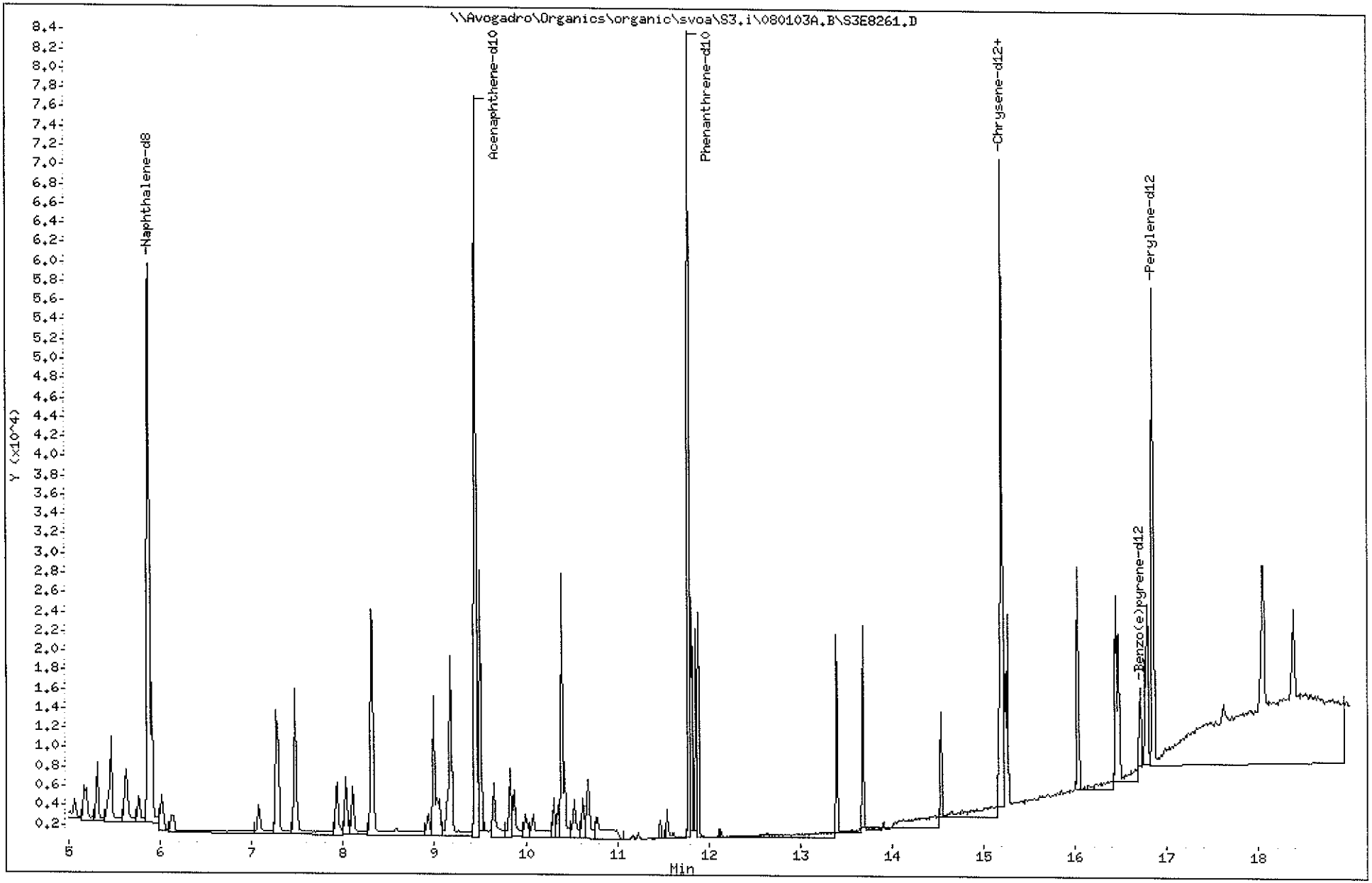


Figure 2

Data File: \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8261.D
Date : 03-JAN-2008 17:57
Client ID: SSTD0013S
Sample Info: SSTD0013S,SSTD0013S
Volume Injected (uL): 1.0
Column phase: DB-5MS

Instrument: S3.i

Operator: CLM SRC: CLM
Column diameter: 0.25



Data File: S3E8261.D
Report Date: 18-Jun-2008 09:33

Mitkem Corporation

SIM-PAH

Data file : \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\S3E8261.D
Lab Smp Id: SSTD0013S Client Smp ID: SSTD0013S
Inj Date : 03-JAN-2008 17:57
Operator : CLM SRC: CLM Inst ID: S3.i
Smp Info : SSTD0013S,SSTD0013S
Misc Info : 2,3
Comment :
Method : \\Avogadro\Organics\organic\svoa\S3.i\080103A.B\s3_pah_sim.m
Meth Date : 14-Jan-2008 12:36 S3.i Quant Type: ISTD
Cal Date : 28-DEC-2007 19:48 Cal File: S3E8155.D
Als bottle: 1 Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE Compound Sublist: PAH.sub
Target Version: 4.14

Concentration Formula: Amt * DF * Uf*(Vt/Vi)*(1/Vo) * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	GPC Factor
Vt	1000.000	Extract Volume (uL)
Vi	1.000	Injection Volume
Vo	1000.000	Sample Volume
Cpnd Variable		Local Compound Variable

Compounds	QUANI SIG MASS	RI	EXP RI	REL RI	RESPONSE	AMOUNTS CAL-AMT (ng)	AMOUNTS ON-COL (ng)
* 7 Naphthalene-d8	136	5.878	5.878	(1.000)	79435	5.00000	
8 Naphthalene	128	5.912	5.912	(1.006)	15041	1.00000	0.93
10 2-Methylnaphthalene	142	7.272	7.272	(1.237)	11217	1.00000	1.0
13 Acenaphthylene	152	9.169	9.169	(0.970)	20023	1.00000	0.89
* 14 Acenaphthene-d10	164	9.456	9.456	(1.000)	54655	5.00000	
15 Acenaphthene	154	9.504	9.504	(1.005)	12230	1.00000	0.91
18 Fluorene	166	10.397	10.397	(1.099)	15413	1.00000	0.95
* 23 Phenanthrene-d10	188	11.783	11.783	(1.000)	91276	5.00000	
24 Phenanthrene	178	11.814	11.814	(1.003)	22279	1.00000	0.89
26 Anthracene	178	11.886	11.886	(1.009)	21351	1.00000	0.88
27 Fluoranthene	202	13.397	13.397	(1.137)	20873	1.00000	0.85
28 Pyrene	202	13.681	13.681	(1.161)	22092	1.00000	0.62
30 Benzo(a)anthracene	228	15.187	15.187	(0.999)	19789	1.00000	0.94
* 31 Chrysene-d12	240	15.198	15.198	(1.000)	59780	5.00000	
32 Chrysene	228	15.230	15.230	(1.002)	18467	1.00000	0.96
33 Benzo(b)fluoranthene	252	16.445	16.445	(0.976)	17653	1.00000	0.86
34 Benzo(k)fluoranthene	252	16.477	16.477	(0.978)	18805	1.00000	0.98
$ 35 Benzo(e)pyrene-d12	264	16.712	16.712	(0.992)	8469	1.00000	0.97
37 Benzo(a)pyrene	252	16.786	16.786	(0.996)	15758	1.00000	0.94
* 38 Perylene-d12	264	16.850	16.850	(1.000)	44148	5.00000	
39 Indeno(1,2,3-cd)pyrene	276	18.034	18.034	(1.070)	16569	1.00000	0.95

Data File: S3E8261.D
Report Date: 18-Jun-2008 09:33

Compounds	QUANI SIG MASS	RI	EXP RI	REI RI	RESPONSE	AMOUNIS CAI-AMI (ng)	ON-COI (ng)
40 Dibenzo(a,h)anthracene	278	18 055	18 055	(1.071)	13767	1 00000	0.98
41 Benzo(g.h.i)perylene	276	18 375	18.375	(1.090)	14677	1.00000	1 00

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C

SOP No. 90.0012

Rev. 10

	Signature	Date
QA Director:		10/8/08
Lab Director:		10/8/08

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

for

Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Analysis by SW846 Method 8260C

Rev. 10

1. Scope and Application

This SOP describes the analysis of volatile organic compounds in aqueous and soil samples using gas chromatography/mass spectrometry (GC/MS). The SOP covers the analyses according to protocols discussed in SW846 Update 8/2006 of Method 8260C. A modified version of Method 8260C for a low level aqueous analysis using a 25 mL aliquot is included as **Section 16**. This SOP meets all of the requirements specified in the method. To further familiarize oneself with the procedures, the analyst is encouraged to consult the following instrument manual:

- Hewlett Packard EnviroQuant GC/MS Manual

2. Personnel Qualifications and Responsibilities

Personnel must be qualified according to the requirements of their job descriptions and trained for this procedure prior to analyzing samples. **Analysts and technicians** are responsible for performing analyses in accordance with the SOP and documenting any variations in the protocol or unusual occurrences noted during analyses. **Supervisors/Managers** are responsible for ensuring that SOPs are accurate and up-to-date, and that they are implemented appropriately. **Supervisors/Managers** review the logbooks and data generated from this procedure and approve reported results. The **Laboratory Director** and/or senior management evaluate laboratory reports for reasonableness of the results and signs the reports. The **QA Director** reviews the QC system and quality control generated to provide an assessment of data accuracy and precision.

3. Summary of Procedure/Instrumentation

3.1 The volatile compounds are introduced into the GC/MS by purge-and-trap system. Analytes are extracted from the sample by bubbling with helium. The analytes are trapped from the helium stream on an adsorbent trap. The analytes are desorbed at high temperature directly onto a narrow-bore capillary column after been split at 1:50 ratio via an EPC controlled injector for analysis. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer.

3.2 Analytes eluted from the capillary column are introduced into the mass spectrometer by direct interface to the ion source. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (primary) ion relative to an internal standard using a five-point calibration curve.

3.3 A list of acronyms used in this SOP is included in **Table 1**.

3.4 The list of compounds to be analyzed and reported may vary from project to project. The SW-846 method contains several different lists of analytes, so there is no "official" EPA list of Method 8260 compounds. Mitkem typically analyzes samples for and reports a fairly extensive list of analytes. Certain projects also may have additional extra compounds not on the normal Mitkem list. Alternatively certain projects may have a shorter list of compounds than the normal Mitkem list. These project-specific lists of analytes are specified by the client through discussion with the Mitkem Project Manager who discusses the list with the Laboratory Supervisor. The lists are managed in the lab by the use of "sublists" in the Target data reduction and reporting software. In addition, when utilizing the Omega LIMS system, the sublist can be viewed using the SEL list option. SEL refers to the select list of target analytes requested by the client. It is used when this list differs from the "routine" analyte list. A list of the routine 8260 analytes used by the laboratory is shown in **Attachment 1**. Refer to the LIMS Test Information category/Test option/ limits of the testcode, for the most current MDL values. Those listed in **Attachment 1** may not be the most up to date.

3.4.1 Several options exist for reporting extra compounds not on the normal Mitkem list. The ideal approach includes purchasing a calibration standard solution and a second source calibration check solution, determination of method detection limit from 7 or more replicate analyses and addition of the compound to initial and continuing calibrations and laboratory control sample analyses. (See Mitkem SOP No. 80.0005 for details on determination of the MDL). Depending on the needs of the client, alternative approaches may be appropriate, including single point calibration or searching for the compound as a Tentatively Identified Compound using the Target software's library search routines. The approach taken must be discussed with the client prior to

analyses, and if needed, sufficient documentation is included in the analysis report to enable validating the data. The analyst will be instructed by the lab supervisor as to what documentation is needed and what is required to be sent to the data reporting area for inclusion in the final report.

3.4.2 The Quality Control requirements contained in this SOP apply to the specific list of analytes being reported. SOP criteria are to be evaluated for all project target analytes. While QC issues with non-project target analytes should be investigated, they are not critical if that compound is not being reported. Mitkem's calibration standards and LCS/MS solutions may contain compounds not being reported for a particular project.

4. Sample Preservation, Containers, Handling and Storage

4.1 Samples are collected by the client and submitted for analysis in pre-cleaned sample containers provided by the laboratory. For volatile organic compound analysis by method 8260, water samples are collected in 40-milliliter (mL) glass vials, typically preserved with HCl. Solid samples may be collected in glass containers, EnCore™ samplers, pre-weighed 40-mL vials preserved by 5 mL of DI water to be frozen upon receipt or similar pre-weighed vials with sodium bisulfate solution for low-level analysis or preserved by 5 mL methanol for medium-level analysis. According to Method 5035, the low-level soil samples shipped in EnCore samplers need to be extruded into a pre-weighed DI water/stir bar vial or similar vial with sodium bisulfate solution as soon as received in the lab (within 48hours of collection). If needed, the soil samples received in EnCore samplers may be preserved by storing in a dedicated freezer until prior to the analysis. This freezer only contains samples and must not contain any analytical standards. ASTM method D6418-04 includes documentation of the ability of EnCore devices to contain volatile compounds without significant loss when frozen for up to 14 days. Sample volume requirements depend upon the number of different preparation procedures necessary for the analyses requested. Additional sample volume may also be required for the analysis of laboratory QC samples. Typical sample submittals are listed below:

- "normal aqueous samples: 2 X 40ml vials, HCl Preserved
- "low-level" aqueous samples: 3 X 40ml vials, HCl Preserved
- preserved soil samples: 2 X DI water/freeze preserved or sodium bisulfate preserved plus 1 X methanol preserved plus a four-ounce jar for percent solids determination

Other sample submittals may be suitable for analysis depending on the needs of the specific project. The Project Manager, Supervisor or client should be contacted to determine the suitability of sample containers to meet SOP and project objectives.

4.2 All aqueous samples and soil samples received in glass jars are stored in the VOA lab at 4°C ± 2°C until analyzed. The soil samples received in 40mL vials preserved either by sodium bisulfate or methanol are also stored at 4°C ± 2°C in the VOA lab. Soil samples received in pre-weighed 5mL DI water vials are stored in a freezer at down to -20°C . For soil samples received in the Encore type of device (typically in silver pouches), these are extruded into pre-weighed vials containing a stir bar and 5mL of DI water, then re-weigh the vials to obtain the final sample weights. The vials are to be placed into a freezer.

4.3 Storage areas used for samples for volatile organic analysis must be free of potential contaminants. To document storage conditions a storage blank consisting of a 40ml vial of organic-free water is placed in every refrigerator used to store VOC samples on a weekly basis. Storage Blanks (refrigerator and freezer where applicable) are logged in to the LIMS system and tracked using the reporting feature. These will be analyzed a weekly basis. When a storage blank is removed for analysis, another blank will be placed in the refrigerator such that there will be a blank in each refrigerator on a 24/7 basis. Highly contaminated samples are stored in a specially marked refrigerator in the VOA lab.

4.4 Sample holding time for volatile organic compound analysis by method 8260 are 14 days from the day of sample collection for both preserved aqueous and soil samples. The holding time for non-preserved aqueous samples is 7 days from the day of sample collection. Samples will be disposed after a minimum of 30 days after the submission of a complete data package.

5. Interference and Potential Problems

5.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non PTFE thread sealant, plastic tubing, or flow controllers with rubber components should be avoided; since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting value result in what the laboratory feels is a false positive result for a sample due to laboratory background contamination; the laboratory should fully explain this in text accompanying the uncorrected data. Compounds detected in method blanks and also detected in samples from the same batch are qualified with a "B" flag on data report forms, and listed on the data review checklist. The definition of the "B" qualifier is included in the data report.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the purging apparatus and sample syringes must be rinsed with at least two portions of organic-free

reagent water between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of one or more blanks to check for cross-contamination.

5.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device in the sample flow path including purging vessel, tubing, or sample valves may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is helpful to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by analysis of an extra aliquot at a dilution beyond the 12 hour instrument tune time or comparison to any available previous results for the sample.

5.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PFTE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination. It is important for analysts to keep this in mind if they enter the organic preparation laboratory and plan to return to the volatiles lab. Their clothing may be a source of contamination in the volatiles lab.

5.5 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water or other matrix and carried through the sampling, handling, and storage protocols serve as a check on such contamination. Trip blanks are typically sent with each shipment of VOA vials and the client is advised to have them analyzed. The client is also advised to prepare field blanks to be sent to the lab if appropriate for their sampling plan.

5.6 Use of sensitive mass spectrometers or larger sample sizes to achieve lower detection levels will increase the potential to detect laboratory contaminants as interference.

5.7 Direct injection (for BFB) – Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in case of extreme contamination.

6. Equipment and Apparatus

6.1 Equipment:

6.1.1 There are four GC/MS instruments in the Volatile Organic Analysis Lab. There are V1, V2, V5 and V6. The GC/MS systems have similar configurations as follows: Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V1); Hewlett Packard (HP) 5890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V2); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5972A mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V5); Hewlett Packard (HP) 6890 GC interfaced to a HP Model 5973 mass spectrometer (GC/MS) connected to an OI Model 4560 Purge and Trap Concentrator with an OI Model 4552 autosampler (V6). The laboratory maintains flexibility to interface various purge and traps to various autosamplers and subsequently to various GC or GC/MS systems. This flexibility is an important tool in trouble-shooting system problems or to minimize the impact of instrument down-time. EnviroQuant Software is used to handle data acquisition. Data files are copied and transferred to the company file server via the computer network. Actual data quantitation and analyses are performed by the analyst using Target chromatographic software (Thru-Put systems, Inc.).

6.1.2 A 30m x 0.25mm id DB-624 capillary column is used for all three GCs. The GC is directly interfaced to the MS. The GC injector operates under split mode (about 50:1) at all times.

6.1.3 OI 4552 autosamplers are fitted with heat function for low level soil analysis (V2, V5 and V1).

6.1.4 High purity helium (99.999%) is used both as GC carrier gas and Purge and Trap purge gas

6.1.5 The instruments scan from amu 35 to 300 at EM voltage similar to those of the tunes.

6.1.6 The BFB data acquisition method is V1TUNE, V2TUNE, V5TUNE and V6TUNE for each of the four instruments.

6.1.7 The data acquisition methods for unheated purge (for aqueous and medium level soil samples) are V1VOA, V2VOA, V5VOA and V6VOA. The data acquisition methods for heated purge (for low-level soil samples) are named similarly.

6.1.8 The purge and trap systems operating conditions are as follows:

Aqueous (including both 5mL and 25mL samples) and medium soil samples

Purge	11 min at ambient temperature
Dry Purge	2 min
Desorb*	2 min at 190 °C
Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)
Transfer Line Temp.	125 °C

Low level soil samples

Preheat	2 min
Purge	11 min at 40 °C
Dry Purge	2 min
Desorb*	2 min at 190 °C
Bake*	8 min at 200 °C
Purge Flow	40 mL/min
Trap Type	OI Analytical Trap #10 (containing 8cm each of Tanax and silica gel and carbon molecular sieve)
Transfer Line Temp.	125 °C

* Desorb time and Bake times may be varied to optimize the instrument performance.

6.1.9 Instrument operating conditions are as follows:

General Gas Chromatography Conditions

For V1 and V2:

Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	10 °C/min to 120 °C, then 15 °C/min to 240 °C
Final Time	2 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

For V5 and V6:

Carrier Gas	Helium (99.999%)
Column Flow	25mL/min
Initial Temperature	38 °C hold for 1.8 min
Temperature Program	12 °C/min to 200 °C, then 20 °C/min to 240 °C
Final Time	2.7 min
Injector Temperature	125 °C
Transfer Line Temperature	280 °C

General Mass Spectrometry Conditions

For V1 and V2:	
Mass Range	35-300 amu
Scan Speed	1.6 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune
For V5 and V6:	
Mass Range	35-300 amu
Scan Speed	0.97 scans/min
Ionization Mode	70 eV positive ion
EM Voltage	same as tune

6.1.10 Balance

- A top-loading balance capable of weighing 200.0 ± 0.1 g.

6.2 Preventative Maintenance - The Purge and Trap GC/MS are maintained according to the manufacturers' recommendations. The lab analyst performs preventive maintenance as discussed below:

6.2.1 The Purge and Trap spargers are rinsed and cleaned automatically by autosampler between each analysis. This cleaning procedure is sufficient for most sample analyses. Some samples exhibited target compounds or TIC at unusual high concentrations result in contaminating the Purge and Trap system that may require additional cleaning and baking out. Under the circumstances, any part on the flow path directly contacting with sample including transfer line, valves and sparger may need to be back-flushed with VOC-free water, then with methanol, and extra baking. Analyst performs this extensive cleaning procedure should record them into the LIMS maintenance logbook (see **Section 6.2.9**). The system must be demonstrated to be contaminant free by analyzing instrument blank before reuse for sample analysis. The trap will be replaced if tailing peak response and loss of gaseous compounds that can not be related to standard solution problems are observed.

6.2.2 The GC septum will be replaced monthly (this is done rarely as the septum is only penetrated whenever BFB tuning compound is analyzed.

6.2.3 The maintenance of GC injection liner will be performed as needed. If necessary, the liner will be replaced.

6.2.4 If needed, the analytical column will be replaced; this is usually indicated by excessive peak tailing and/or repeated failures of initial or continuing calibration verifications to meet SOP criteria.

6.2.5 The ion source will be cleaned when the system drifts out of BFB tune and/or repeated failure of initial and/or continuing calibrations to meet SOP-specified criteria.

6.2.6 If there is a second blown filament, the ion source will be vented to install two new filaments. Whenever the ion source is opened for maintenance, the analyst should make sure two good filaments are in place, or replace any filament blown since the last maintenance. This will minimize the times when both filaments are blown.

6.2.7 The pump oil will be replaced as needed.

6.2.8 Corrective maintenance is needed if the lab analyst or his/her supervisor fails to diagnose and/or correct the problem. The analyst or lab supervisor will promptly notify the manufacturer of the problem to schedule on-site diagnosis and repair.

6.2.9 All non-routine preventive and corrective maintenance shall be documented in the Instrument Maintenance log located in the Omega LIMS system. This can be accessed using the category Analytical and option Instruments in the LIMS menus. All analysts have access to this function in LIMS. If help is needed, ask the Lab Supervisor for assistance.

6.3 Troubleshooting - Refer to troubleshooting section of the HP 5972A MSD and 5973 MSD hardware manuals.

6.4 Glassware:

6.4.1 Class "A" volumetric flasks: 10 mL and 100 mL.

6.4.2 Syringes: 2 uL, 5 uL, 10 uL, 25 uL, 50 uL, 100 uL, 500 uL, 2.5 mL, 5 mL, and 25 mL.

6.4.3 Syringe value - Two-way, with Luer ends (three each) if applicable to the purging devise.

7. Reagents and Standards

7.1 Analyte-Free Reagent Water (also referred-to as VOC-free water or DI water elsewhere in this SOP) - prepared by eluting tap water through a column of activated charcoal granules to remove traces of volatile organic compounds.

7.2 Purge and trap grade methanol - from Fisher Scientific *or equivalent* quality of solvent from other vender will be used for standard preparation. Each new batch of solvent is checked by analyzing a 200µL aliquot of the methanol in a 5mL aliquot of pure water, or 1.6mL per 40ml vial of pure water. The new batch is acceptable if the analysis does not detect any contaminants that interfere with the measurement of target analyte compounds, or contain unacceptable levels of non-target compounds. While the criteria for method blank evaluation can be used for guidance, stricter criteria will be beneficial as potential contamination from various sources may add-up to impact the analysis.

7.3 The standards used for this SOP are discussed below. *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.* Stock solutions for calibration standards are:

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Ultra	DWM-589N	2000 ug/mL
Gas Mix	Restek	30042	2000 ug/mL
Ketone Mix	Restek	30006	5000 ug/mL
Additional Mix	Ultra	CUS-6268	400-8000 ug/mL
Internal Standards	Restek	30241	2500 ug/mL
Surrogates	Restek	30240	2500 ug/mL

7.3.1 All of the primary standards are labeled as VPyymmddX,

where: VP = **V**olatile **P**rimary standard
yymmdd = date the standard is received
X = the order that the standard is logged into the Log Book on that date, in increasing alphabetical order

All of the standards are recorded in the Primary Standard Logbook (**Figure 1**) upon receiving.

7.3.2 All unopened ampulated primary standards will be replaced either by following the expiration instructions from the manufacturer, or after two years from the date received if no expiration date was provided. For an opened stock standard

ampule, replace after 6 months from the date it was opened or sooner if the standards have degraded or evaporated.

7.3.3 The laboratory will at all times archive one complete set of unopened ampulated standards (to include internal standards, tune standards, and surrogate standards). New standards will be ordered if the inventory falls below the above mentioned criteria.

7.3.4 Standards are stored separate from samples, at a temperature of ≤ 6° C or as recommended by the vendor. Mitkem stores standards in a separate freezer which is maintained between -10 and -20 ° C.

7.4 Stock Solution (independent "second" source, *Please note that standards from other vendors could be used as long as the standards are of high purity (> 96%) and traceable to reference materials.*) for ICV:

Standards	Vendor	Cat. No.	Concentration
8260 Mix	Accu Standard	M-502A-R	200 ug/mL
Gas Mix	Accu Standard	M-502B	200 ug/mL
Additional Mix	Accu Standard	M-8260-ADD	200 ug/mL

7.4.1 The labeling and storage for all ICV primary standards should be handled following the similar procedures for primary calibration standards listed above in **Section 7.3.1 through 7.3.3.**

7.4.2 All of the standard information is recorded in the Primary Standard Logbook upon receiving (**Refer to Section 7.3.1**). All vials containing standards must be labeled according to the current version of SOP 80.0001 Standard Preparation, Equivalency and Traceability.

7.5 Working Standard solutions:

7.5.1 Working standard solution for calibration mix: place 2520 µL of methanol into a 4mL vial fitted with Teflon septum. Transfer 200 µL of 8260 Standard Mix (Cat. No. DWM589N), 200 uL of Gas Mix (Cat. No. 30042), 80 uL of Ketone Mix (Cat. No. 30006) and 1000 uL of Additional Mix (Cat. No. CUS-6268) into the vial to make a solution of all analytes at 100 ug/mL.

Note: using the syringe, add the standards below the surface and into the methanol by pushing the syringe plunger smoothly to the end. Make sure all of the standard mix is transferred into the methanol before the syringe is removed.

Gently invert the 4mL vial several times to ensure proper mixing. Repeat this process for all mixtures. This working standard is then transferred into four 1

mL vials with mininert valve. Keep all four vials in freezer at -10 to -20°C. Only take one vial out and use it for calibration and QC samples. A new vial should be taken out and used weekly, or as needed based on standard degradation.

7.5.2 Working standard solution for ICV: place 700 uL of methanol into a 1mL vial fitted with Teflon septum. Transfer 100 uL each of Non-Gaseous standards (M-502A-R), 8260 Additional standards (M-8260-ADD) and Gaseous standards (M-502B) into the vial to make a solution of all analytes at 100 ug/mL.

7.5.3 Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 400 uL each of the IS stock solution (Cat. No. 30241)and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3200 uL of methanol to make a 250 ug/mL solution. This solution once prepared is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 5 mL purge volume, this yields a concentration of 50 ug/L or ug/Kg.

7.5.4 All of the working standards are labeled as VWyymmddX,

where: VW = volatile working standard
yymmdd = date the standard is prepared
X = the order that the standard is logged into the Log Book on that date, in increasing alphabetical order

See **Table 2** for details on making working standard solutions. All of the standard preparation information is recorded in the Working Standard Logbook **(Figure 2)**. All vials containing working standards must be labeled according to the current version of SOP 80.0001 Standard Preparation, Equivalency and Traceability. Be sure the vial label is not worn or difficult to read. Any vial whose label becomes worn or difficult to read should be re-labeled. Working Standards are good for one month. All standards made from a primary standard must not exceed the primary standard's expiration date.

8. Procedure

8.1 Tuning:

8.1.1 The GC/MS must be tuned to meet 4-bromofluorobenzene (BFB) criteria every 12 hours when standards, blanks or samples are to be analyzed.
All of the analysis information is to be recorded in the Instrument Run Logbook (**Figure 3**). The logbook is issued by the QA officer and will be returned back to the QA officer for archiving when all pages are used.

8.1.1.1 Procedure for performing tune - Use the GC/MS conditions in **section 6.1.1.5** to perform the tune analysis.

Inject 2 µL of the working tune standard (50 ng) directly into the GC/MS through the septum injection port using a 10uL syringe. Alternatively, the BFB can be introduced through the purge and trap system, as a sample is.

A typical BFBchromatogram is shown in **Attachment 2.**

8.1.1.2 Acceptance criteria for tune - The mass spectrum of BFB must be acquired across the peak. The primary mean for evaluating ion abundance is averaging three scans: the peak apex scan and the scans immediately proceeding and following the apex. One of following alternates may be used to evaluate the tune: averaging the entire BFB peak, the single scan of apex, the single scan before apex, or the single scan after apex. Background subtraction is required and accomplished by subtracting a single scan no more than 20 scans prior to the beginning of the elution of BFB. It is important that the analyst does not selectively add or subtract scans to meet the tune criteria.

A typical mass spectrum and mass spectral listing of the tune in listed in **Attachment 3**.

The acceptance criteria are as follows:

Mass	Ion Abundance
50	15.0 - 40.0% of mass 95
75	30.0% - 60.0% of mass 95
95	base peak, 100%
96	5.0 - 9.0% of mass 95
173	< 2.0% of mass 174
174	>50.0 % of mass 95
175	5.0 - 9.0% of mass 174
176	95.0 - 101.0% of mass 174
177	5.0 - 9.0% of mass 176

Once the mass spectrometer passes the BFB tune, all subsequent blanks and standards that are analyzed within the 12-hour shift must be analyzed using identical mass spectrometer instrument conditions.

8.2 Initial Calibration - Initial Calibration is performed after the instrument passes the tune requirements. Initial Calibration is required after major instrument maintenance

including source cleaning and/or changing column. Initial Calibration will also be performed if Continuing Calibration analyses do not meet QA/QC criteria.

8.2.1 Five calibration standard solutions are required for all target compounds. Standard concentrations of 5, 20, 50, 100, 200 µg/L (or in µg/kg for soil samples) for typical 5 mL or 5 g sample analyses; 0.5, 4, 10, 20, 40 µg/L for 25 mL aqueous sample analyses are required.(See Section 16 for more details on low level calibration) The lowest standard concentration is typically at or below the reporting limit for the analysis. This is the level closest to the method detection limit (MDL). There may be project-specific requests to calibrate the instrument to the 1 ug/L level using a 5ml purge. This procedure may involve the addition of a sixth level to the initial calibration at the 1 ug/L concentration, or replacement of one of the other concentrations to maintain a 5 level calibration. This is determined by the requirements of the specific project through discussions between the Project Manager and Supervisor. There also may be occasional requests to report results to a limit below the lowest initial standard concentration. These must be documented and discussed in the report narrative. Any request for non-routine calibration should be discussed with the laboratory Supervisor and Project Manager to insure the resulting data meets project and method requirements and the procedures used and the quality of the data are fully documented.

DoD– The ICAL range shall consist of a minimum of 5 contiguous calibration points for organics, for all analytes reported. The low-level standard must be less than or equal to the reporting limit.

Several state and government programs have specific QA/QC Requirements and Performance Standards for the Initial Calibration. Refer to the individual state/government documents for more details. In particular, Dept. of Defense requires the evaluation of SPCC/CCC compounds in both the ICAL and CCV. See Attachment 5 for criteria.

Low level (25 mL) Calibration is documented in **Section 16**.

The calibrations for aqueous and medium level soil samples are performed at ambient temperature purge. The calibrations for low-level soil samples are performed at the same temperature used for sample analysis, using heated (40°C) purge condition.

8.2.2 Initial Calibration standards are made-up as follows:

Initial calibration standards are prepared by adding working standards into 40mL organic-free water. In aqueous analysis, 5mLs of each 40mL solution are transferred into the purge chamber by the auto-samplers. In soil analysis, 5mLs of each 40mL solution are transferred into vials with Teflon caps

manually using gas tight syringes and these vials are heated and purged. The IS/SS are added automatically by the autosampler.

Calculation for Initial Calibration - A typical chromatogram of a 50µg/L standard followed by the Quantitation Report is shown in the attachments to this document.

From the 5 level calibration, the relative response factor (RRF) for each target compound is determined using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where: A_x = area of the characteristic ion for the target compound to be measured
A_{is} = area of the characteristic ion for the associated internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

When using the DB624 column, note the following when determining the RRF:

- Cis- and trans-1,2-dichloroethenes are separately calibrated.
- o-Xylene is calibrated by itself; the m- and p-isomers are summed together.

The mean relative response factor is determined by averaging the 5 level values.

The % Relative Standard Deviation (%RSD) of the RRF is also calculated using:

$$\% RSD = \frac{SD}{Mean} \times 100$$

where: SD = Standard Deviation, and

$$SD = \sqrt{\frac{(Xi - X)^2}{n-1}}$$

where: Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values = 5

8.2.3 Initial Calibration acceptance criteria for SW-846 8260C protocol is as follows:

- The relative retention time (RRT) for each of the target analyte including the surrogates at each calibration level must be within ± 0.06 RRT of the mean RRT for each compound.
- The area response for each internal standard at each calibration level must be within the inclusive range of –50% to +100% of the mean area response of the internal standard in all of the calibration levels.
- The retention time (RT) shift of the internal standards at each calibration level must be within ± 0.5 minutes compared to the mean retention time over the initial calibration range for each internal standard.
- If the RSD of any target analytes and/or surrogate compounds is less than 20% , then the RRF is assumed to be constant over the calibration range and the average RRF is used for quantitation. If the calibration is not linear, make sure whether the problem is related to calibration standards or instruments.
- A minimum RRF is suggested, see **Table 3**.
- No quantitation ion may saturate the detector.
- Given the large number of target analytes, it is likely that some analytes may exceed the acceptance limit. In those instances, the initial calibration is deemed acceptable if the following conditions are met (in order of preference):

  (1) Ten percent (10%) of the compounds are allowed to be greater than 20% RSD with a maximum of 50% RSD. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are not allowed as one of the outliers. Initial Calibrations with over 10% of the compounds above the 20%RSD may be used for screening purposes.

  (2) Linear calibration: a least squares regression may be used. The analyst may employ a regression equation for the analyte(s) that does not pass the earlier approach. The regression will produce the slope and intercept terms for the following linear equation:

  $$y = mx + b$$

  Where y = instrument response (peak area)
  m = slope of the line
  x = concentration of the calibration standard
  b = intercept

  It is important that the origin (0,0) is not included as the sixth calibration point and that the above equation is not forced through the origin.

The linear regression is deemed acceptable if the correlation coefficient **r ≥ 0.995**.

(3) Non linear calibration: The analyst may employ a non linear regression coefficient of determination (COD). The second order quadratic fit will have the following equation:

$$y = ax^2 + bx + c$$

Where y = instrument response (peak area or height)

a and b = slope of the curve
x = concentration of the calibration standard
c = intercept

In performing second order quadratic fit, the analyst should not force the curve to pass through the origin (0,0). In addition, the origin should not be used as an additional calibration point.

From the quadratic fit, the "goodness of fit" is evaluated by calculating the coefficient of determination (COD). In order to be acceptable, the COD of the polynomial must be **≥0.99**.

8.2.4 Second source calibration verification – a second source calibration verification or initial calibration verification (ICV) is performed after the completion of the multi-level calibration. This is performed by analyzing the 50 ppb standard prepared in **Section 7.4.** The standard ID is documented in the run log. The acceptance criteria are as follows:

For routine SW 8260 analyses, the calculated value of the analyte in the ICV should be 70 – 130% of the expected value (35 – 65 ng/uL).

For DoD analyses, the calculated value of the analyte in the ICV should be 75 – 125% of the expected value (37.5 – 62.5 ng/uL), with no allowance for poor performing compounds.

If the above criteria are not met, the analyst has to evaluate the integrity of the primary and second source standards. First, reanalyze the ICV. Preparation and analysis of a new initial calibration may be required, however failure to meet the 70-130% limits does not in itself negate the Initial Calibration validity. Certain compounds may not meet the criteria under the best of circumstances. Some compounds will require a wider recovery limit. In some cases, the analysis of samples may be used for screening purposes when the ICV fails.

8.2.5 Corrective Action for Initial Calibration - Depending on which compound failed the criteria, corrective action included preparing fresh standards, source cleaning, reconditioning or changing the trap. Document the actions and resolution in the LIMS maintenance log.

8.2.6 Initial calibration acceptance criteria must be met before any sample, blanks or QC are to be analyzed. There may be circumstances where project-specific criteria allow the use of an initial calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 20% of the non-CCC analytes (calibration check compounds(CCC) are: vinyl chloride, 1,1-dichloroethene, chloroform, ethylbenzene, toluene, and 1,2-dichloropropane) to have %RSD > 30 or r < 0.99. This situation is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.2.7 If necessary, the reference spectra in Target are updated from mid-point calibration (50 ppb standard), or from the continuing calibration standard.

8.2.8 Upon the successful completion of the initial calibration, the raw data are arranged in increasing concentration levels together with BFB tune. Raw data include chromatograms and quantitation reports plus any documentation of manual integrations. Refer to SOP No. 110.0008 for details on the need for and documentation of manual integration. A copy of the initial calibration summary listing the RRF and %RSD of each target analyte is also included. These raw data are to be filed separately for the four instruments.

8.2.9 Initial calibration data must be archived in the company's organic analysis calibration (OCAL) database. The information in **Section 8.2.8** is brought to the Data Reporting area and left in the tray for filing OCAL data. The Data Reporting department will scan the calibration printouts into the optical filing database for long-term archiving. This may be done at anytime after the ICAL is deemed acceptable.

8.3 Continuing Calibration (CCV) - Continuing Calibration standards containing all the target compounds at 50 µg/L(or 50 µg/kg) is performed every time samples are analyzed to ensure that the GC/MS continues to meet instrument sensitivity and linearity requirements.

8.3.1 Frequency of Continuing Calibration - The Continuing Calibration standard must be performed once every 12 hours. If time remains in the 12-hour time

period after meeting the acceptance criteria for the Initial Calibration, samples may be analyzed using the mid-point ICAL standard as the continuing calibration verification. The Continuing Calibration is required whenever blanks, LCS and samples are analyzed.

8.3.2 Procedure for performing Continuing Calibration - The Continuing Calibration is performed at 50 µg/L (µg/kg) injection. The IS/SS are added automatically by the autosampler. Calculate the % difference between the Continuing Calibration RRF and those from the most recent Initial Calibration.

The % difference is determined as follows:

$$\% \text{ Difference} = \frac{RRF_c - RRF_i}{RRF_i} \times 100$$

where: RRF_c = relative response factor from continuing calibration

RRF_i = mean relative response factor from the most recent initial calibration that meets acceptance criteria.

Use % Drift when using least squares or non-linear calibration.

$$\% \text{ Drift} = \frac{Conc_c - Conc_t}{Conc_t} \times 100$$

where: $Conc_c$ = concentration obtained from continuing calibration

$Conc_t$ = theoretical concentration of standard

8.3.3 Continuing Calibration acceptance criteria:

- the RRF for each compound should be greater than those listed in **Table 3**.
- Twenty percent (20%) of the compounds are allowed to be greater than 20 %D, with a maximum of 50 %D. The number of outliers depends on the number of compounds of interest. Project specific compounds/common compounds are not allowed as one of the outliers.
- The area response for the internal standards must be within the inclusive range of –50% to +100% of the area response of the internal standards in the mid-point ICAL standard level.
- The internal standard retention time of the calibration verification standard must be within 30 seconds from that of the mid-point calibration (50 ug/mL) of the associated initial calibration.
- No quantitation ion may saturate the detector.

Several states have specific QA/QC Requirements and Performance Standards for the Continuing Calibration. Refer to the individual state documents for more details.

8.3.4 Corrective Action for Continuing Calibration - Investigate the calibration to confirm that calculations have been performed correctly and that all integrations are correct. Depending on which compound(s) fail(s) the criteria, corrective action includes preparing fresh standards, source cleaning, reconditioning or changing trap. Repeated failure to pass continuing calibration may necessitate performing new initial calibration. See **Attachment 8** for specific QC criteria and corrective action.

Note—the following symptoms and corrective actions commonly occur in this analysis. If the gaseous compounds are low, this typically indicates too high purge flow rate, "blowing" these compounds through the trap. The gaseous compounds are also more sensitive to small leaks in the system between the purging chamber and the injection port. If the higher boiling point compounds are too low, this typically indicates too low purge flow, or too low desorb temperature. A cold spot in the transfer line could also cause loss of the higher boiling compounds. If the brominated compounds or 1,1,2,2-tetrachloroethane are low, this typically indicates active sites in the system causing break-down of these compounds. If methylene chloride or acetone are too high or too low, this typically indicates contaminated "blank" water used to make the CCV or possibly in the ICAL (CCV too low).

8.3.5 Continuing calibration acceptance criteria must be met before any samples or QC are to be analyzed. There may be circumstances where project-specific criteria allow the use of a continuing calibration where one or more compound exceeds the acceptance criteria. For example, work performed under the Massachusetts Contingency Plan (MCP) allows up to 10% of the non-CCC analytes to have %D > 30. This is to be discussed with the Technical Director or Project Manager for approval. Any compound not passing the calibration criteria will be flagged on Form 7 and the information included in the data report. This information will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

8.4 Sample Analysis - Samples are allowed to warm to ambient temperature before analysis.

8.4.1 Aqueous (method SW5030) - Samples are analyzed in 5mL or 25mL aliquots depending on desired reporting limits or by project specification. The sample is entered into the instrument run log, the bottle number documented, and the vial is placed in a location in the autosampler tray. Immediately prior to analysis, an aliquot of the sample is withdrawn from the VOA vial by the autosampler using a syringe. An aliquot of the combined internal

standard/system monitoring standards (IS/SS) are added and the sample aliquot is transferred to the purge-and-trap sparger and injected into the sparger vessel. The sample is ready for analysis.

8.4.2 Soil samples are analyzed using heated purge for low level analysis and methanol extraction approach for medium level analysis.

8.4.2.1 Low level soil analysis (method SW5035) – Samples are received preserved in DI water or sodium bisulfate (NaHSO4) solution. The vial also contains a small teflon-coated stir bar. The "empty" vial/preservative solution/stir bar is weighed prior to shipment to the client/field. The vial is reweighed prior to analysis and the sample weight is determined by the difference in weight. The weight is recorded in the appropriate log book, and the sample and its jar nuber are logged into the instrument run log book. The vial is allowed to warm to room temperature and loaded into the autosampler. Prior to analysis the autosampler places the vial in a temperature controlled heating block to equilibrate to the analysis temperature 40 °C. For low level soil analysis, the instrument calibration and all QC analyses are to be performed at the same temperature (40 °C) as the sample analyses.

8.4.2.2 EnCore Samples – Samples collected into self-contained EnCore (or similar) devices are often collected. Samples are extruded from the EnCore into preservative solution (typically two EnCores into DI water and one into methanol. The aliquots are then analyzed by the low level soil or medium level soil procedure as appropriate.

8.4.2.3 Unpreserved Soil Samples – Samples for Method 8260 should be preserved per Method 5035. If soil samples are received unpreserved, but per discussion with the client they are still to be analyzed, the following procedure is used. Approximately 5.0-5.5g soil are weighed into a pre-weighed vial containing DI water and a stir bar. This should occur as soon as possible following sample receipt. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. The soil must be below the surface of the DI water in the vial. The sample is then batched up at the autosampler per the procedures listed under **section 8.4.2.1** above.

8.4.2.4 Medium level soil analysis – using field-preserved methanol sample aliquots. The customer collects approximately 5 g of soil sample into a pre-weighed 40mL vial containing 5mL methanol. At the laboratory the sample is weighed again to determine the soil weight by difference. A portion of the methanol extract is transferred into a 40 mL vial for analysis. The typical maximum methanol-water ratio is 100 µL of the methanol extract added to a total volume 5 mL sample, or 800 uL to a 40 mL vial. The prepared sample is analyzed using the aqueous sample procedure, using the water calibration to quantitate the medium level analysis.

8.4.2.5 Medium level soil analysis – if no field-preserved aliquot is submitted for analysis. Weigh 5.0-5.5g of soil sample into a 15 mL vial, then quickly add 5mL of methanol. Be sure to take the soil aliquot from below the soil surface in the sample jar to minimize headspace loss. Cap and shake for 2 minutes. After phase separation, the methanol extract is transferred into a 4mL vial with no headspace for storage. When the extract is ready for analysis, up to 100 µL of the methanol extract is added to a 5 mL aliquot of analyte free water, or 800 uL to a 40 mL vial of DI water. Use the water calibration to quantitate the medium level analysis.

8.4.3 Sample Dilution - Sample dilution is performed to ensure that all of the target analytes are determined within the instrument calibration range. Based on the concentration determined in the initial sample analysis, if needed, the analyst will determine the dilution factor required to perform the diluted analysis such that the target compounds will be determined at or above the mid-point calibration. It is important to note that due to over-saturation (column or detector), the target compounds that were determined to exceed the calibration range are usually underestimated (detected concentration lower than actual) in the initial run.

8.4.3.1 Low level aqueous sample – Dilutions for aqueous samples are prepared in an appropriate sized volumetric flask. Approximately 40mL of analyte-free water is added to the volumetric flask. The proper volume of sample is measured in a gas tight syringe. The unopened sample vial is used for the dilution. The sample is withdrawn by removing the cap, the septum seal is not punctured. The measured amount of sample is slowly injected into the volumetric flask below the surface of the analyte-free water. The flask is filled to the lip with analyte free water and closed. The diluted sample is then transferred to a 40 mL VOA vial for analysis. The amount of sample used to prepare the dilution must be noted on the logbook along with the final dilution factor to allow for double-checking of dilution calculations. Any

secondary dilution used must be clearly described in the logbook. If more space is necessary, the back of the logbook page is to be used, with a note on the front of the page to refer to the back of the page If an unopened vial is not available for dilution analysis, the situation must be discussed with the Supervisor and Project Manager prior to using a previously opened vial. If a previously opened vial is approved for use, this must be noted on the run log book and on the data review checklist submitted with the data for review to allow discussion in the project narrative.

8.4.3.2 Low level soil sample - If a smaller volume preserved soil aliquot is provided by the client, a dilution analysis may be performed. Depending on the dilution factor, sample weight down to 0.5 gram or a 10X dilution may be used. Any dilution more than 10X, using less than 0.5 gram will necessitate using the medium level methanol preserved approach below.

8.4.3.3 Medium level approach -Depending on the dilution factor, reduce the methanol extract from the ratio of 100 µL/5mL to as low as 5 µL/5mL pure water, or from 800 uL to 40 uL per 40 mL vial. Further dilution will require secondary dilution of the 10mL methanol extract. The amount of methanol used per 40mL vial must be noted on the log book along with the final dilution factor to allow for double-checking of dilution calculations. Any secondary dilution used must be clearly described in the logbook. If more space is necessary, the back of the logbook page is to be used, with a note on the front of the page to refer to the back of the page.

8.4.3.4 Criteria for reporting dilutions. The final dilution analysis is always reportable. This analysis should have the concentration of the highest compound near or above the mid range (100 ppb level) of the initial calibration.

8.4.3.5 If an initial analysis is performed that meets all QC criteria with the exception of compounds exceeding the upper calibration limit, this analysis is generally also reported. The sample ID of the initial (less dilute) analysis is unchanged and the ID of the dilution analysis has the letters "DL" appended to the sample ID. Those compounds exceeding the calibration range are qualified with the "E" flag on the data sheet for the less dilute analysis, and, if reported on CLP-type forms, qualified with a "D" if detected and reported in the more dilute (DL) analysis.

8.4.3.6 If the laboratory has prior information that a sample may contain concentrations of target or non-target compounds exceeding the

calibration range of the instrument the initial analysis may be performed at dilution. This information may include project history, prior analyses, screening results, results of other (such as GRO or TPH) analyses, solvent or petroleum odors detected during other analyses or during sampling, etc. This information should be used to prevent overloading and contamination of the autosampler/purge and trap system. If the initial analysis is performed at dilution, and the results of this analysis are acceptable (at or above the mid range calibration standard, or significant non-target compound concentrations), a less dilute analysis is not required. The sample ID is not changed by adding "DL", but the initial analysis at dilution is noted on the data review checklist included with the data submitted for review, to allow discussion in the project narrative.

8.4.3.7 If the initial analysis fails QC criteria, it is not typically reported. If only the dilution analysis is reported, the letters DL are not added to the sample ID, but the dilution is noted on the data review checklist submitted with the data for review. The letters "DL" indicate a second dilution, not an initial analysis at dilution.

8.4.3.8 If the initial and dilution analysis together demonstrate matrix interference, such as with surrogate/internal standard recoveries out of limit in both analyses, both runs are typically reported. Also if the initial analysis provides important information to the project, it should be reported, with the QC exceedences noted on the data sheets (flagged surrogates on Form 2, flagged internal standards on Form 8, "E" qualifiers on Form 1, etc) and fully described in the data review checklist included with the data submitted for review so they may be included in the project narrative.

8.4.4 Acceptance Criteria for Sample Analysis are as follows:

- the sample must meet analysis holding time.
- the sample has to have a compliant tune, initial calibration and continuing calibration.
- the sample has to have a compliant method blank.
- the sample has to have a compliant LCS.
- All surrogate recoveries are within control limits (See **section 10.4**) with the exception of one outlier, unless specified in client project. The outlier must have recovery value above 10%.
- the internal standard areas must meet the –50% to +100% criteria. If the criteria are not met, the sample should be rerun. In some circumstances (high TIC content) this is not necessary, see the corrective action table, **Attachment 8** of this SOP.

- the relative retention time (RRT) of each of the IS must not shift more than ± 0.06 RRT units from the CCV or the mid-point standard of initial calibration.
- all of the target analyte concentration should be below the calibration range excluding the "solvent" front, no ion should saturate the detector
- if the previous run contains any target analyte above the calibration range, and the same target analyte is detected above the reporting limit in the subsequent run, the subsequent run has to be repeated to demonstrate that the compound is not due to carry-over.

9. Data Reduction and Calculations

9.1 Identification of Target Compounds - Two criteria are used to identify target compounds:

9.1.1 Relative retention time (RRT) - The sample component RRT must agree within ± 0.06 RRT units of the RRT of the component in the associated continuing calibration standard. The relative retention time is determined as follows:

$$RRT = \frac{\text{Retention of target compound}}{\text{Retention time of associated internal standard}}$$

9.1.2 Comparability of mass spectra - The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative abundance greater than 25% must be present in the sample spectrum
- the relative intensities of ions specified above must agree within ± 20 % [method allows 30%] between the standard and sample spectra
- ions greater than 10.0% in the sample spectrum but not present in the standard spectrum must be considered; this may be due to potential co-eluting interference
- the halogenated target analytes should contain the characteristic chlorine and bromine isotopic ratios
- if the criteria above are not met but in the technical judgement of the analyst that the identification is correct, the lab will report the identification and proceed with the quantitation. Any suspect identification should be described on the data review checklist submitted with the data for review.

9.2 Identification of non-target compounds [tentatively identified compounds (TICs)] - Client may request the analysis of TICs. Non-target compounds will be searched using the NIST/EPA/NIH library. The non-target compound will be reported as part of the analysis requirement if:

9.2.1 The client requires a full data package deliverable, including CLP, Mitkem Level 4 or New York ASP-B reporting format (exceptions are projects that have a short list of target analytes such as TCLP, BTEX, STAR list or projects that the client specified no TIC reporting).

The non-target compounds will be identified and reported if:

- its response is greater than 10% of the closest eluting interference free internal standard
- its retention time is within the range of 30 seconds before the elution of the first target compounds, and 3 minutes after the elution of the last target compound
- Unless specified, up to **10** TIC are to be reported.

9.2.2 Guidelines for making tentative identification :

- Ions greater than 10% in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ± 20%.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in sample spectrum but not in the reference spectrum should be reviewed for co-eluting interferences.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed with caution because of background contamination and/or co-eluting interferences.
- The lab shall not report semivolatile target compounds.
- The non-target compounds will be reported as "unknown" if no valid tentative identification can be made (as based on analysts' interpretation).
- If the Quality (Qual) of the match as determined by the library search program is above 85%, it typically meets the criteria above, and is considered a tentative identification. If the Qual is less than 85%, the match typically does not meet the criteria above, and is usually identified as "unknown".

9.3 Quantitation of target compounds - The initial calibration is used to quantitate the target compounds. It is important to note that the concentrations of the target compounds not exceed the calibration range of 200 µg/L (µg/kg) for all compounds other than the m- and p-xylenes (at 400 µg/L or µg/kg) in the analyses of 5 mL water or 5 g soil samples. In the case of 25 mL water sample analyses, the concentrations for all the compounds should be less than or equal to 40 µg/L. Any target analyte concentration that exceeds the calibration range will be diluted and reanalyzed.

9.3.1 Manual integration will be performed if needed and documented according to the current revision of SOP 110.0008, Manual Integration of GC and GC/MS Chromatographs. Manual integration is appropriate when sample-specific chromatographic conditions prevent the automatic integration routines from properly assigning baseline, resulting in improper quantitation. Manual integration is prohibited from use to achieve any specific numerical QC criteria, such as to reduce surrogate peak area in order to be within recovery limits. The use of manual integration to purposefully modify non-compliant data for this reason is prohibited, and will subject the analyst to immediate disciplinary action. Any questions should be referred to the QA Director or Technical Director. Hardcopies of the ion chromatogram of the primary and secondary ion will be generated. The analyst will further initial and date the manual integration with the proper reason code per SOP 110.0008, Manual Integration of GC and GC/MS Chromatographs.

9.3.2 Determining the concentration of Target Compounds - Target compounds identified are quantitated using the following equation:

9.3.1.1 Aqueous concentrations are calculated using the equation:

$$Conc = \frac{(Ax)(Is)(V_0)}{(Ais)(RRF)(V_s)}$$

where: $Conc$ = sample concentration in µg/L
Ax = area of the characteristic ion for the compound to be measured
Ais = area of the characteristic ion of the associated internal standard
Is = concentration of internal standard in µg/L
V_0 = purge volume, 5 for 5 mL water sample and 25 for 25 mL water sample
V_s = sample volume analyzed in mL
RRF = relative response factor

9.3.1.2 Soil concentrations are calculated using the equation below:

$$Conc. = \frac{(Ax)(Is)(5)(1000)}{(Ais)(RRF)(S)(W)}$$

Medium Level:

$$Conc = \frac{(Ax)(Is)(V_t)(5)(Df)(1000)}{(Ais)(RRF)(S)(W)(V_a)}$$

where: $Conc$ = Sample concentration in µg/Kg.

Ax = area of the characteristic ion for the compound to be measured
Ais = area of the characteristic ion of the associated internal standard
Is = concentration of internal standard in µg/L
Df = dilution factor, typically is equal to 1; if there is a secondary dilution, the dilution factor refer to the dilution between the first and the secondary dilution
RRF = relative response factor
V_t = total volume of methanol extract, in mL
V_a = volume of the aliquot of the sample methanol extract, in mL
S = solid content expressed in decimal value
W = sample weight added to purge tube or for extraction, in gram

Solid sample results will be reported at dry weight basis unless otherwise specified. To convert soil results to a dry weight basis, divide the sample concentration by the percent solids (see **Section 9.3.1.3**).

9.3.1.3 Percent solid determination:

All solid sample concentrations are normalized to dry weight basis. The solid content is readily determined as follows:

- Weigh out a weighing pan (W1)
- Add about 5-10 grams of the soil sample and reweigh (W2)
- Allow the soil sample to dry overnight in an oven at 105° C; re-weigh the sample (W3) at the end of the drying period
- The solid content is determined as follows:

$$\%Solid = \frac{(W3 - W1)}{(W2 - W1)} \times 100$$

9.4 Determining the concentration of non-target compounds - An estimated concentration for non-target compounds is determined using the closest eluting internal standard. The formula to calculate the concentration is the same as those for water and soil samples described above. Total area counts from the total ion chromatograms are to be used for both the compound to be measured and the associated internal standard. A RRF of one (1) is assumed. An estimated concentration must be calculated for all tentatively identified compounds as well as these identified as unknown.

9.5 Recovery calculations - the recovery of a spiked analyte is calculated as follows:

% Recovery (%R) = 100 x (SSR-SR)/(SA)
where: SSR = spiked sample result
SR = sample concentration

SA = spike added

9.6 Relative percent difference calculations - the relative percent difference (RPD) between replicate determinations is calculated as follows:

$$RPD = \frac{(D1 - D2)}{(D1 + D2)/2} \times 100$$

where: RPD = relative percent difference
$D1$ = first sample value
$D2$ = second sample value

10. Quality Assurance/Quality Control

10.1 Personnel - Use of this method is restricted to analysts who are knowledgeable in the operation of this instrumentation and have performed a proficiency test with acceptable accuracy and precision results. All analysts must have read this SOP and asked questions and received explanation for any areas they are unsure of. This SOP should be referred to often, and used as a reference for this procedure. Details of the procedure for documenting analyst proficiency can be found in the current revision of SOP 80.0016.

10.2 Method Blanks - Method Blanks are analyzed to determine the level of contamination associated with the processing and analysis of samples.

10.2.1 Frequency of Method Blank

- the Method Blank must be analyzed after each initial calibration and during each 12-hour time period when the instrument is used for analysis.
- the Method Blank must be analyzed after the Continuing Calibration and before any samples are analyzed.

10.2.2 Procedure for Method Blank:

The Method Blank is analyzed using 5 mL of organic-free water that is spiked with 1 µL combined IS/SS (Internal Standard/Surrogate Standard) to give a final concentration of 50 µg/mL. For 25mL purge analysis, the sample is spiked with 5 µL of the IS/SS solution to yield a final concentration of 5 µg/L . Blanks are analyzed as ambient purge for aqueous/medium soil samples. For

low soil analysis, 5.0g of VOA-free Ottawa sand will be weighed into a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water and analyze by the heated purge procedure. The auto-sampler adds the IS/SS solution automatically.

10.2.3 Acceptance criteria for Method Blank:

- Percent recovery of surrogate must be within the control limits listed in **Section 10.4.**

- All internal standard response must be within the –50% to +100% criteria. If the criteria are not met, the blank should be rerun.

- The concentration of each target compound found in the Method Blank must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10.0 µg/L or µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1.00 µg/L, and acetone and 2-butanone must be less than 10.0 µg/L).

For *DoD* projects, the concentration of the target compounds in the method blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

Any Method Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze the blank. There may be situations where other corrective actions are appropriate depending on project-specific criteria, such as when the sample analysis resulted in a non-detect for the compound that failed the blank acceptance criteria.

10.2.4 All compounds present in method blanks that are also present in samples will be qualified with a "B" flag on data sheets reported to the client. The meaning of this qualifier will be described in the report. This will also be noted on the data review checklist when the data are submitted for review to allow for discussion in the narrative.

10.3 Storage Blanks- Storage Blanks are analyzed to determine the level of contamination associated with the storage of samples. They are analyzed as a sample at the end of the analytical sequence.

10.3.1 Acceptance criteria for Storage Blanks:

- The concentration of each target compound found in the Storage Blanks must be less than its reporting limit except for certain common laboratory contaminants which have expanded acceptance criteria. In the case of 5 mL water/5 g soil sample analyses, common contaminants must be less than- 2 times their PQL (methylene chloride, acetone and 2-butanone must be less than 10.0 µg/L or µg/Kg); in the case of 25 mL water sample analyses, common contaminants must be less than 2 times their PQL (methylene chloride must be less than 1.00 µg/L, and acetone and 2-butanone must be less than 10.0 µg/L).

For *DoD* projects, the concentration of the target compounds in the Storage Blank must be less than one-half of the Method Reporting Limit; the concentration for common laboratory contaminants such as methylene chloride and ketones, must not exceed the Method Reporting Limit.

- Any Storage Blank that fails to meet any of the above criteria must receive corrective action. First investigate the ISS integrations and subsequent quantitation of the analytes in question to verify concentration. Check calculations. If the analysis is valid, a common corrective action is to reanalyze another Storage Blank. If the reanalysis confirms the contamination, the situacion must be investigated and the affected client(s) must be notified of the potential contamination issue in the applicable refrigerator.

10.4 Laboratory Control Sample (LCS) -One LCS is prepared with each batch of up to 20 samples of the same matrix. The LCS is spiked with all compounds being reported for the method. If a non-routine compound is being reported, but no LCS is available, this must be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

- For an aqueous LCS sample, mixed standards are spiked into a 40 mL vial of organic-free water, resulting in concentrations at the mid-level standard. See **Table 2** for details on spiking volumes and solutions.

- For a solid LCS sample, add 5.0g of VOA-free Ottawa sand to a 40ml VOA vial. (This information should be written in the VOA soil extraction logbook). Add 5ml of organic-free water to the vial. Then add the commercially prepared standards with known values of VOC concentrations by spiking standards into the vial and

analyzing by the heated purge procedure. Where applicable, a Lab Control Sample Duplicate (LCSD) will also be performed to evaluate reproducibility.

10.4.1 Acceptance criteria for LCS:

- Percent recovery of surrogates must be within the control limits listed in **Section 10.4 .** For regular SW8260 projects, the recovery is evaluated against the established in-house limits. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits. See **Attachment 4** for *DoD* QC limits.
- All internal standard response must be within the –50% to +100% criteria. If the criteria are not met, the LCS should be rerun.
- If target analytes are outside of the acceptance limits, corrective action is required. Project-specific requirements, if available, will dictate the corrective action performed. See **Attachment 8** for further guidance.
- Due to the large number of target analytes, some recoveries (up to 5 for full list) may be out.

Per *DoD* requirements, analyses of <11 analytes, no marginal exceedences (ME) are allowed. For the analysis of 11-30 analytes, one ME is allowed; for the analysis of 31-50 analytes, two ME are allowed; for 51-70 analytes, (typical 8260 analysis) three ME are allowed; for the analysis of 71-90 analytes, four ME are allowed; for the analysis of >90 analytes, five ME are allowed. See **Attachments 5 and 6** for further guidance.

- Reporting LCS Results – If any compounds are outside of the acceptance limits, their recoveries are qualified with the "*" flag on the LCS recovery summary report (Form 3) for CLP-type data reports, and flagged with an "S" on Level 2 LIMS type data reports. This information is noted on the data review checklist submitted with the data for review, to allow for inclusion in the project narrative.

10.5 Surrogate recoveries - The recovery of the surrogate compounds (also called System Monitoring Compounds) in all samples, blanks and LCS will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

10.5.1 Acceptance criteria - The percent recovery of each of the surrogate compounds in blanks, samples, duplicate matrix spikes and LCS must be within the in-house acceptance window with the exception of one surrogate per fraction allowed out. Refer to the LIMS Test Information category/Test option/ specs for the most current QC control limits.

- For *DoD* projects, values from QSM:

	Solid	Aqueous
1,2-dichloroethane-d4	no limits given	70-120
dibromofluoromethane	no limits given	85-115
toluene-d8	85-115	85-120
4-bromofluorobenzene	85-120	75-120

10.5.2 Corrective action - If the recovery of the system monitoring compound is out of the acceptable window in the method blank or LCS, corrective action must be implemented. Corrective action should include verification of the Internal standard area integrations, checking for errors in calculations and confirming the use of appropriate standards. In addition, the blank and/or LCS may be re-analyzed. If the recovery of the system-monitoring compound is outside of the acceptance limit for a sample, the data will be evaluated and corrective action (commonly reanalysis) will be taken. See **Attachment 8** for corrective action guidelines.

10.5.3 Reporting – The Target data reduction and reporting programs will flag any surrogate recovery outside of the acceptance limits with a "*"; the LIMS Level 2 reporting will flag any surrogate recovery outside of the acceptance limits with a "S". If the sample is reanalyzed and the system monitoring compounds are within the acceptance criteria for the reanalysis, and the reanalysis is within holding time, report the results of the reanalysis only. If the same system monitoring compounds are out in the reanalysis, report both sets of analysis results to demonstrate matrix-related problems. This should be noted on the data review checklist submitted with the data for review for inclusion in the project narrative.

10.6 Matrix spike/matrix spike duplicate samples (MS/MSD) are analyzed at a frequency of once per twenty samples of similar matrix and procedure. The duplicate matrix spikes are used to assess the effect of matrix on the analytical accuracy and precision for the batch of samples. Where the client has not provided sufficient sample aliquots for a MS/MSD to be included in every batch, a duplicate LCS should be performed so analytical precision can be demonstrated. The duplicate matrix spikes are typically spiked with all of the target analytes. There may be project-specific MS spiking lists and criteria which take precedence.

10.6.1 The percent recovery of each compound is compared to the in-house acceptance limits, project-specific limits or **Attachment 6**. These limits are the same as used for LCS, but are used as advisory guidelines.

10.6.2 The following factors could greatly affect the accuracy and precision of the matrix spikes and matrix spike duplicates: sample heterogeneity, much higher analyte concentration in the sample, and matrix effect. The best measurement is obtained if the spike concentration is two to four times the analyte concentration in the unspiked sample.

10.6.3 If target compound recoveries are outside of the MS acceptance limits corrective action is required. See **Attachment 8** of this SOP for corrective action guidelines. Evaluate the percent recovery for those compounds outside of the recovery limit to the same compound in the LCS. At a minimum the corrective action will involve flagging any MS value outside of the control limit with an "*" on the recovery summary report form (Form 3) or an "S" on the LIMS Level 2 report. This is also noted on the data review checklist submitted with the data for review to allow for inclusion in the report narrative. Other corrective actions may include reanalysis of the MS/MSD at a higher spiking concentration, reanalysis of the MS/MSD by dilution of the sample, discussion of the issue with the Project Manager and the client.

For *DoD* projects, the %RPD limits for the duplicate set is 30%.

11. Data Validation and Reporting

All raw data, including calibrations, QC results, and samples results, are reviewed for technical accuracy and completeness. The guidelines and procedures taken to ensure the data quality are listed in Section 11 of Quality Assurance Plan.

12. Corrective Action Procedures

12.1 All QC excedences require a corrective action response and documentation. The proper corrective action depends on the specific situation. Many actions are spelled-out in this SOP. A table describing common occurrences, corrective actions and documentation is attached as **Attachment 8**.

12.2 Further information on Mitkem's corrective action policy and procedures are included in the current revision of SOP No. 80.0007.

13. Health and Safety

13.1 The toxicity or carcinogenicity of each reagent used in the method has not been fully established. However, each chemical should be regarded as a potential health hazard, and exposure to these compounds should be as low as reasonably achievable. A reference file of material safety data sheets (MSDS) is available to all laboratory personnel. MSDS sheets were kept in safety officer's recover. In addition, laboratory

personnel should follow the precautions outlined in the laboratory's Chemical Hygiene Plan. In general, use gloves, a lab coat, and safety glasses when handling these reagents and work in a hood whenever possible.

13.2 Basic good housekeeping practices, such as the wiping up of spills immediately and regular cleaning of counters and hoods, will help reduce the potential for cross-contamination and create a safe working environment.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

1. Environmental Protection Agency. Gas Chromatography/Mass Spectrometry Method 8260C, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 3 August 2006.

2. Mitkem Laboratories Quality Assurance Plan (QAP), current revision.

3. "Quality Systems Manual for Environmental Laboratories" Department of Defense, Final Version 3, January, 2006.

4. Environmental Protection Agency. Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples Method 5035, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 0, December 1996.

5. Environmental Protection Agency. Purge-and-Trap for Aqueous Samples Method 5030, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, Revision 2, December 1996.

6. Hewlett Packard and OI Analytical Instrument instruction manuals.

7. ASTM D6418 - 04 Standard Practice for Using the Disposable EnCore Sampler for Sampling and Storing Soil for Volatile Organic Analysis.

16. Low Level Calibration and Analyis

1. A low level calibration procedure is used to achieve a low level reporting limit at 0.5 ug/L for most of the target analytes, except for ketones. The purge volume for all standards, QC samples and field samples are 25 mL instead of 5 mL. The 5 level calibration standards are 0.5, 4.0, 10, 20 and 40 ug/L, except for ketone compounds are 5.0, 40, 100, 200 and 400 ug/L.

2. Internal Standard and Surrogate Standard Mix solution (IS/SS): The working standard of IS/SS solution is prepared by transferring 200 uL each of the IS stock solution (Cat. No. 30241)and SS stock solution (Cat. No. 30240) into a 4 mL vial with 3600 uL of methanol to make a 125 ug/mL solution. This solution once prepared is stored in the Standard Adding Module of either 4551A or 4552 autosamplers. 1uL of the solution is added to all Calibration Standards, ICV, blanks, LCS and samples. At a 25 mL purge volume, this yields a concentration of 5 ug/L.

3. The working standard for the calibration standards is prepared by diluting the 5 mL analysis calibration standard 5 times to a concentration of 20 ug/mL. Additional ketone standards are added at this step to result in a concentration of 100 ug/mL for the ketone compounds.

4. The 5 level initial calibration standards are prepared by adding 1, 8, 20, 40 and 80 uL of the calibration standard, 1, 8, 20, 40 and 80 uL of the surrogate standard and 10 uL of the internal standard to each 40 mL DI water. 25 mL of each solution are used for calibration analysis. Initial calibration criteria are the same as the 5 mL analysis.

5. A second source Initial Calibration Verification (ICV) is performed after the completion of the multi-level calibration, at 10ug/L. The calculated value of the analytes in the ICV should be 70 – 130% of the expected value (7.0-13.0 ng/uL). DoD limits are 75-125%.

6. The continuing calibration standard is prepared by adding 20 uL of the calibration standard, 20 uL of the surrogate standard and 10 uL of the internal standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency and criteria of continuing calibration are the same as the 5 mL analysis.

7. Method blank is prepared by adding 1uL of the IS/SS standard to a 25 mL DI water. The frequency and criteria of method blank are the same as 5 mL analysis. No target compound can be detected above one half of the required reporting limits, except for Methylene Chloride which must be less than 2 ug/L.

8. LCS is prepared by adding 20 uL of the calibration standard to a 40 mL DI water. 25 mL of this solution is used for analysis. The frequency and criteria of LCS are the same as 5 mL analysis.

9. Samples are analyzed after all calibration and QC samples have been analyzed and passed their criteria. Each sample is spiked with 1uL of the IS/SS standard by the autosampler. 25 mL of each spiked sample is used for analysis. The criteria for sample analysis are the same as the 5 mL analysis.

10. MS/MSD samples are spiked with 20 uL of the calibration standard. 25 mL of each spiked sample is used for analysis and is spiked with 1uL of the IS/SS standard by the autosampler. The frequency and criteria of MS/MSD samples are the same as the 5 mL analysis. However, since the whole sample vial is spiked and used for each analysis, MS/MSD for 25

mL analysis can only be performed when there are three or more sample vials available for the designated sample.

Attachments:

Table 1: List of Acronyms
Table 2: Working Standard / LCS Detail.
Table 3: Suggested minimum RFs (Table 4 from Published Method).
Figure 1: Primary Standard Logbook.
Figure 2: Working Standard Logbook.
Figure 3: Instrument Run Logbook.
Attachment 1: SW8260 Target Analyte List
Attachment 2: BFB Tune Chromatogram and Mass Listing.
Attachment 3: BFB Tune Mass Spectrum and Ion Abundance Criteria.
Attachment 4: Chromatograph and Quantitation Report of 50ug/L Standard.
Attachment 5: DoD Specific QC Requirements: DoD-B SW846 box, Table B-3.
Attachment 6: DoD Specific QC Control Limits, Tables D-4, D-5.
Attachment 7: Additional QA/QC Requirements for MA_DEP
Attachment 8: Corrective Action and Documentation Examples.

Table 1
List of Acronyms

BFB	Bromofluorobenzene
DoD	Department of Defense (including Army, Navy, Air Force)
LCS	Lab control sample
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method detection limit
MQL	Method quantitation limit
ME	Marginal Exceedence
MS	Matrix spike
MSD	Matrix spike duplicate
QSM	Quality Systems Manual for DoD work
RL	Reporting Limit (occasionally referred to as PQL or Practical Quantitation Limit {in the LIMS}, or MRL or Method Reporting Limit)

Table 2

Working Standard / LCS Detail

8260

Aqueous uL spike amounts for 40mL Vials											
	QC				Sample	ICAL (Concentration)					
	CC	Blank	LCS	MS/MSD		5	20	50	100	200	1
GAS	20	-	20	20	-	2	8	20	40	80	0.4
STD	20	-	20	20	-	2	8	20	40	80	0.4
APPIX	20	-	20	20	-	2	8	20	40	80	0.4
SS	M.I.	M.I.	M.I.	M.I.	M.I.	2	8	20	40	80	0.4
IS	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.	M.I.

* M.I. Machine Injection: equal to 20 uL per 40 mL (if hand spiked)

Soil uL spike amounts for 5mL H_2O										
	QC				Sample	ICAL (Concentration)				
	CC	Blank	LCS	MS/MSD		5	20	50	100	200
GAS	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
STD	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
APPIX	2.5	-	2.5	2.5	-	0.25	1	2.5	5	10
SS	2.5	2.5	2.5	2.5	2.5	0.25	1	2.5	5	10
IS	2.5	2.5	2.5	2.5	2.5	M.I.	2.5	2.5	2.5	2.5

* Vials should contain approx. 5g VOC-free soil preserved in 5mL D.I. Water

* For QC samples use an additional 5mL H_2O in 40 mL vial

Table 3
Suggested minimum RFs
(Table 4 from Published Method)

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

[a] The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

[b] Data provided by EPA Region III laboratory.

Figure 1

Primary Standard Logbook

Mitkem Laboratories

VOA Primary Standard Receipt Logbook

Date	Description, Vendor Name	Catalog #, Lot #	Concentration	Mitkem ID	*Exp. Date	**Date Opened	Analyst

* Expiration dates are 2 years for unopened stock solutions and 5 years for neat materials, unless noted otherwise by manufacturer.

** Expiration dates for opened stock solutions are 6 months.

Logbook ID 90.0151-01/07

Reviewed by:_____

Figure 2

Working Standard Logbook

Mitkem Laboratories, A Division of Spectrum Analytical, Inc.

VOA Working Standard Logbook

DATE	DESCRIPTION	STOCK ID	STOCK CONC	INITIAL VOL	FINAL VOL	METHANOL LOT #	FINAL CONC	WORKING STD ID	EXP DATE	Analyst

Logbook ID 90.0152-10/08

Reviewed by : _____

Figure 3

Instrument Run Logbook

Instrument V 7
Injection Log

Mitkem Laboratories
Volatiles Laboratory

METHOD:_____ CAL ID:_____ ANALYST:_____

INITIAL CAL:_____ IS/SS ID:_____ DATE:_____

COMMENTS:

DATE PRINTED:_____

DATE LOADED:_____

AS #	FILE		MITKEM ID		CLIENT ID	SAMPLE SIZE	DIL	COMMENTS	IS	SS	pH

Logbook ID 90.0220-06/08

Attachment 1

Target Analyte List for 8260

Method 8260 Target Analyte List

type	Target Analyte	type	Target Analyte
A	1,1,1,2-Tetrachloroethane	I	1,4-Dichlorobenzene-d4
A	1,1,1-Trichloroethane	I	Chlorobenzene-d5
A	1,1,2,2-Tetrachloroethane	I	Fluorobenzene
A	1,1,2-Trichloroethane	S	1,2-Dichloroethane-d4
A	1,1-Dichloroethane	S	Bromofluorobenzene
A	1,1-Dichloroethene	S	Dibromofluoromethane
A	1,1-Dichloropropene	S	Toluene-d8
A	1,2,3-Trichlorobenzene		
A	1,2,3-Trichloropropane	X	1,1,2,2-Tetrachloroethane-d2
A	1,2,4-Trichlorobenzene	X	1,1,2-Trichloro-1,2,2-trifluoroethane
A	1,2,4-Trimethylbenzene	X	1,1-Dichloroethene-d2
A	1,2-Dibromo-3-chloropropane	X	1,2-Dichlorobenzene-d4
A	1,2-Dibromoethane	X	1,2-Dichloroethene, Total
A	1,2-Dichlorobenzene	X	1,2-Dichloropropane-d6
A	1,2-Dichloroethane	X	1,2-Dichlorotetrafluoroethane
A	1,2-Dichloropropane	X	1,3,5-Trichlorobenzene
A	1,3,5-Trimethylbenzene	X	1,4-Difluorobenzene
A	1,3-Dichlorobenzene	X	1,4-Dioxane
A	1,3-Dichloropropane	X	1-Chlorohexane
A	1,4-Dichlorobenzene	X	2-Butanone-d5
A	2,2-Dichloropropane	X	2-Chloro-1,3-butadiene
A	2-Butanone	X	2-Chloroethyl vinyl ether
A	2-Chlorotoluene	X	2-Ethyl-1-hexanol
A	2-Hexanone	X	2-Hexanone-d5
A	4-Chlorotoluene	X	2-methyl-2-propanol
A	4-Isopropyltoluene	X	Acetonitrile
A	4-Methyl-2-pentanone	X	Acrolein
A	Acetone	X	Acrylonitrile
A	Benzene	X	Alkylbenzenes, Total
A	Bromobenzene	X	Allyl chloride
A	Bromochloromethane	X	Benzene-d6
A	Bromodichloromethane	X	bis(2-Chloroethyl)ether
A	Bromoform	X	Bromoform-d
A	Bromomethane	X	Chloroethane-d5
A	Carbon disulfide	X	Chloroform-d
A	Carbon tetrachloride	X	Cyclohexane
A	Chlorobenzene	X	Diethyl ether
A	Chloroethane	X	Diisopropyl ether
A	Chloroform	X	Ethanol
A	Chloromethane	X	Ethyl methacrylate
A	cis-1,2-Dichloroethene	X	Ethyl tert-butyl ether
A	cis-1,3-Dichloropropene	X	Freon-113
A	Dibromochloromethane	X	Hexachloroethane
A	Dibromomethane	X	Isobutyl alcohol
A	Dichlorodifluoromethane	X	Isopropyl alcohol
A	Ethylbenzene	X	Methacrylonitrile

A	Hexachlorobutadiene	X	Methyl acetate
A	Iodomethane	X	Methyl methacrylate
A	Isopropylbenzene	X	Methylcyclohexane
A	m,p-Xylene	X	Pentachloroethane
A	Methyl tert-butyl ether	X	Pentafluorobenzene
A	Methylene chloride	X	Propionitrile
A	n-Butylbenzene	X	tert-Amyl methyl ether
A	n-Propylbenzene	X	Tetrahydrofuran
A	Naphthalene	X	trans-1,3-Dichloropropene-d4
A	o-Xylene	X	trans-1,4-Dichloro-2-butene
A	sec-Butylbenzene	X	Vinyl chloride-d3
A	Styrene	X	Xylenes (Total)
A	tert-Butylbenzene		
A	Tetrachloroethene		
A	Toluene		
A	trans-1,2-Dichloroethene		
A	trans-1,3-Dichloropropene		
A	Trichloroethene		
A	Trichlorofluoromethane		
A	Vinyl acetate		
A	Vinyl chloride		

type	explanation
A	Analyte (routine)
I	Internal Standard
S	Surrogate
X	Analyte (non-routine)

Attachment 2

BFB Tune Chromatogram

Data File: \\AVOGADRO\ORGANICS\organic\voa\V6.i\060201A.B\V6E0620.D

Date : 01-FEB-2006 12:27

Client ID: BFB6Z

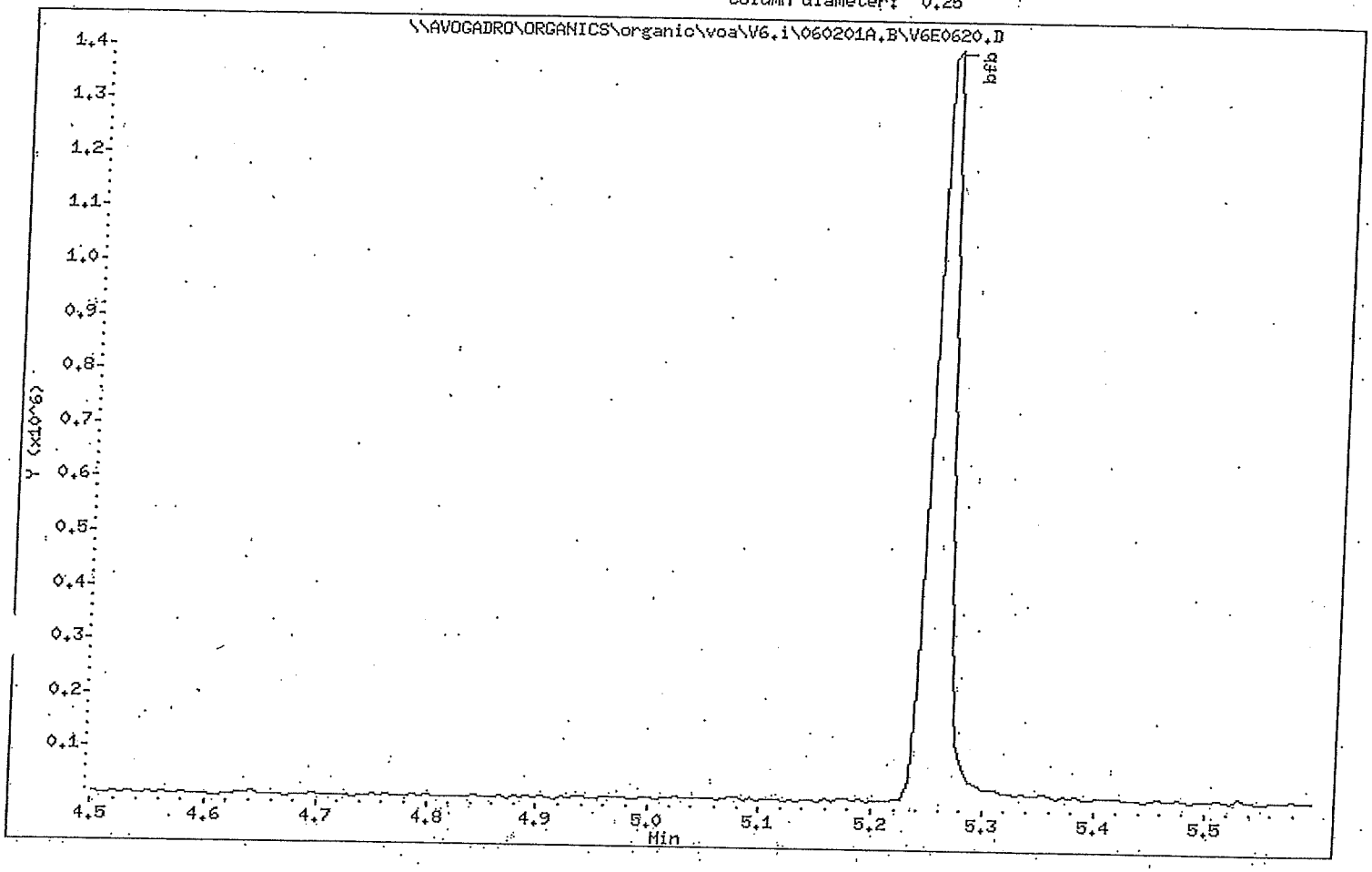
Instrument: v6.i

Sample Info: 2ul,BFB6Z,BFB6Z,

Operator: LG

Column phase: DB-624

Column diameter: 0.25



Attachment 3

BFB Tune Mass Spectrum and Ion Abundance Criteria

Data File: \\AVOGADRO\ORGANICS\organic\voa\V6.i\060201A.B\V6E0620.D

Date : 01-FEB-2006 12:27

Client ID: BFB6Z

Instrument: v6.i

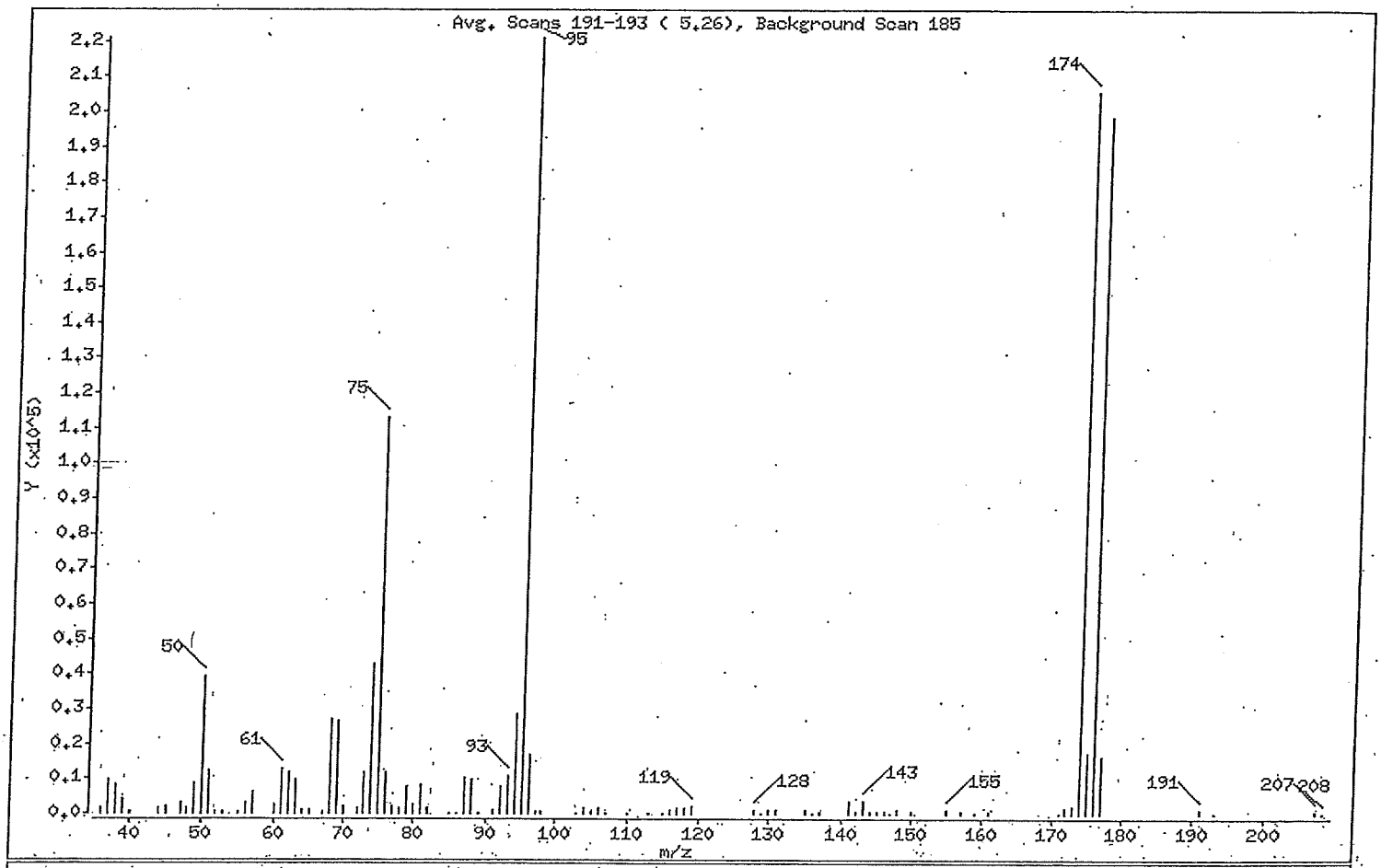
Sample Info: 2ul,BFB6Z,BFB6Z,

Operator: LG

Column phase: DB-624

Column diameter: 0.25

1 bfb



m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
95	Base Peak, 100% relative abundance	100.00
50	15.00 - 40.00% of mass 95	17.82
75	30.00 - 60.00% of mass 95	51.26
96	5.00 - 9.00% of mass 95	7.72
173	Less than 2.00% of mass 174	1.03 (1.11)
174	50.00 - 100.00% of mass 95	93.19
175	5.00 - 9.00% of mass 174	7.74 (8.30)
176	95.00 - 101.00% of mass 174	89.94 (96.52)
177	5.00 - 9.00% of mass 176	7.42 (8.25)

Data File: \\AVOGADRO\ORGANICS\organic\voa\V6.i\060201A.B\V6E0620.D

Date : 01-FEB-2006 12:27

Client ID: BFB6Z

Instrument: v6.i

Sample Info: 2ul,BFB6Z,BFB6Z,

Operator: LG

Column phase: DB-624

Column diameter: 0.25

Data File: V6E0620.D

Spectrum: Avg. Scans 191-193 (5.26), Background Scan 185

Location of Maximum: 95.00

Number of points: 94

m/z	Y	m/z	Y	m/z	Y	m/z	Y
36.00	1702	68.00	26928	96.00	17040	143.00	3728
37.00	9822	69.00	26472	97.00	644	144.00	398
38.00	7957	70.00	1977	98.00	258	145.00	624
39.00	4095	72.00	1384	103.00	202	146.00	379
40.00	472	73.00	11595	104.00	1583	147.00	189
44.00	1567	74.00	42872	105.00	398	148.00	812
45.00	2280	75.00	113192	106.00	1441	150.00	287
47.00	2965	76.00	11922	107.00	172	155.00	936
48.00	1681	77.00	1906	110.00	236	157.00	479
49.00	8514	78.00	1301	113.00	169	159.00	119
50.00	39344	79.00	7593	115.00	227	161.00	288
51.00	12361	80.00	2364	116.00	1139	171.00	174
52.00	506	81.00	8306	117.00	1693	172.00	1756
53.00	289	82.00	1472	118.00	1309	173.00	2279
55.00	538	85.00	167	119.00	2109	174.00	205760
56.00	3101	86.00	243	128.00	1110	175.00	17088
57.00	6291	87.00	10388	129.00	191	176.00	198592
60.00	2730	88.00	9715	130.00	1050	177.00	16375
61.00	12548	89.00	186	131.00	824	191.00	1084
62.00	11630	91.00	1240	135.00	961	193.00	212
63.00	9920	92.00	7868	136.00	170	207.00	648
64.00	1146	93.00	10870	137.00	701	208.00	107
65.00	1241	94.00	28568	141.00	3681		
67.00	497	95.00	220800	142.00	445		

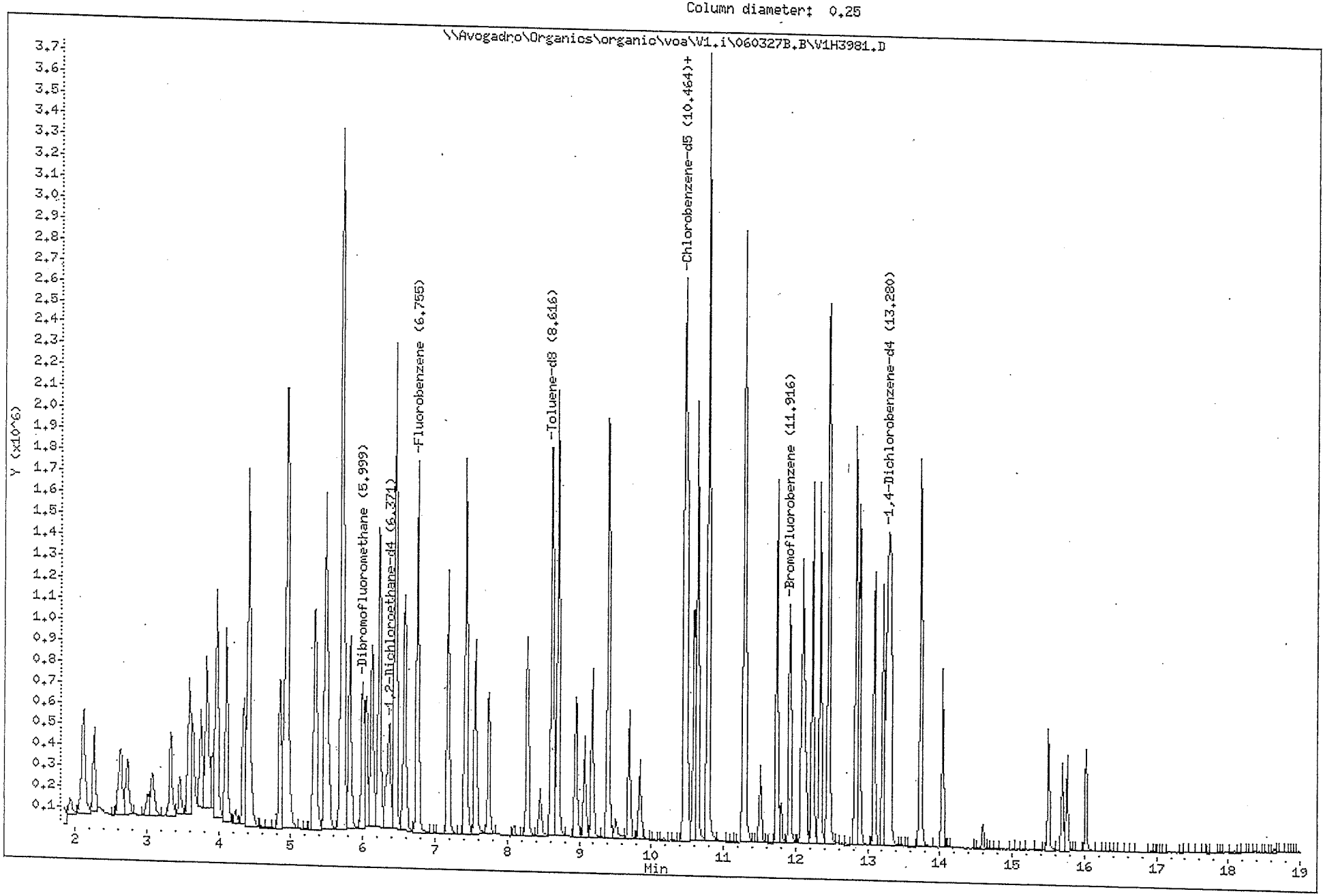
Attachment 4

Chromatogram and Quantitation Report of 50 μg/L Standard

Data Fi\ \\Avogadro\Organics\organic\voa\V1.i\060327B.B\V1H3981.D
Date : AR-2006 12:36
Client ID: VSTD0501E
Sample Info: 5ml,VSTD0501E,VSTD0501E,
Purge Volume: 5.0
Column phase: DB-624

Instrument: V1.i

Operator: LG SRC: LG
Column diameter: 0.25



Data File: V1H3981.D
Report Date: 07-Apr-2006 16:21

Mitkem Corporation

Method 8260 Water and Medium Soil

Data file : \\Avogadro\Organics\organic\voa\V1.i\060327B.B\V1H3981.D
Lab Smp Id: VSTD0501E
Client Smp ID: VSTD0501E
Inj Date : 27-MAR-2006 12:36
Operator : LG SRC: LG
Inst ID: V1.i
Smp Info : 5ml,VSTD0501E,VSTD0501E,
Misc Info : ,2
Comment :
Method : \\Avogadro\Organics\organic\voa\V1.i\060327B.B\v18260.m
Meth Date : 07-Apr-2006 16:18 V1.i
Quant Type: ISTD
Cal Date : 25-MAR-2006 15:39
Cal File: V1H3935.D
Als bottle: 1
Continuing Calibration Sample
Dil Factor: 1.00000
Integrator: HP RTE
Compound Sublist: all.sub
Target Version: 4.14

Concentration Formula: Amt * DF * Uf * 5/Vo * CpndVariable

Name	Value	Description
DF	1.000	Dilution Factor
Uf	1.000	ng unit correction factor
Vo	5.000	Sample Volume purged (mL)
Cpnd Variable		Local Compound Variable

Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
1 Dichlorodifluoromethane	85	1.929	1.929	(0.286)	278789	50.0000	32
116 Freon114	85	2.091	2.091	(0.310)	335530	50.0000	27
2 Chloromethane	50	2.116	2.116	(0.313)	764768	50.0000	46
3 Vinyl Chloride	62	2.264	2.264	(0.335)	661339	50.0000	47
4 Bromomethane	94	2.624	2.624	(0.389)	482722	50.0000	48
5 Chloroethane	64	2.723	2.723	(0.403)	397638	50.0000	48
6 Trichlorofluoromethane	101	3.083	3.083	(0.456)	308677	50.0000	41
119 Ether	59	3.331	3.331	(0.493)	341064	50.0000	43
7 Acrolein	56	3.455	3.455	(0.512)	270949	250.000	150
8 1,1-Dichloroethene	96	3.592	3.592	(0.532)	429394	50.0000	45
9 Acetone	43	3.629	3.629	(0.537)	77539	50.0000	25
111 1,1,2-Trichloro-1,2,2-Trifluo	101	3.641	3.641	(0.539)	296467	50.0000	33
10 Iodomethane	142	3.753	3.753	(0.556)	962097	50.0000	48
11 Carbon Disulfide	76	3.827	3.827	(0.567)	1988926	50.0000	47
12 Acetonitrile	41	3.964	3.964	(0.587)	1231593	500.000	440
13 Allyl Chloride	39	3.964	3.964	(0.587)	616964	50.0000	46
112 Methyl Acetate	43	3.989	3.989	(0.591)	317451	50.0000	38
14 Methylene Chloride	84	4.100	4.100	(0.607)	622555	50.0000	46
120 tert-Butanol	59	4.237	4.237	(0.627)	143552	100.000	82
15 Acrylonitrile	53	4.348	4.348	(0.644)	705347	250.000	180
16 trans-1,2-Dichloroethene	96	4.410	4.410	(0.653)	574717	50.0000	49
17 Methyl tert-butyl ether	73	4.423	4.423	(0.655)	1224188	50.0000	44
18 1,1-Dichloroethane	63	4.844	4.844	(0.717)	972183	50.0000	50

Data File: V1H3981.D
Report Date: 07-Apr-2006 16:21

	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
	19 Vinyl acetate	43	4.907	4.907	(0.726)	1659795	50.0000	42
	121 Diisopropyl Ether	45	4.944	4.944	(0.732)	2148432	50.0000	47
	20 2-Chloro-1,3-Butadiene	53	4.956	4.956	(0.734)	682414	50.0000	50
	122 Ethyl tert-butyl ether	59	5.341	5.341	(0.791)	1510068	50.0000	46
	23 cis-1,2-Dichloroethene	96	5.477	5.477	(0.811)	597046	50.0000	48
	22 2-Butanone	43	5.477	5.477	(0.811)	155230	50.0000	32
	24 2,2-Dichloropropane	77	5.490	5.490	(0.813)	631972	50.0000	51
	25 Propionitrile	54	5.527	5.527	(0.818)	446693	500.000	360
	26 Methacrylonitrile	41	5.490	5.490	(0.813)	345627	500.000	540
	27 Bromochloromethane	128	5.738	5.738	(0.849)	299396	50.0000	45
	29 Tetrahydrofuran	72	5.800	5.800	(0.859)	45393	50.0000	38
	28 Chloroform	83	5.824	5.824	(0.862)	886677	50.0000	50
$	30 Dibromofluoromethane	113	5.998	5.998	(0.888)	573068	50.0000	49
	31 1,1,1-Trichloroethane	97	6.048	6.048	(0.895)	603321	50.0000	51
	113 Cyclohexane	56	6.122	6.122	(0.906)	650531	50.0000	37
	32 1,1-Dichloropropene	110	6.221	6.221	(0.921)	251305	50.0000	48
	33 Carbon Tetrachloride	117	6.234	6.234	(0.923)	504076	50.0000	50
	34 Isobutyl Alcohol	43	6.321	6.321	(0.936)	261625	1000.00	810
$	35 1,2-Dichloroethane-d4	102	6.370	6.370	(0.943)	116416	50.0000	42
	37 Benzene	78	6.445	6.445	(0.954)	2213191	50.0000	49
	36 1,2-Dichloroethane	62	6.445	6.445	(0.954)	441092	50.0000	47
	123 tert-Amyl methyl ether	73	6.581	6.581	(0.974)	1327050	50.0000	44
M	21 1,2-Dichloroethene (Total)	96				1171763	100.000	97
*	38 Fluorobenzene	96	6.755	6.755	(1.000)	2047434	50.0000	
	39 Trichloroethene	130	7.177	7.177	(1.062)	640273	50.0000	51
	114 Methylcyclohexane	83	7.425	7.425	(1.099)	425254	50.0000	33
	40 1,2-Dichloropropane	63	7.425	7.425	(1.099)	536317	50.0000	48
	41 Methyl Methacrylate	69	7.549	7.549	(1.118)	235457	50.0000	39
	42 Dibromomethane	93	7.561	7.561	(1.119)	282273	50.0000	42
	124 1,4-Dioxane	88	7.586	7.586	(1.123)	61146	1000.00	760
	43 Bromodichloromethane	83	7.747	7.747	(1.147)	586154	50.0000	47
	44 2-Chloroethyl vinyl ether	63	8.107	8.107	(1.200)	31453	50.0000	33
	45 cis-1,3-Dichloropropene	75	8.281	8.281	(1.226)	741784	50.0000	45
	46 4-Methyl-2-pentanone	43	8.454	8.454	(1.252)	268128	50.0000	36
$	47 Toluene-d8	98	8.628	8.628	(0.826)	1692964	50.0000	50
	48 Toluene	91	8.702	8.702	(1.288)	1988522	50.0000	48
	49 trans-1,3-Dichloropropene	75	8.950	8.950	(1.325)	522490	50.0000	43
	50 Ethyl Methacrylate	69	9.074	9.074	(1.343)	375280	50.0000	38
	51 1,1,2-Trichloroethane	97	9.186	9.186	(1.360)	343947	50.0000	42
	52 1,3-Dichloropropane	76	9.397	9.397	(0.899)	619425	50.0000	45
	53 Tetrachloroethene	164	9.409	9.409	(0.900)	432301	50.0000	57
	54 2-Hexanone	43	9.509	9.509	(0.910)	124012	50.0000	35
	55 Dibromochloromethane	129	9.695	9.695	(0.928)	454186	50.0000	45
	56 1,2-Dibromoethane	107	9.843	9.843	(0.942)	353159	50.0000	42
*	57 Chlorobenzene-d5	117	10.451	10.451	(1.000)	1302062	50.0000	
	115 1-Chlorohexane	91	10.464	10.464	(1.001)	476509	50.0000	44
	58 Chlorobenzene	112	10.489	10.489	(1.004)	1236847	50.0000	50
	59 1,1,1,2-Tetrachloroethane	131	10.600	10.600	(1.014)	446477	50.0000	48
	60 Ethylbenzene	106	10.637	10.637	(1.018)	576740	50.0000	50
	61 m,p-Xylene	106	10.786	10.786	(1.032)	1413854	100.000	98
	62 o-Xylene	106	11.282	11.282	(1.080)	701247	50.0000	48
	64 Styrene	104	11.295	11.295	(1.081)	1037062	50.0000	45
	65 Bromoform	173	11.518	11.518	(1.102)	230524	50.0000	42
	66 Isopropylbenzene	105	11.741	11.741	(1.123)	1436233	50.0000	49

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	Compounds	QUANT SIG MASS	RT	EXP RT	REL RT	RESPONSE	AMOUNTS CAL-AMT (ug/L)	AMOUNTS ON-COL (ug/L)
$	68 Bromofluorobenzene	95	11.915	11.915	(1.140)	530593	50.0000	46
	69 1,1,2,2-Tetrachloroethane	83	12.076	12.076	(0.909)	358654	50.0000	40
	70 Bromobenzene	156	12.101	12.101	(0.911)	417102	50.0000	50
	67 trans-1,4-Dichloro-2-butene	75	12.138	12.138	(1.161)	368024	50.0000	39
	71 1,2,3-Trichloropropane	75	12.138	12.138	(0.914)	368024	50.0000	43
	72 n-Propylbenzene	120	12.238	12.238	(0.922)	373615	50.0000	51
	73 2-Chlorotoluene	126	12.337	12.337	(0.929)	430653	50.0000	54
	74 1,3,5-Trimethylbenzene	105	12.448	12.448	(0.937)	1164756	50.0000	52
	75 4-Chlorotoluene	126	12.461	12.461	(0.938)	398201	50.0000	52
M	63 Xylene (Total)	106				2115101	150.000	140
	76 tert-Butylbenzene	119	12.833	12.833	(0.966)	1128259	50.0000	49
	77 1,2,4-Trimethylbenzene	105	12.883	12.883	(0.970)	1089852	50.0000	50
	78 sec-Butylbenzene	105	13.093	13.093	(0.986)	1189318	50.0000	49
	80 1,3-Dichlorobenzene	146	13.217	13.217	(0.995)	701992	50.0000	49
	79 4-Isopropyltoluene	119	13.255	13.255	(0.998)	902361	50.0000	47
*	81 1,4-Dichlorobenzene-d4	152	13.279	13.279	(1.000)	424865	50.0000	
	82 1,4-Dichlorobenzene	146	13.317	13.317	(1.003)	701337	50.0000	48
	83 n-Butylbenzene	91	13.726	13.726	(1.034)	575184	50.0000	40
	84 1,2-Dichlorobenzene	146	13.738	13.738	(1.035)	661406	50.0000	47
	85 Hexachloroethane	117	14.036	14.036	(1.057)	250475	50.0000	52
	86 1,2-Dibromo-3-chloropropane	75	14.594	14.594	(1.099)	31076	50.0000	34
	87 1,2,4-Trichlorobenzene	180	15.500	15.500	(1.167)	217935	50.0000	39
	88 Hexachlorobutadiene	225	15.698	15.698	(1.182)	92526	50.0000	44
	89 Naphthalene	128	15.760	15.760	(1.187)	424623	50.0000	26
	90 1,2,3-Trichlorobenzene	180	16.021	16.021	(1.206)	178172	50.0000	36

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Attachment 5

DoD-Specific QC Requirements

QSM Table B-3

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix is based on the method versions current at the time of publication, regardless of status (promulgated, draft, proposed, etc.). As methods are revised, subsequent versions of this manual may incorporate the changes. If the requirements in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the method shall be followed where appropriate.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081 only, DDT - Methods 8081 and 8270)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	This is a required QC procedure. Continuing calibration blank responses above two times the MDL require corrective action.
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector such as MS detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	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).	NA	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18)	NA	Samples cannot be analyzed without a valid MDL.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria.	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 8270C only)	Daily prior to analysis of samples	Degradation ≤ 20% for DDT	Correct problem then repeat breakdown check	Flagging criteria are not appropriate	No samples shall be run until degradation ≤ 20%. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be observed.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	1. Average response factor (RF) for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs - ≥ 0.050. 2. RSD for RFs for CCCs: VOCs and SVOCs - ≤ 30% and one option below; Option 1: RSD for each analyte ≤ 15% Option 2: linear least squares regression r ≥ 0.995 Option 3: non-linear regression - coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 25% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	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 initial calibration curve.	NA	NA	
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	1. Average RF for SPCCs: VOCs ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for CCCs: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.)	Correct problem, then rerun CV. If that fails, repeat initial calibration. See section 5.5.10 and DoD clarification box 55.	Apply Q-flag if no sample material remains and analyte exceeds criteria.	
Internal standards verification	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within - 50% to + 100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch	No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected > RL.	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
LCS containing all analytes required to be reported, including surrogates	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	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 J-flag 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.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (METHODS 8260 AND 8270) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MSD or sample duplicate	One per preparatory batch per matrix	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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS published by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria.	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.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

Attachment 6

DoD Specific QC Control Limits

QSM Tables D-4, D-5

TABLE D-4. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 WATER MATRIX[11]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,3,5-Trimethylbenzene	102	10	75	130	65	140
1,3-Dichlorobenzene	100	8	75	125	65	130
1,3-Dichloropropane	100	9	75	125	65	135
1,4-Dichlorobenzene	99	8	75	125	65	130
2,2-Dichloropropane	103	11	70	135	60	150
2-Butanone	91	20	30	150	10	170
2-Chlorotoluene	100	9	75	125	65	135
2-Hexanone	92	12	55	130	45	140
4-Chlorotoluene	101	9	75	130	65	135
4-Methyl-2-pentanone	96	13	60	135	45	145
Acetone	91	17	40	140	20	160
Benzene	102	7	80	120	75	130
Bromobenzene	100	8	75	125	70	130
Bromochloromethane	97	11	65	130	55	140
Bromodichloromethane	98	8	75	120	70	130
Bromoform	99	10	70	130	60	140
Bromomethane	88	19	30	145	10	165
Carbon disulfide	100	21	35	160	15	185
Carbon tetrachloride	102	12	65	140	55	150
Chlorobenzene	102	7	80	120	75	130
Chlorodibromomethane	96	13	60	135	45	145
Chloroethane	99	12	60	135	50	145
Chloroform	100	12	65	135	50	150
Chloromethane	83	15	40	125	25	140
cis-1,2-Dichloroethene	99	9	70	125	60	135
cis-1,3-Dichloropropene	100	10	70	130	60	140
Dibromomethane	101	8	75	125	65	135
Dichlorodifluoromethane	93	21	30	155	10	175
Ethylbenzene	100	9	75	125	65	135
Hexachlorobutadiene	97	15	50	140	35	160
Isopropylbenzene	101	9	75	125	65	135
m,p-Xylene	102	9	75	130	65	135
Methyl tert-butyl ether	94	10	65	125	55	135
Methylene chloride	96	14	55	140	40	155
Naphthalene	96	14	55	140	40	150
n-Butylbenzene	103	11	70	135	55	150
n-Propylbenzene	101	9	70	130	65	140
o-Xylene	100	7	80	120	75	130
p-Isopropyltoluene	102	10	75	130	65	140
sec-Butylbenzene	100	9	70	125	65	135
Styrene	100	11	65	135	55	145
tert-Butylbenzene	99	10	70	130	60	140
Tetrachloroethene	96	18	45	150	25	165
Toluene	100	7	75	120	70	130
trans-1,2-Dichloroethene	99	13	60	140	45	150
trans-1,3-Dichloropropene	98	15	55	140	40	155
Trichloroethene	99	9	70	125	60	135
Trichlorofluoromethane	103	15	60	145	45	160
Vinyl chloride	99	16	50	145	35	165

TABLE D-5. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 SOLID MATRIX[12]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	100	9	75	125	65	135
1,1,1-Trichloroethane	101	11	70	135	55	145
1,1,2,2-Tetrachloroethane	93	13	55	130	40	145
1,1,2-Trichloroethane	95	11	60	125	50	140
1,1-Dichloroethane	99	9	75	125	65	135
1,1-Dichloroethene	100	12	65	135	55	150
1,1-Dichloropropene	102	11	70	135	60	145
1,2,3-Trichlorobenzene	97	12	60	135	50	145
1,2,3-Trichloropropane	97	11	65	130	50	140
1,2,4-Trichlorobenzene	98	11	65	130	55	140
1,2,4-Trimethylbenzene	100	12	65	135	55	145
1,2-Dibromo-3-chloropropane	87	16	40	135	25	150
1,2-Dibromoethane	97	9	70	125	60	135
1,2-Dichlorobenzene	97	7	75	120	65	125
1,2-Dichloroethane	104	11	70	135	60	145
1,2-Dichloropropane	95	8	70	120	65	125
1,3,5-Trimethylbenzene	99	11	65	135	55	145
1,3-Dichlorobenzene	98	9	70	125	65	135
1,3-Dichloropropane	100	8	75	125	70	130
1,4-Dichlorobenzene	98	9	70	125	65	135
2,2-Dichloropropane	101	11	65	135	55	145
2-Butanone	94	22	30	160	10	180
2-Chlorotoluene	98	10	70	130	60	140
2-Hexanone	97	16	45	145	30	160
4-Chlorotoluene	100	9	75	125	65	135
4-Methyl-2-pentanone	97	17	45	145	30	165
Acetone	88	23	20	160	10	180
Benzene	99	9	75	125	65	135
Bromobenzene[13]	93	9	65	120	55	130
Bromochloromethane	99	9	70	125	60	135
Bromodichloromethane	100	9	70	130	60	135
Bromoform	96	13	55	135	45	150
Bromomethane	95	21	30	160	10	180
Carbon disulfide	103	19	45	160	30	180
Carbon tetrachloride	100	11	65	135	55	145
Chlorobenzene	99	8	75	125	65	130
Chlorodibromomethane	98	11	65	130	55	140
Chloroethane	98	20	40	155	20	175
Chloroform	98	9	70	125	65	135
Chloromethane	90	13	50	130	40	140

[12] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Methyl tert-butyl ether and Total Xylene although those compounds do appear on the target analyte list for method 8260 (Table C-1 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for MTBE during the LCS study. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section D.5 and for surrogate compounds in section D.6.

[13] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data become available.

TABLE D-5. LCS CONTROL LIMITS FOR VOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8260 SOLID MATRIX[12]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
cis-1,2-Dichloroethene	96	10	65	125	55	135
cis-1,3-Dichloropropene	99	9	70	125	65	135
Dibromomethane	100	9	75	130	65	135
Dichlorodifluoromethane[13]	85	17	35	135	15	155
Ethylbenzene	101	9	75	125	65	135
Hexachlorobutadiene	98	15	55	140	40	155
Isopropylbenzene	103	9	75	130	70	140
m,p-Xylene	102	8	80	125	70	135
Methylene chloride	97	14	55	140	40	155
Naphthalene	84	14	40	125	25	140
n-Butylbenzene	101	12	65	140	50	150
n-Propylbenzene	99	12	65	135	50	145
o-Xylene	101	8	75	125	70	135
p-Isopropyltoluene	104	10	75	135	65	140
sec-Butylbenzene	97	11	65	130	50	145
Styrene	101	9	75	125	65	135
tert-Butylbenzene	99	11	65	130	55	145
Tetrachloroethene	103	12	65	140	55	150
Toluene	99	9	70	125	60	135
trans-1,2-Dichloroethene	100	11	65	135	55	145
trans-1,3-Dichloropropene	96	10	65	125	55	140
Trichloroethene	101	8	75	125	70	130
Trichlorofluoromethane	106	27	25	185	10	215
Vinyl chloride	92	11	60	125	45	140

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120
Benzo[b]fluoranthene	81.8	12.1	45	120	35	130
Benzo[k]fluoranthene	84.6	13.2	45	125	30	135

[14] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

Attachment 7

Additional QA/QC Requirements for MA-DEP

Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
GC/MS Tunes with BFB	Inter-laboratory consistency and comparability	(1) Criteria listed in Table 4 of SW-846 Method 8260B (the same criteria must be used for all analyses) (2) Every 12 hours	No	Perform instrument maintenance as necessary; retune instrument	Suspend all analyses until tuning non-compliance is rectified
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards (2) Low standard must be ≤ Reporting Limit (RL) (3) %RSD should be ≤15 or "r" should be ≥0.99 for all compounds except CCCs which must be ≤30 %RSD or "r" ≥0.99 (4) Must contain all target analytes (5) If regression analysis is used, the curve must not be forced through the origin.	No	Recalibrate as required by method (1) if any of CCC % RSDs >30 or any of CCC "r" <0.99 or (2) if >20% of remaining analytes have % RSDs >30 or "r" <0.99.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds in Environmental Laboratory case narrative. If the average response factor or linear regression are not used for analyte quantitation (e.g., use of a quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Every 12 hours prior to the analysis of samples (2) Concentration level near midpoint of curve (3) Must contain all target analytes (4) Percent difference or percent drift must be ≤20 for CCCs and should be ≤30 for other compounds	No	Recalibrate as required by method (1) if %D of any of CCCs >20, or (2) if %D of >10% of other analytes >30.	Report non-conforming compounds in Environmental Laboratory case narrative.
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Every 20 samples prior to running samples and after calibration standards (2) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$) (3) Target analytes must be <RL except for common laboratory contaminants (such as acetone, methylene chloride, and MEK which must be <5x the RL)	Yes	Locate source of contamination; correct problem; reanalyze method blank.	(1) Report non-conformance in Environmental Laboratory case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the Environmental Laboratory case narrative.
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration (3) Concentration level must be at or near the mid-level (50%) standard (4) Must contain all target analytes (5) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$) (6) Laboratory–determined percent recoveries must be between 70 – 130 for target compounds. (7) Can also be used as CCAL	Yes	Recalculate the percent recoveries; Reanalyze the LCS; Locate source of problem; reanalyze associated samples.	(1) Report non-conformances in Environmental Laboratory case narrative. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined recovery ranges routinely exceed the 100 ± 30% criterion. Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit.

Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
LCS Duplicate	Laboratory Method Precision	(1) Every 20 samples or for each new tune clock, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Recommended to be run immediately after LCS in analytical sequence. (5) Laboratory–determined percent recoveries must be between 70 – 130 for target compounds (6) Matrix and preservative-specific (e.g., water, MeOH, $NaHSO_4$). (7) Laboratory–determined Relative Percent Difference (RPD) must be ≤ 25 except for "difficult" (**) analytes which must be ≤ 50.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Individual laboratories must identify and document "difficult" (**) analytes for which laboratory-determined RPDs routinely exceed the ≤ 25 criterion. (3) Exceedances for these "difficult" analytes must be qualified in Environmental Laboratory case narrative. Analytical data to support the "difficult" analyte classification must be available for review during an audit. (4) Narrate non-conformances
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data-user) (2) Matrix-specific (3) Prepared by fortifying field sample with standard from source different than source used for initial calibration (4) Concentration level - between low (RL) and mid-level (50%) standard (5) Must contain all target analytes. (6) Percent recoveries - between 70 – 130 (7) RPDs should be ≤30 for waters and solids	Yes Only when requested by the data-user	Check LCS; if recoveries acceptable in LCS, narrate non-conformance.	Note exceedances in Environmental Laboratory case narrative.
Surrogates	Accuracy in Sample Matrix	(1) Evaluate surrogate recovery from individual field samples. (2) Minimum of 3 surrogates, at retention times across GC run. (3) Percent recoveries must be between 70-130 for individual surrogate compounds. Laboratory-determined surrogate recovery limits that exceed ± 30% are acceptable for some difficult matrices (wastes, sludges, etc.) with appropriate analytical documentation.	Yes	If one or more surrogates are outside limits, reanalyze sample unless one of the following exceptions applies: (1) obvious interference present (e.g., UCM). (2) for methanol-preserved samples, re-analysis is not required if % moisture >25 and recovery is >10%. (3) if one surrogate exhibits high recovery and target analytes are not detected in sample.	(1) Note exceedances in Environmental Laboratory case narrative. (2) If re-analysis yields similar surrogate non-conformances, the laboratory should report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.

Title: Table II A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 8260B

Required QA/QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Internal Standards (IS)	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 3 at retention times across GC run (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard (Section 5.10 of 8260B) (3) Retention times of internal standards must be within ±30 seconds of retention times in associated continuing calibration standard	No	If one or more internal standards are outside limits, reanalyze sample unless obvious interference present (e.g., UCM)	(1) Note exceedances in Environmental Laboratory case narrative. (2) if re-analysis yields similar internal standard non-conformances, the laboratory should report both results. (3) If re-analysis is performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not re-analyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Quantitation	NA	(1) Quantitation must be based on IS calibration. (2) The laboratory must use the average response factor or linear regression curve generated from the associated initial calibration for quantitation of each analyte The IS used for quantitation must be the one nearest the retention time of the subject analyte.	NA	NA	(1) If the average response factor or linear regression are not used for analyte quantitation (e.g. quadratic equation), this must be noted in the Environmental Laboratory case narrative with a list of the affected analytes. (2) It is essential that the laboratory clearly document the calculation of analyte concentrations when non-linear calibrations are employed.
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc) for each analysis must be "reported". (3) Refer to Section 3.3, TIC Compounds by GC/MS for guidance	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Complete analytical documentation for diluted and undiluted analyses is to be available for review during an audit. (3) TICs will be evaluated at the discretion of the LSP consistent with the guidelines presented in Appendix II A–4. (4) The performance of dilutions must be documented in the Environmental Laboratory case narrative.

GC/MS = Gas Chromatography/Mass Spectrometry
BFB = 4-Bromofluorobenzene
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
%RSD = Percent Relative Standard Deviation
UCM = Unresolved Complex Mixture

"r" = Correlation Coefficient
CCC = Calibration Check Compounds
RPDs = Relative Percent Differences
TIC = Tentatively Identified Compound
NA = Not Applicable

** Potentially "difficult" analytes include: acetone, bromomethane, chloroethane, dichlorodifluoromethane, diethyl ether, dibromochloromethane, hexachlorobutadiene, MEK, 4-methyl-2-pentanone, 1,4-dioxane and trichlorofluoromethane

Attachment 8

Corrective Action and Documentation Examples

OCCURRENCE	ACTION	DOCUMENTATION
1. Initial calibration does not meet QC criteria.	1. Investigate source of problem, determine if source is an instrument problem or a standard solution problem. If problem is with a single point of the ICAL, reanalyze the bad standard and reevaluate. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	1. Notation in instrument run log book, and if necessary notation in instrument maintenance log book.
2. Initial calibration verification check does not meet QC criteria.	2. Investigate source of problem, determine if source is with ICAL or ICV, is it an instrument problem or a standard solution problem, reanalyze ICV or perform new ICAL.	2. Notation in instrument run log book, and if necessary notation in instrument maintenance log book. If source determined to be bad standard solution, formal corrective action form must be initiated.
3. Continuing calibration verification check does not meet QC criteria.	3. Investigate source of problem. If source is instrument, perform instrument maintenance and reanalyze CCV. If CCV still will not pass, repeat the above, or perform new initial calibration. Depending on extent of problem, major maintenance or invoking manufacturer service contract for instrument repair will be performed.	3. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
4. GC/MS tune does not meet method criteria.	4. Investigate source of problem, evaluate instrument response to cal gas (PFTBA), when instrument response to PFTBA is improved, re-inject BFB tune.	4. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
5. Method blank contains target		

compound above reporting limit.	5. Investigate source of problem. Reanalyze all effected samples. If reanalysis is within holding time, report only these analyses. If they are beyond holding time, report both sets and notify project manager. If contaminant is not present in samples, data may be released with commentary.	5. Notation in instrument run log book. If instrument maintenance performed, notation in maintenance log book.
6. Surrogate standard outside of acceptable range.	6. Investigate source of problem. If it is determined to be an instrument problem, reanalyze sample. If it is determined to be a preparation problem, reextract/reanalyze the sample. If it can be determined to be an obvious matrix problem (masking of surrogate by target or non-target compound at significantly greater concentration, excessive hydrocarbons in sample, other knowledge of sample matrix, etc.) the sample may be reanalyzed at dilution to reduce interference or reported with notation in narrative, depending on project objectives.	6. If only the reanalysis is reported make a notation in the instrument run log. If both sets of data are to be reported, notation in instrument run log, reextraction request form, preparation logbooks, commentary on data review checklist to be included in project narrative, flagging all non-compliant values on Form 2 of data report. If source of problem found to be systematic (bad spike solution, etc). a formal corrective action form must be initiated.
7. Compound out of acceptance range in laboratory control sample.	7. Investigate source of problem. If LCS is acceptable per method/SOP specifications, associated sample data can be reported. If LCS recovery is above upper QC limit, and if analyte is not detected in associated samples, data may be flagged and reported. If LCS is not acceptable per method/SOP requirements, reanalyze all associated samples. If insufficient sample volume, notify project manager to discuss with client, report initial data if no other sample can be provided.	7. If LCS is acceptable per method/SOP, flag all compounds out of range on Form 3 of data report, if samples are reanalyzed within holding times, note in instrument run logbook. If samples are beyond holding time and both sets of data are to be reported, note in instrument run logbook and commentary in data review checklist to be included in project narrative. If reanalysis cannot be performed due to insufficient sample, commentary in data review checklist to be included in project narrative. If source of problem found to be systematic (bad spike solution, etc). a formal corrective action form must be initiated.
8. Compound in sample exceeds upper calibration standard concentration.		

9. Instrument blank (GC) contains contamination above QC criteria. 10. Matrix spike recovery out of QC range. 11. Duplicate (or MSD) relative percent difference exceeds QC limit. 12. Internal Standard areas exceed QC criteria. (-50% to +100%)	8. Reanalyze sample at dilution. If calibration limit exceedence is the only QC problem, report both initial and dilution analyses. If initial analysis has multiple QC problems, evaluate further to determine if initial run is to be reported (often this cannot be determined until the results of the dilution are evaluated). Instrument must be shown to be free of carryover contamination prior to acceptable analysis of next sample. If running instrument using autosampler, evaluate following sample. If following sample contains less than reporting limit of compound, the analysis is valid, and no instrument blank is required. If following sample(s) contain compound (typically in decreasing concentration—carryover typically occurs at 1% of concentration of high sample in following analysis, with effect more pronounced for later-eluting compounds. Effected samples must be reanalyzed if sufficient volume exists. 9. Investigate source of problem, decontaminate purge and trap instrument, reanalyze all effected samples. 10. Evaluate problem. If duplicate spike (MSD) shows same effect, it is generally matrix interference. If concentration of spike analyte is significantly (approx. 4 times) greater in unspiked sample, this is matrix interference masking quantitation of spike concentration. If source cannot be determined, reanalyze spike sample. 11. Evaluate problem. If concentration of analyte is	8. Notation in instrument run log book. If initial analysis is reported, flag compound exceeding calibration limit with "E" on data report and commentary on data review checklist to be included in project narrative. If only diluted analysis is to be reported, commentary in data review checklist to be included in project narrative. If both initial and dilution are to be reported, all of the above. 9. Notation in instrument run logbook, and if instrument maintenance performed, in instrument maintenance logbook. 10. Flag percent recovery on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative. 11. Flag RPD on data reporting Form 3. Include commentary on issue on data review checklist for inclusion in report narrative.

A. CCV. B. QC (blank, LCS) C. Samples and MS/MSD.	close to reporting limit, variation of analysis is acceptable. If sample is soil or other heterogeneous matrix, high RPD is typical. If sample is a typically homogeneous matrix, reanalyze duplicate sample. 12. A. Evaluate problem. Re-analyze CCV. If the CCV does not meet criteria re-analyze the initial calibration and proceed with CCV/QC/samples. B. Evaluate CCV and QC. As blank and LCS are "interference-free matrix" the IS areas should be within the same limits as the CCV. Evaluate for potential problems. If time allows (data not required on a rush basis), reanalyze QC prior to sample analysis. If insufficient time due to client deadline, data may be reported (as they meet method requirements) but the issue should be noted for the data reviewer and for the client. C. Evaluate CCV and QC. The IS areas may indicate a potential problem, or matrix interference. If the CCV and batch QC meet criteria, document and report results as matrix interference. In particular, if the recovery of the surrogate standard associated with the IS compound is within the recovery range, then the internal standard method is effectively quantifying the compounds. If the associated surrogate is outside of the recovery criteria, the IS issue is impacting quantitation. Evaluate whether this indicates a potential high or low bias for the associated compound results (low IS=high surrogate=high bias; high IS=low surrogate=low bias) This requires notation and	12. A. Document in the analytical run log. B. If the criteria are met after re-analysis, document in run log. If the criteria have not been met and the results of the sample batch are reported, document in the run log and the Corrective Action Logbook. Have the supervisor review situation, initial/date, and include a comment on the data review checklist for the data reviewer and for inclusion in the narrative information to the client. C. If the CCV and QC meet criteria, document in the run log and on the package checklist for inclusion in the narrative submitted to the client. Note that certain compounds may be potentially high bias or potentially low bias due to IS recoveries outside of range. If QC and samples do not meet criteria and the results are reported, document in the run log, document in the LIMS Corrective Action Logbook with a CAR number, have supervisor initial/date, include a comment on the package checklist for the data reviewer and for inclusion in the narrative.

	communication of the effect to the data reviewer and the client. Based on the severity of the problem, discuss with supervisor, technical director and /or reanalyze effected samples. If results are reported as is, document per 12C.	

MITKEM LABORATORIES,
A DIVISION OF SPECTRUM ANALYTICAL INC.

STANDARD OPERATING PROCEDURE

For

Gasoline Range Organics in Aqueous and Soil Samples

By

Gas Chromatography-Flame Ionization Detector (GC-FID)

using Methods SW-846 8015 and Maine 4.2.17

Rev. 10

	Signature	Date
QA Director:	Shanyn B Sanh	1/23/09
Lab Director:	Kin Chin	1/23/09

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Rev. 10

1. Scope and Application

This Standard Operating Procedure (SOP) describes the analysis of gasoline range petroleum hydrocarbon products in aqueous and soil samples using Gas Chromatography with a Flame Ionization Detector (GC-FID) according to protocols discussed in SW846 Method 8015 and Maine Method 4.2.17. These methods are used in conjunction with Purge and Trap sample introduction technique. The Gasoline Range Organics (GRO) methods facilitate the general identification and quantitative analysis of hydrocarbons derived from petroleum distillate products with boiling points approximately equal to or less than that of gasoline.

This SOP is developed to follow the guidelines specified in the SW846 methods. To further familiarize with the procedures, the analyst is highly encouraged to consult the following instrument manuals as well as the methods themselves (see reference section):

- Hewlett Packard HP 5980 Gas Chromatograph with Flame Ionization Detector Manual
- Hewlett Packard EnviroQuant GC/MS Manual
- OI Corporation Manuals on Model 4560 Purge and Trap Concentrator and Model 4551A Autosampler
- OI Corporation Manual on Model 4410 Flame Ionization Detector

2. Personnel Qualifications and Responsibilities

Laboratory analysts must be sufficiently trained and familiarized with the GC instrumentation and software in order to perform sample testing and analysis, as well as instrument maintenance and troubleshooting, to a degree considered sufficient to accomplish the tasks necessary with a reliable level of accuracy and efficiency.

The analysts may revise the SOP in conjunction with the QA Director as necessary to improve upon the Gasoline Range Organics method or to accommodate for any changing conditions that would affect the performance of the method.

3. Summary of Procedure

Samples containing volatile compounds are loaded onto a Model 4551A autosampler and are introduced into the GC by purge-and-trap system. The analytes are desorbed onto the column directly. The column is temperature programmed to separate the compounds, and they are then detected using a flame ionization detector.

The GRO compounds' responses are compared to the collective response of a six-point calibration curve in order to quantify the concentration of gasoline range organics in the sample being tested.

4. Sample Preservation, Containers, Handling and Storage

4.1 Water samples are collected in the field in 40mL glass vials with a Teflon septum. Two glass vials for each water sample should be provided in order to accommodate re-analysis. The water sample is preserved in the field to a pH of <2 with HCl. When received, samples are refrigerated to a temperature of 4°C ± 2°C, and are stored in the lab at that same temperature.

4.2 Soil samples are generally collected into pre-weighed VOA vials containing 5mL of purge and trap grade methanol. The vials are re-weighed at the laboratory to determine the sample weight. Mitkem typically purchases pre-weighed vials from the company's bottle supplier. These are available in either 5mL or 10mL methanol aliquots, as printed on the bottle label. The weight of sample is not critical, but the ratio of sample to methanol should be 1:1, and the soil should be completely covered by methanol. The client is instructed not to add additional labels to the sample vial. A separate aliquot of unpreserved soil needs to be collected for the determination of percent moisture, as described in a separate SOP. In areas where soil samples for VOC analysis are not required to be preserved (New York State as of January, 2006 for example), samples may also be collected in 2 or 4 ounce glass containers and are stored at 4°C ± 2.

4.3 The samples are recorded in the laboratory-tracking log upon receipt.

4.4 All samples must be analyzed within 14 days of collection.

5. Interferences and Potential Problems

Interference can occur with non-gasoline components eluting within the GRO range, such as certain chlorinated and oxygenated hydrocarbons. If it is known or suspected that compounds are present which are not constituents of gasoline, the analyst should suggest additional analysis.

Sample contamination can occur when volatile organic compounds diffuse through the septum of the sample container during transport or storage. Trip blanks may be provided for analysis if such contamination is expected to occur.

Contamination may also occur due to carryover of compounds from a high concentration sample to a low concentration sample if sequentially analyzed. Care should be taken with the cleaning procedures of syringes and other sample handling equipment in order to be sure carryover does not occur. Carryover may also occur if a high concentration sample contaminates the sample sparger and trap. Screening of samples is recommended to protect laboratory equipment and to prevent carryover. If carryover occurs or is suspected, a re-analysis of the affected sample must be done. Proper cleaning and baking of the trap and sample sparger should be done after a concentrated sample in order to prevent further carryover contamination from occurring.

6. Equipment and Apparatus

A Hewlett Packard Model 5890 GC fitted with a Flame Ionization Detector (FID), or an OI Model 4410 Flame Ionization Detector in series with an OI Model 4430 Photoionization Detector (PID) is used for the analysis. The GC is connected to an OI Model 4560 Purge and Trap Concentrator and an OI Model 4551A Autosampler. With the PID-FID detectors in series, the PID is not used during GRO analysis. Compounds of interest are swept through the PID into the FID for analysis.

The GC operates under the control of a Hewlett Packard Chemstation System consisting of a Hewlett Packard Vectra PC. Hewlett Packard EnviroQuant Software is used to control both data acquisition and data reduction.

The following accessories are used with the GC system:

a) Column: DB-624, 105m x 0.45 mm ID, 2.55um film, Cat.124-1374, J&W Scientific.

b) The GC-FID operating conditions are as follows:

General GC conditions:	
Column linear velocity	30cm/min
Make-up gas flow rate	90mL/min
Injector Temp	n/a
Detector	250°C
Initial GC Temp	40°C

Initial GC Time	1.0 min
Initial Temp Ramp	10°C/min
Secondary GC Temp	160°C
Secondary GC Hold	2.0 min
Secondary Temp Ramp	20°C/min
Final GC Temp	240°C
Final GC Hold	8.0 min

c) Purge and Trap Conditions:

Trap Material	Tanax/silica
Purge Gas Flow Rate	40mL/min
Purge Temp	35°C
Purge Time	11 min
Dry Purge	0 min
Desorb Preheat	100°C
Desorb Temp	190°C
Desorb Time	2 min
Bake Temp	210°C
Bake Time	15 min

Maintenance schedules and procedures for all of the equipment used in the GRO method should be followed as described in each piece of equipment's respective manual. All non-routine maintenance performed should be documented in the instrument maintenance logbook, located in LIMS.

7. Reagents

The following description is based on stock standards purchased from the quoted sources. Equivalent standards from other commercial sources can be used as long as the standards are traceable to sources that can be compared to reference materials. It is also important that only high purity standards (> 96% purity) be used.

7.1 Surrogate Standard:
p-Bromofluorobenzene, Restek (Cat. No. 30003) at 5,000µg/mL.

7.2 GRO Standard:
PVOC/GRO Mix (Wisconsin), Ultra (Cat. No. UST-100) at 1000µg/mL.

7.3 GRO Laboratory Control Sample (LCS)/ Initial Calibration Verification(ICV):
Unleaded gasoline composite standard, Restek (Cat. No.30081) at 2,500µg/mL.

7.4 Analyte-Free Reagent Water (ASTM Type II water) – prepared by filtering tap water through a column of activated charcoal granules.

7.5 Purge and trap grade methanol, Fisher Scientific or another source of equivalent quality, is used for standard preparation.

7.6 Gasses used for the instrumentation and detectors are listed below:

Carrier Gas and Purge Gas	99.999% Helium
FID gas	Hydrogen (0.5 Grade) at about 25mL/min. Air at about 100mL/min.

8. Procedure

8.1 Standard preparation: All standard preparations are documented in the Primary, Intermediate and the Working Standard Logbooks located in the lab.

8.1.1 Primary Standards:

All primary standards received from vendors are logged into the Volatiles Primary Standard Logbook. The standards are labeled PV*yymmddX* where:

PV = Primary Volatile standard,
yymmdd = date the standard is received, and
X = the order the standard is logged into the Logbook on that date, in increasing alphabetical order.

Thus, VP990725C represents the third primary Volatile standard logged into the Primary Volatile Standard Logbook on July 25, 1999.

Where applicable, certificates of analysis which accompany the standards should also be labeled with the same identifiers and archived together to facilitate tracing of the standards.

8.1.2 Working Standards:

All of the working standards are logged into the Volatiles Working Standard Logbook and labeled as VW*yymmddX* where:

VW = Volatile Working standard
yymmdd = date the working standard is prepared, i.e., 070719 for a standard prepared on July 19, 2007
X = the order that the working standard is prepared on that date, in increasing alphabetical order
The working standards are stored in amber vials fitted with Teflon septa. They are stored in the freezer (-10°C to –20°C) to minimize volatilization

losses. The standards are replaced every 30 days or sooner if degradation has occurred.

A smaller or larger volume of the primary stock solution may be used to prepare the surrogate standard working solution. The final volume will be adjust accordingly to achieve the same working concentration.

8.1.2.1 Surrogate Standard (BFB):

The working surrogate standard is prepared by combining 80µL of the primary p-Bromofluorobenzene standard at 5,000µg/mL and diluting to 4mL using methanol. Final concentration of the surrogate standard solution is 100µg/ml. The addition of 1µL of this solution to 5mL sample aliquot gives a Surrogate concentration of 20µg/L on column.

8.1.2.2 GRO working standard:

The GRO working standard is prepared by taking 100uL of the primary standard at 1000µg/mL and diluting it to 1ml with methanol. The final concentration of the working GRO standard is 100µg/mL. The addition of 20µL of this solution to a 40mL aliquot gives a Standard concentration of each individual component is at 50µg/L on column, and GRO at 500µg/L on column.

8.1.2.3 GRO ICV (Gasoline Composite Standard):

The instrument is calibrated using a mixture of gasoline components (individual compounds). The initial calibration is verified using a gasoline product standard. The LCS and MS/MSD are also typically prepared using this gasoline product standard. The GRO ICV is prepared by taking 80µL of the primary standard at 2500µg/mL and diluting it to 1.0 mL with methanol. The final concentration of the working GRO ICV is 200µg/mL. The addition of 100µL of this solution to a 40mL aliquot gives a standard concentration of 500µg/L on column.

8.2 Initial Calibration:

The six levels of the calibration curve are loaded onto the autosampler and purged. The six levels used are at concentrations of 2.5, 5, 20, 50, 100, and 200µg/L.

Preparation is as follows:

Standard Conc. (µg/L)	Amount Working Std. Used (µL)	BFB(µL)
2.5	1	1
5.0	2	1
20	8	1
50	20	1
100	40	1
200	80	1

The concentrations above are diluted to 40mL using analyte-free reagent water A typical chromatogram (10 components at 50µg/L) is shown in **Attachment 1** with the Integration Report.

8.2.1 Procedure for Initial Calibration:

Purge each calibration standard using the same technique that will be applied to the samples. Tabulate the peak height or peak area responses against the concentration of each individual ten compounds and BFB.

8.2.1.1 The response Factor (RF) is determined as follows:

$$RF = \frac{A_x}{C_x}$$

where: A_x = peak area of BFB or gasoline component to be measured
C_x = concentration of BFB or gasoline component to be measured

8.2.1.2 If the RF value over the working range is constant (≤ 20% RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations.

8.2.1.3 The % Relative Standard Deviation (%RSD) of the RF is also calculated using:

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

where: Standard Deviation = $\sqrt{\sum (Xi - \bar{X})^2 / (n-1)}$
where: Xi = each individual value used to calculate the mean
X = the mean of n values
n = the total number of values

8.2.1.4 A collective response factor must also be established for the entire hydrocarbon range. To calculate the collective RF, tabulate the summation of the peak areas of all 10 components against the total mass injected . The collective RF is the average of the responses for the Deisel Range Organics (DRO) individual components.

8.2.1.5 The Initial Calibration also sets the retention times necessary for the (DRO)integration for field and QC samples. The RT_i is set at the time where methyl-tertiary-butyl ether (MTBE) starts to elute, and RT_f is set after the elution of naphthalene. RT_i and RT_f are saved as reference points and will be used for all subsequent instrument and method blanks, lab control samples, duplicate matrix spikes and sample analysis.

8.2.1.6 The area count of BFB is independently integrated.

8.2.2 Each initial calibration is verified by the analysis of a standard from an independent second source. This is typically a gasoline product standard. This initial calibration verification (ICV) must be within +/- 20% of the true value for the initial calibration to be acceptable.

The ICV integration is performed by constructing a baseline from the point (RT_i) on the chromatogram where the gasoline components start to elute (methyl-tertiary-butyl ether (MTBE)) to a point after the hydrocarbons have completely eluted off the GC column (naphthalene) (RT_f).

A gasoline product standard chromatogram and integration report are shown in **Attachment 2.**

8.3 Continuing Calibration:

Continuing Calibration standards are analyzed to ensure that the GC-FID continues to meet instrument sensitivity and linearity requirements.

8.3.1 Frequency of Continuing Calibration:

The Continuing Calibration standard must be performed once every 24 hours, or after 20 injections, whichever is more frequent. If time remains in the 24 hour time period after meeting the acceptance criteria for the

Initial Calibration, samples may be analyzed. It is not necessary to analyze continuing calibration standards if the Initial Calibration standards meet the Continuing Calibration acceptance criteria.

8.3.2 Procedure for performing Continuing Calibration:

The Continuing Calibration is analyzed at 50µg/L.

Calculate the percent difference between the Continuing Calibration RF and the values from the most recent initial Calibration.

The percent difference is determined as follows:

$$\% \text{ Difference} = \frac{RF_c - RF_i}{RF_i} \times 100$$

where: RF_c = response factor from Continuing Calibration
RF_i = mean relative response factor from the most recent Initial Calibration

8.4 Analytical Sequence:

The order used for the testing of samples is as follows:

1. Initial Calibration if necessary or Continuing Calibration
2. Instrument Blank
3. Method Blank
4. Laboratory Control Sample/Duplicate
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
18. Sample
19. Sample
20. Sample

21. MS
22. MSD
23. Ending CC, etc.

This sequence is documented in the Volatiles Instrument Run Log (**Attachment 3**).

8.5 Initial Procedure:

8.5.1 Hardware Start-up:

The Gas Chromatograph, Sample Concentrator, and Multi-sampler should all be turned on, and the Gas Chromatograph and Sample concentrator should be allowed to reach their proper operating equilibrium temperatures.

The method file in the Sample Concentrator which is appropriate for the GRO method should be loaded from memory, and the detectors for the Gas Chromatograph should be on and operational. Make sure the Flame Ionization Detector has been lit.

8.5.2 Software Start-up:

On the computer that controls the Gas Chromatograph and Sample Concentrator, run the appropriate Enviroquant programs that are needed for sample running and processing.

Make sure a sequence has been entered properly in the Enviroquant software that will give all information on sample identification and concentration, and is in the same order in which the samples are to be run.

The Sample Concentrator needs to have the appropriate sequence entered into memory that will match the sequence entered into the Enviroquant software.

8.5.3 Sample loading:

8.5.3.1 Instrument/Method Blanks: Aqueous Instrument/Method Blanks are prepared by spiking 1µL of the BFB spiking solution into 5mL of analyte-free water. Soil Instrument/Method Blanks are prepared by spiking 1µL of the BFB spiking solution and 100µL of methanol into 4.9mL of analyte-free water. The Instrument Blank is analyzed using the same procedure for sample analysis.

8.5.3.2 Aqueous samples: Samples are analyzed in 5mL aliquots. The 40ml vial is placed in a location in the autosampler tray. Immediately prior to analysis, an aliquot of the sample is withdrawn from the VOA vial by the autosampler using a syringe. An aliquot of the surrogate is added and the sample aliquot is transferred to the purge-and-trap sparger and injected into the sparger vessel. The sample is ready for analysis.

8.5.3.3 Soil samples: Mix the soil/methanol preserved aliquot by shaking and allow to settle. Note in the preparation logbook if the soil is not covered by the methanol layer.

Weigh the vial, being sure to tare-out the weight of the Mitkem sample label. Record the weight in the Soil Weight Logbook. Also record the initial tare weight of the preservative/vial printed on the sample label by the bottle supplier.

Open the vial and remove 800 µL into a 40ml VOA vial with VOA-free water and then follow the same procedure of aqueous samples.

8.5.3.4 For unpreserved soils, first tare weigh the sparger tube with the test tube holder. Then weigh 5.0 to 5.5g of soil sample into a pre-weighed VOA vial containing 5mL of methanol. For Maine GRO samples received as unpreserved soils, the method requires a minimum of 10 grams of soil to be extracted, with a 1:1 ratio of soil to methanol. Shake vigorously and allow to settle. Analyze per procedure above.

8.5.4 Sample Analysis:

Once enough of the samples are loaded and the sequence is entered properly, start the sequence in the Enviroquant software and on the autosampler. The samples are analyzed using the previously listed GC conditions in the Equipment and Apparatus section.

Note: While both may be started before all the samples are loaded, make sure all necessary samples are loaded in the proper positions on the autosampler.

8.5.5 Quantitation of Samples:

From the chromatogram, the area of the peaks is integrated using the RT_i to RT_f approach. The sample integration is performed by constructing a baseline from a point (RT_i) on the chromatogram where the gasoline

components start to elute to a point after the hydrocarbons have completely eluted off the GC column (RT_f). RT_i and RT_f are established from the initial calibration, and used for all method blanks, lab control samples, duplicate matrix spikes and sample analysis. For samples that contain higher boiling distillates such as diesel, only those hydrocarbon components within RT_i and RT_f are included for the quantitation.

8.5.6 Data Processing:

8.5.6.1 Once a run is complete, the data file will be stored in the computer. Load the appropriate file, and process the sample using the Enviroquant software. If the sample is a calibration level, update the five point calibration curve or the continuing calibration as necessary before processing any other samples.

8.5.6.2 After processing, the GRO are manually integrated using the Enviroquant software, as described in **section 8.5.5**. This is because the GRO compounds cover a range that is too extensive to be integrated by the Enviroquant software itself. After the GRO compounds are properly integrated, check the integration of the surrogate standard to be sure it is also integrated properly. The analyst should feel free to perform manual integrations as necessary, but only if such integrations follow the guidelines put forth in this document.

8.5.6.3 The Instrument Blank area is subtracted from ALL field and QC sample analyses run in the associated sequence by the Enviroquant software. The area of integration from the instrument blank is entered in N4 under user defined items. Open the GC data acquisition software, under Initial Cal> Edit compounds> View GRO> Page 2. After the entire sequence has been processed, type MAC "TPHBS" at the bottom of the data acquisition window.. Then type DOLIST "THPBS". Select the files you want to subtract the IBLK from and process.

8.5.7 Analysis of Data:

After the sample is properly processed and integrated, the analyst must check to see if the sample passes the acceptance criteria appropriate for that particular type of sample. The results of the analysis are then recorded in the instrument run log, as well as any corrective action necessary (i.e. OK, needs dilution…). Corrective action responses are detailed in **section 12**.

9. Data Reduction and Calculations

9.1 Integration Range:

The concentration of Gasoline Range Organics is determined from a summation of the total peak area for all chromatographic peaks eluting from methyl-tertiary-butyl-ether (RT_{I}) to naphthalene (RT_{f}), inclusive. Quantitation is based on a collective average response from the calibration curve.

9.2 Calculation:

Qualitative identification of the petroleum product, if requested by the client, is made by comparing the sample chromatogram to those of the standard gasoline chromatograms.

Quantitate the sample as follows:

Aqueous sample concentration (in mg/L) $= \frac{A_x}{RF_i} * DF$

where: RFi = Mean relative response factor from the most recent calibration.
Ax = Peak area of gasoline or BFB to be measured.
DF = Dilution Factor.

Soil sample concentration (in mg/Kg) $= \frac{A_x}{RF_i} * \frac{5}{(W_s)(\%S)}$

where: RFi = Mean relative response factor from the most recent calibration.
Ax = Peak area of gasoline or BFB to be measured.
W_s = Volume of sample used for analysis, in g.
%S = percent solids as calculated below.

9.3 The percent solids is calculated as follows:

$$\% \text{ solids (S)} = \frac{DW}{WW} \times 100\%$$

where: DW = Sample weight (g) dried at 105°C overnight
WW = Sample weight (g) before drying

9.4 Significant Figures:

All results for GRO range concentrations are reported to two (2) significant figures.

10. Quality Assurance/Quality Control

10.1 Method Detection Limits:

Method Detection Limits required per Maine GRO are 10ug/L for waters, and 2.5 mg/Kg for soils. There is no annual requirement for the study in the document. These limits have been achieved and are entered into LIMS. According to the NELAC standard, chapter 5; tests which are not reported below the concentration of the lowest level of the initial calibration, are not required to perform annual MDL studies. Mitkem does not report GRO results below our reporting limits.

Navy NFESC projects using GRO by SW846 8015 methodology require annual MDL studies.

Annually, the laboratory performs the Maine method's Initial Demonstration of Capability to generate acceptable accuracy and precision for this method, by preparing and analyzing 7 LCS for both water and soil. The recoveries and RPD are calculated and stored in the QA Office.

10.2 Quality Control:

10.2.1 Initial Calibration acceptance criteria:

The initial calibration for GRO and BFB is acceptable if the percent RSD of the RF does not exceed 20 %.

ICV analysis following the ICAL must meet the +/- 20% recovery criteria.

Initial Calibration acceptance criteria must be met before any samples, Lab Control Samples, Duplicate Matrix Spikes or Blanks are to be analyzed.

10.2.2 Continuing Calibration acceptance criteria:

The Continuing Calibration is deemed acceptable if the RF does not exceed those of the Initial Calibration for more than 20% for both GRO and BFB.

10.2.3 Instrument Blank:

An Instrument Blank is analyzed to determine the level of background noise/baseline drift associated with the processing and analysis of samples.

An Instrument Blank is analyzed immediately after the Continuing Calibration (or last level of an ICAL) and immediately before the Method Blank. The instrument blank integration is performed the same as that for all analyses, from RT_i to RT_f. The resultant area is subtracted from ALL analyses run in the sequence, excluding individual component standards.

10.2.4 Surrogate recoveries:

The recovery of the system monitoring compound BFB in all samples, blanks, matrix spikes and matrix spike duplicates will be calculated using the equation below:

$$\% \text{ Recovery} = \frac{\text{Concentration (amount) found}}{\text{Concentration (amount) spiked}} \times 100$$

The percent recovery control limits for BFB are set annually using control charts of in-house data points. See Mitkem SOP No. 80.0010 for details. Recovery of BFB in Method Blanks must be within the control limits. The annual limits can be found in the test information form in LIMS.

10.2.5 Method Blank:

Method Blanks are analyzed to determine the level of contamination associated with the processing and analysis of samples.

Method Blanks are analyzed immediately after the Instrument Blank and immediately before the Lab Control Sample.

10.2.5.1 Preparation of Method Blanks:

Aqueous Method Blanks are prepared by spiking 1µL of the BFB spiking solution into 5mL of analyte-free water . Soil Method Blanks are prepared by spiking 1µL of the BFB spiking solution and 100µL of methanol into 4.9mL of analyte-free water. The Method Blank is analyzed using the same procedure for sample analysis.

10.2.5.2 Frequency of Method Blanks:

One Method Blank is analyzed per batch of samples. A Method Blank is generated at a frequency of one for up to 20 samples of a similar matrix.

10.2.5.3 Acceptance criteria for Method Blanks:

- The percent recovery control limits for BFB are set annually using control charts of in-house data points.
- The instrument blank area must be subtracted from the method blank prior to evaluation.
- The concentration of target compounds found in the Method Blank must be less than the reporting limit of 25µg/L.

For Navy NFESC work and Maine GRO projects, the concentration cannot exceed one half the reporting limit.

10.2.6 Laboratory Control Sample (LCS):

Laboratory Control Samples are analyzed to determine the accuracy associated with the processing and analysis of samples. The LCS is prepared from a different source than that of the calibration standards.

10.2.6.1 Preparation of LCS:

An aqueous matrix LCS is prepared by spiking 100µL of a gasoline spiking solution at into a 40mL VOA vial with analyte-free water and 1µL of the BFB spiking solution will be added to 5ml of the spiked solution when the Autosampler takes the aliquote to the sparge tube Soil matrix LCS are prepared by spiking 100µL of a gasoline spiking solution and 700 µL of P&T methanol into a 40mL VOA vial with analyte-free water and 1µL of the BFB spiking solution will be added to 5ml of the spiked solution when the Autosampler takes the aliquote to the sparge tube. The LCS is then analyzed using the same procedure for sample analysis.

10.2.6.2 Frequency of LCS:

One LCS is analyzed per batch of samples per matrix. An LCS is generated at a frequency of once every 20 samples. If an MS/MSD is not analyzed, an LCSD must also be analyzed.

10.2.6.3 Acceptance criteria for Laboratory Control Samples:

- The percent recovery control limits for BFB are set annually using control charts of in-house data points.
- The instrument blank area must be subtracted from the LCS prior to evaluation.

- The percent recovery of GRO for LCS is 80-120 for either matrix per Maine Method 4.2.17.
- When an LCSD is run, the %RPD should be ≤20%.

10.2.7 Matrix Spikes and Matrix Spike Duplicates:

Matrix spikes and matrix spike duplicates may be analyzed to assess sample matrix effect with the processing and analysis of samples.

10.2.7.1 Preparation of Matrix Spikes and Matrix Spike Duplicates:
Aqueous matrix spikes are prepared by spiking 100µL of a gasoline spiking solution at into a 40mL VOA vial with sample and 1µL of the BFB spiking solution will be added to 5ml of the spiked sample when the Autosampler takes the aliquote to the sparge tubeInsure soil samples have a total methanol volume of 800µL of methanol in a 40ml of Vial with analyte-free water. The sample is then analyzed using the same procedure for sample analysis.

10.2.7.2 Frequency of Matrix Spikes and Matrix Spike Duplicates:
Duplicate matrix spikes are analyzed per batch of samples per matrix. Duplicate matrix spikes are generated at a frequency of at least once every 20 samples for samples of similar matrix.

10.2.7.3 Acceptance criteria for Duplicate Matrix Spikes:
Please note that the following criteria are advisory as accuracy and precision could be adversely affected by sample matrix. Recovery and precision outside of the limits of recovery should be documented in the project narrative to flag the possibility of sample matrix effects.

- The percent recovery control limits of BFB for MS/MSD is set annually using control charts of in-house data points.
- The percent recovery limits of GRO for MS/MSD is 60-140% per Maine method 4.2.17.
- The instrument blank area must be subtracted from the MS/MSD prior to evaluation.
- % RPD for GRO recoveries should be ≤20%, where %RPD is determined as follows:

$$\%RPD = (R_{ms}-R_{msd}) * 200/(R_{ms} + R_{msd})$$

10.2.8 Acceptance criteria for Samples:

Please note that the following criteria are advisory as accuracy and precision could be adversely affected by sample matrix. See corrective actions for samples in **section 12** for more information on sample matrix effect.

- The BFB recovery should fall within the in-house limits for the run to be considered valid, excluding matrix effect.
- The instrument blank area must be subtracted from the sample prior to GRO concentration quantitation.

10.3 All standards prepared from a primary standard expire on or before the primary standard's expiration date.

11. Data Validation and Reporting

11.1 Reporting Limits:

The Reporting Limits (RL) for GRO are set to be equal to the lowest calibration standard that is still within the linear range of the calibration curve. The reporting limit for the entire range is based upon the summation of the reporting limit for each individual analyte in the component standard.

11.2 Data Review:

All data calculations must be reviewed 100% by the analyst or a peer. A final full technical review is done by the Lab Manager or another senior chemist, according to Mitkem's data review and validation procedures. GRO range integration is always checked for accuracy in all samples, Method Blanks, Laboratory Control Spikes, Matrix spikes, and Matrix spike duplicates.

12. Corrective Action Procedures

12.1 Corrective Action for Continuing Calibration:

Repeat the Continuing Calibration if the 20% RF criteria is not met. Repeated failure to meet the criteria will necessitate performing a new Initial Calibration.

12.2 Corrective Action for Method Blanks:

Any Method Blank that fails to meet any of the criteria in **section 10** must be reanalyzed. Any samples that are analyzed with the non-compliant Method Blank will be reanalyzed.

12.3 Corrective Action for LCS:

Any LCS that fails to meet any of the criteria in **section 10** must be reanalyzed.. Any samples analyzed with the non-compliant LCS will be reanalyzed.

12.4 Corrective Actions for Matrix Spikes and Matrix Spike Duplicates:

If the LCS is valid in the same batch, then no corrective action is required for Matrix spikes out of control limits, as long as there is reasonable consistency between the matrix spike and the matrix spike duplicate. In particular, if the sample contains significant GRO in comparison to the spike amount, calculation of percent recovery is typically difficult. If the sample contains no or low GRO in comparison to the spike amount, MS percent recovery should be within the QC limits.

12.5 Corrective Actions for Samples (also see section 10):

12.5.1 Hydrocarbon Concentration:

Corrective action involves diluting and re-analyzing samples if the concentration of hydrocarbons in the initial analysis exceeds the instrument calibration range. As GRO is a collection of many peaks, and the instrument is calibrated using selected individual peaks representing GRO, the height of the largest peak on the sample may not exceed the height of the largest component peak in the initial calibration.

12.5.2 Matrix Effect:

Samples will be re-analyzed for matrix effect if the percent recovery of BFB is outside the control limits listed above for samples. If both runs are out of control limits in a consistent fashion (both above or both below percent recovery, not one above and one below percent recovery), and if the percent recovery problem can not be determined by other factors (such as poor purging or degradation of the BFB surrogate), then both runs will be reported, and the matrix effect of the sample on the BFB recovery will be explained in the report narrative.

12.6 Any corrective action which must be performed for an Initial or Continuing Calibration, Method Blank, or Lab Control Sample as described in this document should be executed before other runs are performed, or the data for subsequent analyses are accepted.

Examples:

- If a continuing calibration does not pass acceptance criteria, then corrective action must be performed before any other samples are run.

- If an individual Initial Calibration level does not pass acceptance criteria, it may be run after other Initial Calibration levels, but it must be run before any other types of samples (Method Blanks, samples, etc.).

13. Health and Safety

All safety precautions as recommend by the instrument vendor should be followed. Analytical standards, samples and sample extracts may contain hazardous constituents and should be handled with care in a well-ventilated environment, and exposure to these compounds should be kept as low as reasonably possible.

14. Pollution Prevention, Waste Management, Acronyms and Definitions

See sections 19.0 and 20.0 of Mitkem's current Quality Assurance Plan.

15. References

Maine Health and Environmental Testing Laboratory. Modified GRO Method for Determining Gasoline Range Organics, Method 4.2.17, September 6th 1995.

U.S. Environmental Protection Agency. Nonhalogenated Organics Using Gas Chromatography/FID, Method 8015B, SW-846 Test Methods for Evaluating Solid Wastes, 3rd Edition, 1996.

U.S. Environmental Protection Agency. Nonhalogenated Organics Using Gas Chromatography/FID, Method 8015D, SW-846 Test Methods for Evaluating Solid Wastes on-line Edition, June 2003.

Attachments:

Attachment 1: A Component Standard chromatogram and integration report.
Attachment 2: A Gasoline Product Standard Chromatogram and integration report.
Attachment 3: Volatile Instrument Run log.

Attachment 1:

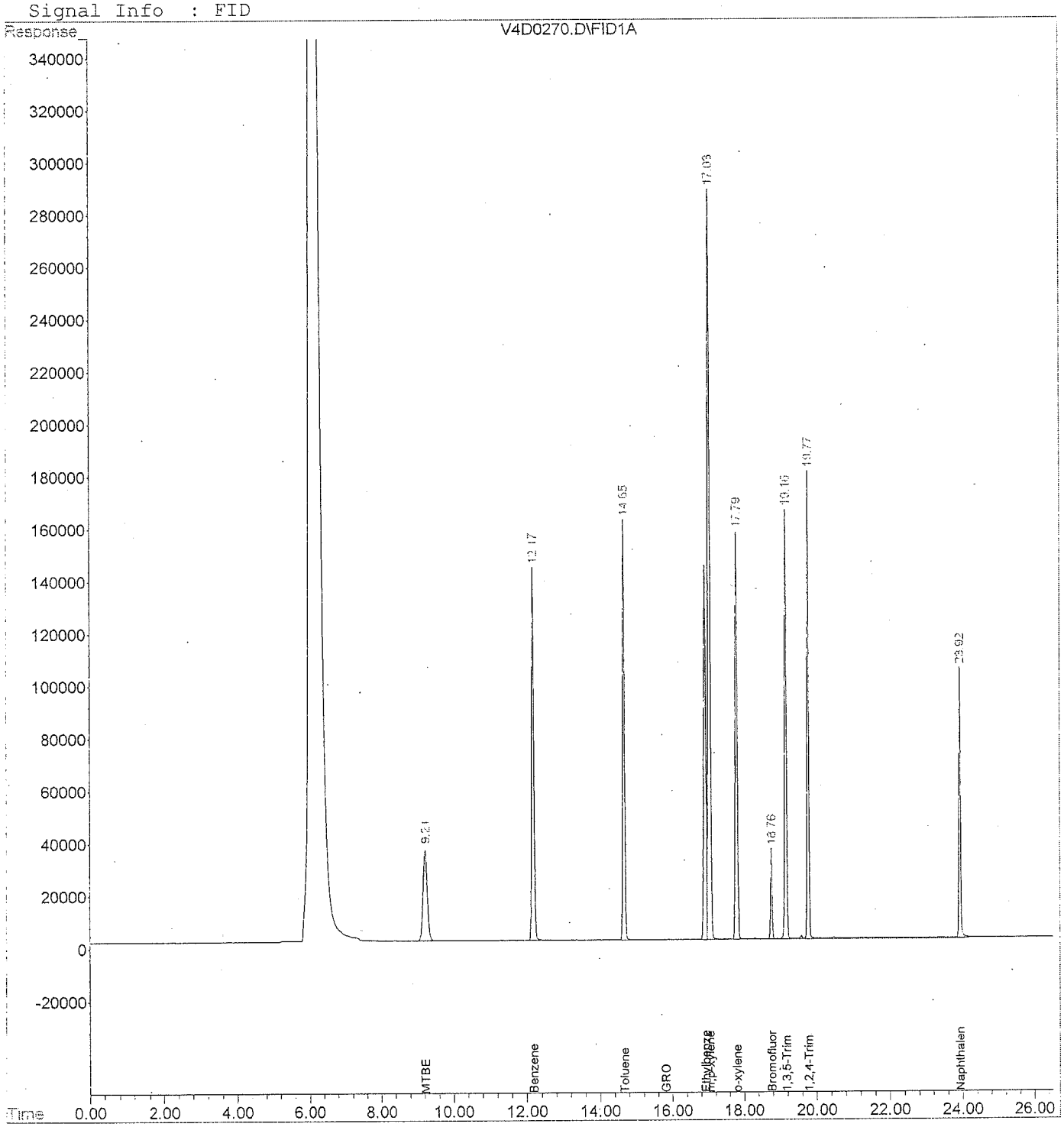
A Component Standard chromatogram and integration report

Quantitation Report (QT Reviewed)

Data File : O:\ORGANIC\VOA\V4.I\090119.B\V4D0270.D Vial: 40
Acq On : 20 Jan 2009 9:09 am Operator: HZA
Sample : 5ML,VSTD0504I,,, Inst : V4
Misc : AQ Multiplr: 1.00
IntFile : GRO.E
Quant Time: Jan 20 10:31 2009 Quant Results File: GRO_N1~1.RES

Quant Method : O:\V4.I\081009.B\GRO_N1~1.M (Chemstation Integrator)
Title : Method 8015 - Gasoline Range Organics
Last Update : Tue Dec 09 13:45:15 2008
Response via : Multiple Level Calibration
DataAcq Meth : V4GRO.M

Volume Inj. :
Signal Phase :
Signal Info : FID



Quantitation Report (QT Reviewed)

Data File : O:\ORGANIC\VOA\V4.I\090119.B\V4D0270.D Vial: 40
Acq On : 20 Jan 2009 9:09 am Operator: HZA
Sample : 5ML,VSTD0504I,,, Inst : V4
Misc : AQ Multiplr: 1.00
IntFile : GRO.E
Quant Time: Jan 20 10:31 2009 Quant Results File: GRO_N1~1.RES

Quant Method : O:\V4.I\081009.B\GRO_N1~1.M (Chemstation Integrator)
Title : Method 8015 - Gasoline Range Organics
Last Update : Tue Dec 09 13:45:15 2008
Response via : Initial Calibration
DataAcq Meth : V4GRO.M

Volume Inj. :
Signal Phase :
Signal Info : FID

Compound	R.T.	Response	Conc Units
System Monitoring Compounds			
7) S Bromofluorobenzene	18.76	1100844	20.112 ug/L
Spiked Amount 20.000 Range 87 - 112 Recovery = 100.56%			
Target Compounds			
1) T MTBE	9.22	2900118	52.280 ug/L
2) T Benzene	12.18	6025881	52.972 ug/L
3) T Toluene	14.66	5878367	52.757 ug/L
4) T Ethylbenzene	16.91	5656617	52.188 ug/L m
5) T m,p-xylene	17.03	11572373	105.810 ug/L m
6) T o-xylene	17.79	5898686	53.572 ug/L
8) T 1,3,5-Trimethylbenzene	19.16	5695742	51.989 ug/L
9) T 1,2,4-Trimethylbenzene	19.77	5763706	52.079 ug/L
10) T Naphthalene	23.92	3477622	52.259 ug/L
11) GRO	15.80	52869111	526.239 ug/L m

HZA 1/20/09

Attachment 2:

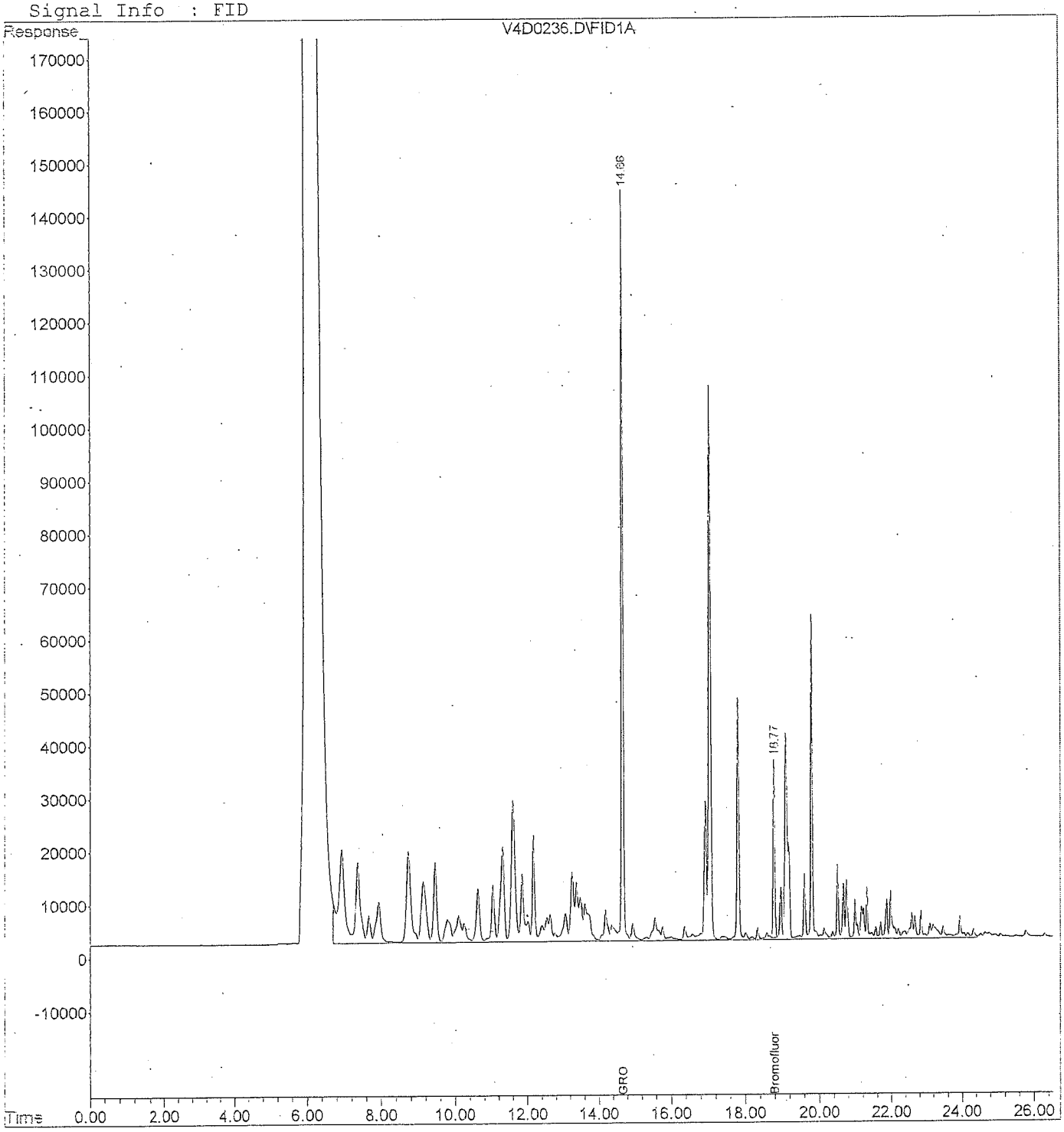
A Gasoline Product Standard Chromatogram and integration report

Quantitation Report (QT Reviewed)

Data File : O:\ORGANIC\VOA\V4.I\090119.B\V4D0236.D Vial: 6
Acq On : 19 Jan 2009 1:58 pm Operator: HZA
Sample : 5ML,LCSD-41374,V4GLCSD,41374, Inst : V4
Misc : AQ Multiplr: 1.00
IntFile : GRO.E
Quant Time: Jan 19 14:53 2009 Quant Results File: GRO_N1~1.RES

Quant Method : O:\V4.I\081009.B\GRO_N1~1.M (Chemstation Integrator)
Title : Method 8015 - Gasoline Range Organics
Last Update : Tue Dec 09 13:45:15 2008
Response via : Multiple Level Calibration
DataAcq Meth : V4GRO.M

Volume Inj. :
Signal Phase :
Signal Info : FID



Quantitation Report (QT Reviewed)

Data File : O:\ORGANIC\VOA\V4.I\090119.B\V4D0236.D Vial: 6
Acq On : 19 Jan 2009 1:58 pm Operator: HZA
Sample : 5ML,LCSD-41374,V4GLCSD,41374, Inst : V4
Misc : AQ Multiplr: 1.00
IntFile : GRO.E
Quant Time: Jan 19 14:53 2009 Quant Results File: GRO_N1~1.RES

Quant Method : O:\V4.I\081009.B\GRO_N1~1.M (Chemstation Integrator)
Title : Method 8015 - Gasoline Range Organics
Last Update : Tue Dec 09 13:45:15 2008
Response via : Initial Calibration
DataAcq Meth : V4GRO.M

Volume Inj. :
Signal Phase :
Signal Info : FID

	Compound	R.T.	Response	Conc	Units	
	System Monitoring Compounds					
7) S	Bromofluorobenzene	18.77	1074222	19.626	ug/L	m
Spiked Amount	20.000	Range 87 - 112	Recovery =	98.13%		
	Target Compounds					
1) T	MTBE	0.00	0	N.D.	ug/L	d
2) T	Benzene	0.00	0	N.D.	ug/L	d
3) T	Toluene	0.00	0	N.D.	ug/L	d
4) T	Ethylbenzene	0.00	0	N.D.	ug/L	d
5) T	m,p-xylene	0.00	0	N.D.	ug/L	d
6) T	o-xylene	0.00	0	N.D.	ug/L	d
8) T	1,3,5-Trimethylbenzene	0.00	0	N.D.	ug/L	d
9) T	1,2,4-Trimethylbenzene	0.00	0	N.D.	ug/L	d
10) T	Naphthalene	0.00	0	N.D.	ug/L	d
11)	GRO	14.66	46800463	465.834	ug/L	m

HZA 1/20/09

Attachment 3

Volatile Instrument Run log

DATE:

INSTRUMENT V4
INJECTION LOG

MITKEM LABORATORIES
VOLATILES LABORATORY

METHOD:__________________ CAL ID:______________ ANALYST:______________

INITIAL CAL:_______________ IS/SS ID:_____________ DATE:__________________

COMMENTS:

DATE PRINTED:____________________

DATE LOADED:____________________

AS #	FILE		MITKEM ID		CLIENT ID	SAMPLE SIZE	DIL	COMMENTS	IS	SS	pH

Logbook ID 90.0065-12/08

Mitkem Precision and Accuracy Limits (%)
PAHs by 8270 SIM - Solids
(Received at TtNUS 5/15/09)

AT	&Analyte	Low	High	RPD
A	2-Methylnaphthalene	45	135	40
A	Acenaphthene	45	135	40
A	Acenaphthylene	45	135	40
A	Anthracene	45	135	40
A	Benzo(a)anthracene	45	135	40
A	Benzo(a)pyrene	45	135	40
A	Benzo(b)fluoranthene	45	135	40
A	Benzo(g,h,i)perylene	45	135	40
A	Benzo(k)fluoranthene	45	135	40
A	Chrysene	45	135	40
A	Dibenzo(a,h)anthracene	45	135	40
A	Fluoranthene	45	135	40
A	Fluorene	45	135	40
A	Indeno(1,2,3-cd)pyrene	45	135	40
A	Naphthalene	45	135	40
A	Pyrene	45	135	40

APPENDIX F

COLUMBIA ANALYTICAL SERVICES
SELECTED STANDARD OPERATING PROCEDURES AND
QUALITY ASSURANCE PROGRAM MANUAL

SELECTED COLUMBIA ANALYTICAL SERVICES
STANDARD OPERATING PROCEDURES CONTENTS

SOP NUMBER	SUBJECT
EXT-3550B	Ultrasonic extraction
GEN-TOCLK/9060/TICLK	Total Organic Carbon or Total Inorganic Carbon in Soils
MET-200.7/6010B	Determination of Metals and Trace Elements by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP)
MET-3050B	Metals Digestion, Soils, Sediments, and Sludge for ICP-AES, ICP-MS, GFAA Analysis and ICP-MS Waters
MET-6020	Metals (EPA 6020)
MET-7471A/245.5	Mercury (EPA 7471A)
SOC-680	PCBs by GC/MS
SOC-8270C	Semivolatile Organic Compounds by GC/MS (EPA 8270C)

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

for

ULTRASONIC EXTRACTION

SOP No.: EXT-3550B

Revision: 2

April 3, 2002

Approved by: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Manager Date

© Columbia Analytical Services, Inc., 2002
One Mustard St., Suite 250
Rochester, NY 14609

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

UNCONTROLLED COPY

1. SCOPE AND APPLICATION

1.1. Method 3550B is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent.

1.2. The method is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of less than or equal to 20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The medium/ high concentration method (individual organic components of greater than 20 mg/kg) is much simpler and therefore faster.

1.3. It is highly recommended that the extracts be cleaned up prior to analysis. See CAS SOPs, EXT-3620B, EXT-3640A, EXT-3660B, or EXT-3665A.

2. SUMMARY OF METHOD

2.1. Low concentration method - A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using ultrasonic extraction. The extract is then concentrated and a portion of the concentrate is removed for cleanup and/or analysis.

2.2. Medium/ high concentration method - A 1, 2, or 5 g sample is mixed with sodium sulfate and solvent extracted one time using the micro-tip ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis.

3. DEFINITIONS

3.1. **Extraction batch** – a group of no more than 20 field samples extracted on the same day with the same reagents under the same conditions.

3.2. **Laboratory Control Sample (LCS)** - An aliquot of sodium sulfate to which a known quantity of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.3. **Matrix** - the predominant material, component, or substrate (e.g., soil, sludge, etc.) of which the sample to be analyzed is composed.

3.4. **Matrix Spike (MS/MSD)** - In the matrix spike analysis, a predetermined quantity of a standard solution of the analytes of interest is added to a sample matrix prior to sample extraction. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recovery is calculated for the analytes

detected to measure accuracy. The RPD between the MS and MSD is calculated to measure precision.

3.5. **Method Blank (MB)** – an artificial sample known to be free of the analytes of interest. Used to measure contamination introduced during extraction and analysis.

3.6. **Sample** - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

3.7. **Surrogates (Surrogate Standards)** - an organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process. For semivolatiles and pesticides/Aroclors, surrogate compounds are added to every blank, sample, matrix spike, matrix spike duplicate, LCS, matrix spike blank, and standard. These are used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

4. INTERFERENCES

4.1. 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.

4.2. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary. Refer to Method 3600 for guidance on cleanup procedures.

4.3. Phthalate esters contaminate may types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

4.4. Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, Aldrin, Heptachlor, and most organophosphorus pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500-ml K-D flask). These items should be hand-rinsed very carefully to avoid this problem.

5. SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. Concentrated sulfuric acid and the 50% sodium hydroxide solution are moderately toxic and extremely irritating to skin and mucous membranes. Use these

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reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1. Sample collection, preservation, and custody management is handled by the sample maintenance office. Refer to the SMO-GEN for specifics.

6.2. Purchased, precleaned, certified sample containers should be glass or Teflon, and have screw-caps with Teflon lined septa. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples shall be stored at 0-6°C and shipped to the laboratory within 48 hrs.

6.3. Soil samples must be iced or refrigerated at 0-6°C from the time of collection until extraction. Samples should be extracted within 14 days from collection using SW846 methodologies. Samples shall be extracted within 10 days from sample receipt for all CLP methods. Sample extracts shall be stored in extract coolers at 0-6°C and analyzed within 40 days of extraction. See specific analytical SOPs for holding time requirements.

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7. APPARATUS AND EQUIPMENT

7.1. Ultrasonic Disrupter - A horn type device equipped with a titanium tip, or a device that will give equivalent performance. The horn should be tuned prior to sample extraction. The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. Follow the manufacturers instructions for preparing the disrupter for extraction of samples with low and medium/high concentration. Use a 3/4" horn for the low concentration method and a 1/8" tapered microtip attached to a 1/2" horn for the medium/high concentration method.

7.2. Beakers - 400 ml.

7.3. Vacuum filtration apparatus.

7.3.1. Buchner funnel.

7.3.2. Filter paper - Whatman No. 41 or equivalent.

7.3.3. Side- Arm Flask - 1000 mL Pyrex.

7.4. Erlenmeyer flasks - 250 mL and 500 mL.

7.5. Kuderna-Danish (K-D) apparatus.

7.5.1. Concentrator tube - 10 ml, graduated (Kontes K-570050-1025 or equivalent).

7.5.2. Evaporation flask - 250 mL and 500 mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with clamps.

7.5.3. Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

7.5.4. Plastic Clips - 19/24 Joint (KT675300-0019 or equivalent).

7.6. Glass boiling beads - 3 mm glass beads.

7.7. Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

7.8. Vials and Caps - 20 mL scintillation vials with screw caps and aluminum foil liner, 2 mL capacity glass with crimp tops for GC auto sampler.

7.9. Pipets - glass volumetric 1 mL or 2 mL.

7.10. Eppendorf Repeater Plus with 5 mL and 10 mL tips

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7.11. 5 inch Pyrex funnel with a small pad of Pyrex glass wool.

7.12. Volumetric flasks - 10 mL and 1 and 2 mL

7.13. Balance - Top loading, capable of accurately weighing to the nearest 0.01g.

7.14. Tongue Depressors - 6 inch standard.

7.15. Nitrogen Evaporation Device - equipped with a heated bath that can be maintained at 35 to 40°C (N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent).

7.16. Oven - drying.

7.17. LabConco RapidVap Evaporation System

7.17.1. 600mL Sample Tubes – End point Stem 1.5mL
7.17.2. 8 place rack for 600ml Sample tubes

8. PREVENTIVE MAINTENANCE

8.1 Ultrasonic probes - Pitting of the probe tip will occur with use. When the bottom begins to cup, the probe needs to be sent out to be machined. Eventually, the probe will need to be replaced.

8.2 Bath / RapidVap temperature should be monitored. It is not to exceed 15-20°C of the boiling point of the solvent being evaporated.

9. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1. MB – extract sodium sulfate as a sample

9.2. Standards, LCS, MS/MSD, and surrogate spiking solutions - concentrations and recipes are discussed in the CAS analytical SOPs.

9.3. LCS/MS – spike sodium sulfate or a sample, as applicable, with the spiking solutions described in the Methods Summary Spreadsheet located in EXT-3510C.

9.4. Sodium sulfate - granular - anhydrous reagent grade, heated at 120°C for 16 hours, and stored in a glass bottle.

CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

9.5. Extraction solvent(s) - .Methylene chloride:Acetone (50:50) for 8081/8082 or Methylene Chloride for all others - pesticide quality or equivalent. It is recommended that each lot of

solvent used be analyzed to demonstrate that it is free of interference before use. Solvents are stored at room temperature and expire upon manufacturer's indications or 3 years from receipt, whichever is sooner. Boiling point 39°C.

9.6. Exchange solvent - .Hexane, - Pesticide quality or equivalent. Boiling point 68.7°C.

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of he results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

11. PROCEDURE

11.1. Sample handling

11.1.1. Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.1.2. Gummy, fibrous or clay-like materials should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling of these difficult matrices.

11.2. Extraction method for samples expected to contain low concentrations of organics and pesticides (≤ 20 mg/Kg):

11.2.1. The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample into a 400 ml beaker. Record the weigh to the nearest 0.1 g. Add 60 g of sodium sulfate and mix thoroughly. Nonporous or wet samples (gummy or clay type) that do not have a freeflowing sandy texture may be mixed with more sodium sulfate if needed. After addition of sodium sulfate, the sample should be free flowing. Add amount of surrogate standards specified in the analytical method, usually 1mL, to all samples, spikes, LCS, and blanks (see Method Summary Spreadsheet located with the extraction logs). For the sample in each analytical batch selected for QC, add the specified amount of matrix spike, usually 1mL (see Method Summary Spreadsheet). Immediately add 100 mL of extraction solvent.

11.2.2. Place the bottom surface of the tip of the disrupter horn about 1/2 in. below the surface of the solvent, but above the sediment layer.

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11.2.3. Extract ultrasonically for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50%. Do not use microtip probe.

11.2.4. Decant and filter extracts through a Buchner funnel, using Whatman No. 41 filter paper and vacuum filtration, into a sidearm flask.

11.2.5. Repeat the extraction two more times with two additional 100 ml portions of solvent. On the final ultrasonic extraction, pour the entire sample into the Buchner funnel and rinse with extraction solvent. Keep the solvent extract and discard the sample on the filter.

11.3. Dry and Concentrate Extract:

11.3.1. K-D Concentrator:

11.3.1.1. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporation flask. Pour the extract into the K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20-30 ml of Methylene chloride and add it to the K-D concentrator flask.

11.3.1.2. Add one or two clean glass boiling beads to the flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1 ml of Methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-100°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 5-7 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

NOTE: If a solvent exchange is required, pour 50 mLs of the exchange solvent down the Snyder column and concentrate the extract, raising the temperature of the water bath, if necessary, to maintain proper distillation.

11.3.1.3. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of Methylene chloride or exchange solvent. The extract may be further concentrated by using the technique outlined in the next section or adjusted to 10.0 mL with the solvent last used.

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11.3.2. If further concentration is needed, a nitrogen blow-down technique is used to adjust the extract to the final volume required.

11.3.2.1. Place the concentrator tube in a warm bath (35-40°C) and evaporate the solvent volume to the appropriate final volume using nitrogen gas. The internal wall of the tube must be rinsed down several times with Methylene chloride or appropriate solvent during the operation.

CAUTION: Avoid concentrating the sample to less than 1 ml. When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

11.3.2.2.The final volume is transferred to a vial with a Teflon lined screw-cap or crimp top, and label appropriately. The extract may now be analyzed for the target analytes using the appropriate determinative technique. Store extract in extract cooler at temperature of 0-6°C.

11.3.3. LabConco RapidVap Nitrogen Evaporation System

11.3.3.1.Add extract to a 600 mL Sample tube

11.3.3.2.Operation

11.3.3.2.1.Turn on power switch

11.3.3.2.2.Set parameters – Input the % Speed, Temperature, Time and Number of Samples to be concentrated. (See users manual for Time, Temperature and Speed Setting Guidelines per volume per solvent.)

11.3.3.2.3.Turn on Nitrogen gas.

11.3.3.2.4.Load sample tubes into RapidVap chamber and place Teflon cap on sample tube. Close lid and swing both latched over the lid and tighten knobs.

11.3.3.2.5.Press Run to begin concentration. Be sure to check the samples at the beginning of the program to ensure samples are not rotating so quickly that they are splashing out of the sample tube. If this occurs press the Stop button and adjust the speed. To resume press Run.

11.3.3.2.6.Allow samples to evaporate to a final volume of ~ 1 mL. When the time in the program is complete the instrument will beep and stop. Check sample volume. If additional time is needed reset program and press Run. Once desired final volume is reached the extract may be further concentrated by using the techniques outlined previously

(see 11.3.2) or adjusted to final volume (Table 1) with the solvent last used.

11.4. Extraction method for samples expected to contain medium/high concentrations of organics (>20 mg/kg). Used primarily for PCB analysis.

11.4.1. Weigh sample (typically 1, 2 or 5 g) into a 40 mL vial. Add an equal amount of sodium sulfate and mix thoroughly.

11.4.2. Add appropriate amounts of surrogate spike solution and matrix spike solution (see Method Summary Spreadsheet). Bring to a final volume of 10 mLs for 1 or 2 g samples, or 20 mLs for 5 g samples. For solvents use Methylene chloride for semi-volatile GC/MS analysis and hexane for pesticide/PCB GC/ECD analysis.

11.4.3. Using the micro-tip disrupter horn, place sonicator probe about 1/2 inch below the surface of the solvent, but above the sediment layer.

11.4.4. Extract ultrasonically for 2 minutes with output control knob set at 5 and with mode switch on pulse (pulsing energy rather than continuous energy) and percent - duty cycle knob set at 50%.

Note 1: When using a micro-tip do not allow it to vibrate in air for more than 15 seconds and do not set the output control above "5". Ignoring these instructions will cause the micro-tip to fracture.

Note 2: If the microtip extraction does not work because of sample matrix, hand shake for 2 minutes.

11.5. At this point samples are ready to transfer to vials and do further cleanup procedures or may be analyzed for the target analytes using the appropriate determinative technique. No concentration is required for the medium/high extraction.

12. QA/QC REQUIREMENTS

12.1. All blanks, LCS, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples. QC criteria are specified in the appropriate analytical methods.

12.2. The results of the extracted QC samples will be evaluated at the time of analysis according to the determinative SOPs. QC limits are given in Appendix C of the Quality Assurance Manual.

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13. DATA REDUCTION AND REPORTING

13.1. Be sure all documentation is complete and legible.

13.2. All benchsheets shall be maintained in the Extraction log binder for reference. On a routine basis, the benchsheets are bound and archived.

13.3. Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

13.4. All standards must be traceable using the CAS lot system, see ADM-DATENTRY.

14. METHOD PERFORMANCE

Reporting limits are based upon MDL studies performed according to ADM-MDL and filed in the MDL binders in the QA Office.

15. WASTE MANAGEMENT AND POLLUTION PREVENTION

Standards and reagents are prepared on an as-needed basis in small quantities and minimum sample volumes are used during analysis. It is the laboratory's responsibility to comply with all federal, state and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. Excess unused sample and testing byproducts are disposed following the procedures in SMO-SPLDIS.

16. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18. REFERENCES

"Test Methods For Evaluating Solid Waste Physical/Chemical Methods," USEPA SW-846, December 1996.

19. TRAINING OUTLINE

19.1 Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2 Observe Sample Preparation. Follow Extraction Training Plan Form.

19.3 Participate in the methodology and documentation with guidance.

19.4 Perform an IDC (Initial Demonstration of Capability) by independently extracting four mid-range standards prior to analyzing client samples. If recovery is within acceptable limits, complete Training Plan Form, IDC certificate, and summary spreadsheet. File with QA. Continuing demonstration of capability (CDC) will be performed annually using a PE sample, LCS, single blind, or new 4 replicate study.

20. METHOD MODIFICATIONS

None

21. INSTRUMENT-SPECIFIC ADDENDUM

None

22. ATTACHMENTS

- Extraction Benchsheet
- Table 1 Specific Extraction Conditions
- Flow Chart of Extraction

23. CHANGES FROM PREVIOUS REVISION

- Added sections 14, 16, 17, and 20 for NELAP compliance
- Modified batch, MS/MSD, LCS, and MB to definitions (section 3)
- Changed all references to 4±2 to 0-6 °C
- Added need to analyze extracts within 40 days of extraction
- Added reference to SMO-GEN, ADM-DREV, and ADM-TRANDOC
- Changed Training Outline to reflect current practice
- Added RapidVap, Sample Tubes, and rack to Equipment (7)
- Added need to monitor bath/RapidVap temps in Preventive Maintenance (8)
- Added MB, LCS, MS/MSD, surrogate solution to Standards and Reagents (9)
- Added storage and expiration to reagents (9)

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- Added surrogate and spike amount of, “usually 1mL” to Section 11.2.1
- Added RapidVap to procedure in 11.3
- Modified QA/QC REQUIRMENTS Section 12
- Added 12.2 statement referencing determinative SOPs and QAM for QC discussion
- Modified Section 13 , Added 13.2 – Benchsheets are maintained in a binder
- Added attachments in Section 22.

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Example Extraction Benchsheet

Extraction Tech: _____ Extraction Date: _____ Concentration Tech: _____ 40 Day HT: _____	Spiked By: _____ Spk Witness: _____	Prep Method: ☐ 3540 ☐ 3550 ☐ 3510 ☐ 3520 ☐ 3580	Key: Color: C = Colorless; Y = Yellow; B = Brown; BL = Black; G = Grey Clarity: CLR = Clear; CDY = Cloudy; OP = Opaque Soils: F = Fine/Sand; M = Medium/Soil; C = Coarse/Rocks	Batch ID:

Client / Sub. #	Sample ID	Initial Wt. (g) or Volume (ml)	Appearance (see key)	Analysis (Test) Requested	pH REC'D pH	pH Check (water only) BN > 11 1	BN > 11 2	Acid < 2 1	Acid < 2 2	Conc. Date	Final Volume (ml)	Date Complete	Comments / Emulsions

Spikes:

☐	AE/BN Surrogate	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	BN Surrogate	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	680 Surrogate	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	95-2 Surrogate	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	8270 LCS MIX 1	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	95-2 Spike	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	TANK List Spike	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	Phthalate Spike	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	680 Spike	*Amt.* ____ml	*Conc.* ____ppm	*Lot#*_____
☐	Other: _____	*Amt.*____*ml; Conc.*____*ppm; Lot#*_____		

Clean-Ups: ☐ None

☐	3620 Florisil	*By/Date*_____	*Lot#*_____
☐	3640 GPC	*By/Date*_____	*Lot#*_____
☐	3660 Cu/TBA	*By/Date*_____	*Lot#*_____
☐	3665 Acid	*By/Date*_____	*Lot#*_____

Method Summary:
Continuous Liquid-Liquid @ pH < 2 with 300-500mls MeCl2 for 18 hours.
Start Time: _____ **End Time:** _____

Solvents:

☐	50:50 Ace:MeCl2 Lot # _____	☐	Hexane Lot# _____	☐	Sulfuric Acid Lot#:_____
☐	MeCl2 Lot # _____	☐	Ether Lot# _____	☐	Sodium Hydroxide Lot#:_____
☐	Acetone Lot #	☐	Sodium Sulfate Lot#	☐	Other: Lot#:_____

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TABLE 1

SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Initial extraction pH	Secondary extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)
8041	≤2	none	2-propanol	hexane	1.0
8061	5-7	none	hexane	hexane	2.0
8070	as received	none	methanol	CH_2Cl_2	2.0
8081	5-9	none	hexane	hexane	10.0
8082	5-9	none	hexane	hexane	10.0
8091	5-9	none	hexane	hexane	2.0
8100	as received	none	none	cyclohexane	2.0
8111	as received	none	hexane	hexane	2.0
8121	as received	none	hexane	hexane	2.0
8141	as received	none	hexane	hexane	10.0
8270[c,d]	<2	>11	none	-	-
8310	as received	none	acetonitrile	-	-
8321	as received	none	methanol	-	-
8325	7.0	none	methanol	-	-
8410	as received	none	CH_2Cl_2	CH_2Cl_2	10.0

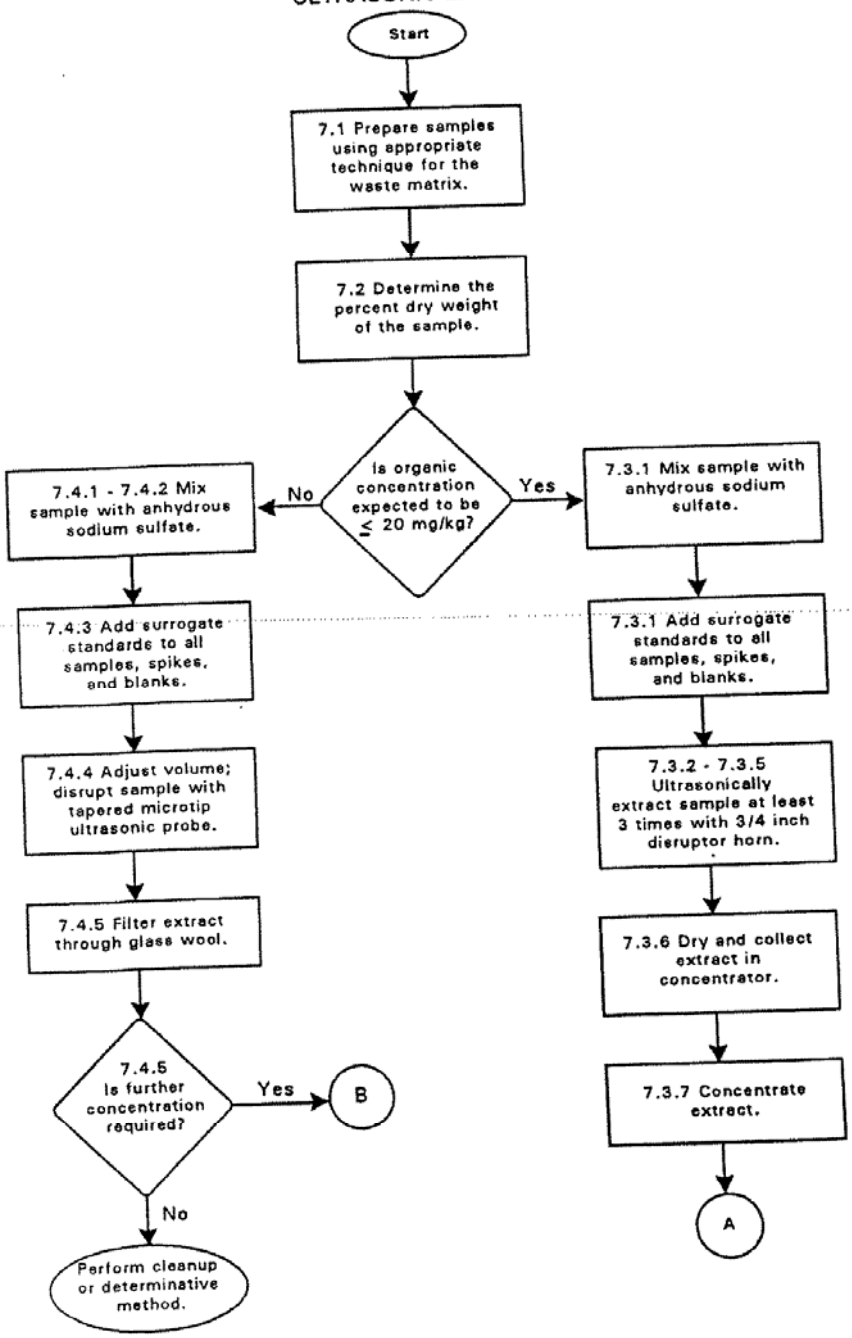
[a] For methods where the suggested final extract volume is 10.0 mL, the volume may be reduced to as low as 1.0 mL to achieve lower detection limits.

[b] Phenols may be analyzed, by Method 8041, using a 1.0 mL 2-propanol extract by GC/FID. Method 8041 also contains an optionalderivation procedure for phenols which results in a 0.5 mL hexane extract to be analyzed by GC/ECD.

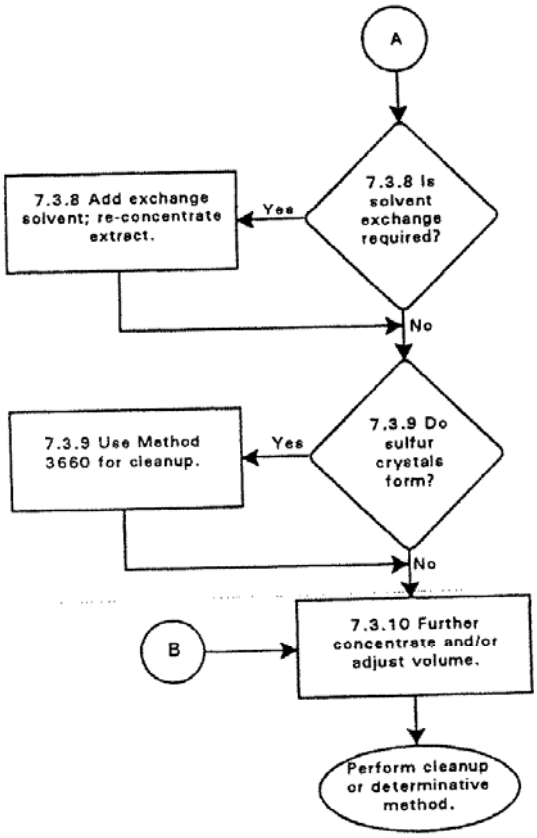
[c] The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available of required.

[d] Extraction pH sequence may be reversed to better separate acid and neutral waste components. Excessive pH adjustments may result in the loss of some analytes.

METHOD 3550B
ULTRASONIC EXTRACTION



METHOD 3550B
continued



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STANDARD OPERATING PROCEDURE

TOTAL ORGANIC CARBON or TOTAL INORGANIC CARBON IN SOILS

GEN-TOCLK/9060/TICLK

Revision 2

January 17, 2005

Approved By: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Manager Date

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One Mustard St., Suite 250
Rochester, NY 14609

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

1. SCOPE AND APPLICATION

*See DoD Summary for DoD Specific Criteria

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1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) in soils and sludges using the EPA document "Determination of Total Organic Carbon in Sediment" prepared by Lloyd Kahn (EPA 1988) or EPA SW846 9060. Total Inorganic Carbon (TIC) may be calculated by subtraction of TOC from Total Carbon(TC).

1.2. The current system will measure up to about 1500 ug/g C when the smallest sample volume is used. When a range extender is used on the instrument, the range is extended to about 8000 ug/g C.

1.3. Normal operating parameters yield a Practical Quantitation Limit (PQL) of 300 mg/Kg. Equivalent nomenclature for PQL includes Estimated Quantitation Limit (EQL) and Method Reporting Limit (MRL).

1.4. Data are reported in mg/kg on a dry weight basis.

2. METHOD SUMMARY

2.1. Treatment prior to analysis of carbon

2.1.1. For TOC - Inorganic Carbon from carbonates and bicarbonates is removed by treatment with acid.

2.1.2. For TC - Inorganic Carbon from carbonates and bicarbonates is NOT removed by treatment with acid.

2.1.3. For TIC - The samples are put through the process twice – once to measure Total Carbon (TC), and once to measure Total Organic Carbon (TOC). The Total Inorganic Carbon (TIC) is calculated by difference.

2.2. The carbon compounds are decomposed into carbon dioxide and other gases by pyrolysis at 800 °C in the presence of air. The interfering gases are removed by a sparger/scrubber system. The carbon dioxide that is formed is determined by direct nondispersive infrared detection that has been calibrated to directly display the mass of carbon dioxide detected. The resulting carbon mass in the form of carbon dioxide is the equivalent to the mass of carbon (TOC or TC) originally in the sample.

3. DEFINITIONS

3.1. **Initial Calibration -** analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the system.

3.2. **QA/QC Samples**: Samples added to a sample preparation batch, or an analytical batch to provide quality assurance checks on the analysis.

3.2.1. **Matrix Spike (MS)** - In the matrix spike analysis, a predetermined quantity of

*See DoD Summary for DoD Specific Criteria

standard solution is added to a sample matrix prior to sample analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for the analyte detected.

3.2.2. **Duplicate Sample (DUP)** - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.2.3. **Laboratory Control Standard (LCS)** - In the LCS or blank spike analysis, predetermined quantities of standard solutions of certain analytes are added to a blank prior to sample analysis. Percent recoveries are calculated for the analyte detected and used to verify the linear range daily.

3.3. **Relative Percent Difference (RPD)** – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.4. Batch - Samples processed together as a unit, not to exceed 20 investigative samples. See ADM-BATCH for further discussion.

3.5. **Independent Calibration Verification (ICV)** - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.6. **Continuing Calibration Verification Standard (CCV)** - A reference analyzed daily at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.7. **Instrument Blank (ICB/CCB)** - The instrument blank (also called initial or continuing calibration blank) is a volume of blank reagent of composition identical to the samples (DI in this test). The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

3.8. **Method Detection Limit (MDL):** a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero

3.9. **Method Reporting Limit (MRL):** The minimum amount of a target analyte that can be measured and reported quantitatively. The MRL is equivalent to Practical Quantitation Level (PQL) and Estimated Quantitation Level (EQL). Typically, the MRL is calculated as five times the MDL (although this is a rule of thumb and not intended to be a strict policy of establishing the MRL for a compound).

4. INTERFERENCES

Volatile organics in sediments may be lost in the decarbonation step resulting in low bias.

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Minimize loss of organic compounds by bacterial decomposition and/or volatilization by maintaining the sample at 0-6 °C.

Contamination by carryover can occur when high level samples immediately precede samples containing significantly lower levels of contamination. Pay close attention to samples which follow high level samples. Re-analyze if contamination is suspected.

5. SAFETY

- All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

- Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

- Nitric Acid is used in this method. This acid is extremely corrosive and care must be taken while handling it. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

- The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

- Refer to the Safety Manual for further discussion of general safety procedures and information. Always wear chemical eye, skin, and clothes protection when handling samples or working with reagents.

6. SAMPLE CONTAINERS, COLLECTION, PRESERVATIONS, AND STORAGE

6.1. Samples are collected in purchased certified clean containers free of organic contaminants. Glass is preferable.

6.2. Because of the possibility of oxidation or bacterial decomposition of certain components, the samples should be kept cool (0-6°C) and protected from sunlight and atmospheric oxygen from time of collection to analysis. Holding time for samples is 14 days.

6.3. Further sample handling, storage, and custody procedures are discussed in SMO-GEN.

7. APPARATUS AND EQUIPMENT

7.1. Model DC-190 Total Organic Carbon Analyzer with Boat Module. (Dohrmann)

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7.2. Combustion boat: Platinum.

7.3. The linear range extender (Dohrmann part # 885-462) is used for high level samples. It consists of an expansion chamber attached to the 200 ml/min air line from the furnace/sparger and to a 400 ml/min line of air that acts to dilute the CO_2 gas in the standards and samples by a factor of 1/3.

7.4. Volumetric pipets/glassware and/or adjustable micro-pipets.

7.5. Printer

7.6. Drying oven capable of maintaining 70 ± 2 °C.

7.7. Drying tins

7.8. Small glass test tubes

8. PREVENTIVE MAINTENANCE

8.1. For the most reliable performance of the instrument, the following schedule of routine maintenance is suggested:

8.1.1. Daily:

- Condition the boat: Place fresh quartz wool plug and a few drops of HNO_3 in the Pt boat. All boats should be "conditioned" prior to any runs in order to remove any CO_2 that may have built up on the boat overnight. Place the boat in the hatch. Close the hatch (the sparger will bubble if the hatch is closed properly. Always watch for this every time the hatch is closed.) Advance the Pt Boat into the furnace. Allow it to bake for ~2-3 minutes, so that any carbonaceous material on the boat is removed. Retract the Pt Boat into the hatch block, allow to cool at least 30 seconds. Do this for all boats.

- Check the Cu/Sn scrubber. If dissolved or clogged, replace with fresh Cu and/or Sn.

8.1.2. Weekly:

- Check gas cylinder – replace when necessary
- Adjust IR "zero" if necessary
- Leak check the carrier gases
- Replace the sparger DI with UPDI acidified with phosphoric acid to pH <2

8.1.3. Every 2 Weeks or Earlier:

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Check the Boat Module's combustion tube. Replace the cobalt oxide catalyst if necessary. If the combustion tube becomes etched/"cloudy" looking, consider replacing it. The etching is produced by interfering gases in samples, and can cause a "slowdown" of movement of the carrier gas through the tube, resulting in "time out errors" during detection.

8.2. Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

9. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1. Reagent (laboratory deionized) water. If DI appears to be a source of contamination, use DI that has been run through the Millipore system in the VOA lab. It is suggested to occasionally make reagents with this Millipore water and analyze the tap DI at the low level to determine if it has a value above 0.1 mg/L.

9.2. Phosphoric Acid: reagent grade, purchased commercially. Store at room temperature. Expires upon manufacturer's indications or 3 years from receipt if no other indication is given.

9.3. Cobalt oxide catalyst: Place about 2 inches of cobalt oxide in the combustion tube with both ends bound by a tuft of quartz wool. Condition fresh cobalt oxide by placing a piece of tubing from the exit of the tube in dilute NaOH for at lease 1 hour with the air and furnace on.

9.4. Nitric Acid, concentrated: Purchased commercially. Store at room temperature. Expires upon manufacturer's indications or 3 years from receipt if no other indication is given.

9.5. 1+1 Nitric Acid (HNO_3): Add a volume of DI to an equal volume of concentrated nitric acid. Store at room temperature. Expires in one year.

9.6. Sparger solution: Fill the sparger with UPDI from a squirt bottle. Acidify the water with a couple drops of concentrated phosphoric acid to pH 2.

9.7. Gas Service: Compressed Air.

9.8. **<u>Standards Preparation General Information and Disclaimers</u>**

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 ppb standard may be made by

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adding 1 uL of 200 ppb to 10 mLs or may be made by adding 4 uL of 50 ppb to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to detection levels needed. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

9.9. Organic Carbon stock solutions:

9.9.1. Standard Stock Solution (10,000 ppm C): Stock solution is prepared by adding 10.64 g of Potassium Biphthalate (KHP) (previously dried to a constant weight at 104°C) into a 500 mL volumetric flask. Dilute to volume with reagent water. Store in amber glass at room temperature for up to 1 year.

9.9.2. Reference Stock Solution (10,000 ppm C): Stock solution is prepared by adding 10.64 g of KHP (previously dried to a constant weight at 104°C and from a different manufacturer) into a 500 mL volumetric flask. Dilute to volume with reagent water. Store in amber glass at room temperature for up to 1 year.

9.10. Inorganic Carbon Stock Solution (10,000 mg/L inorganic C): Stock solution is prepared by adding 8.824 g of Sodium Carbonate (previously dried to a constant weight at 104°C and from a different manufacturer) into a 100 mL volumetric flask. Dilute to volume with reagent water. Store in amber glass at room temperature for up to 1 year.

9.11. ICV/CCV: Add 50 uL of 10,000 mg/L Reference Stock to 100 mg of Ottawa Sand. True Value = 5000 ug/g.

9.12. LCS for TC or TOC: Add 20 uL of 10,000 mg/L Standard Stock to 100 mg of Ottawa Sand. True Value = 2000 ug/g

9.13. LCS for TIC – Add 20 uL of 10,000 mg/L Inorganic Carbon Stock to 100 mg of Ottawa Sand. True Value = 2000 ug/g.

9.14. Matrix Spikes: Add 20 uL of 10,000 mg/L Standard Stock to a similar mass of sample which was used for the sample analysis. TV = 2000 ug/g when 100 mg of sample is used. Prepare fresh before use.

9.15. ICB/CCB/Method Blanks – analyze 100mg of Ottawa Sand as a sample

9.16. Calibration Standards

***Note: When using an adjustable pipet, record the pipet ID of the pipet used.

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9.16.1. Prepare Single Point Calibration Standard by adding 50 uL of 10,000 mg/L Organic Standard Stock to 100 mg Ottawa Sand.

9.16.2. Prepare a 1000 ppm working stock for the less concentrated check standards – Dilute 1.0 mL of 10,000 ppm Standard Stock to 10 mLs with reagent water. Prepare fresh before use.

9.16.3. Prepare curve check standards as follows:

uL 10,000 ppm Standard Stock	Weight (mg)	Final Conc. (ug/g)
80	100	8000
50	100	5000
30	100	3000
100 uL 1000 ppm working stock	100	1000
50 uL 1000 ppm working stock	100	500
30 uL 1000 ppm working stock	100	300

** Note – These (above) curve check standards are to verify the instrument response only. They are not used to calculate sample results

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

11. PROCEDURE

11.1. Be sure the analyst has a current Demonstration of Capability and the system has a current MDL.

11.2. Turn on the air gas flow and confirm 30 psi delivery pressure. Maintain this delivery pressure.

11.3. **Initial Power Up**:

11.3.1. Turn on:

- The 70 °C oven
- The Boat Module Furnace
- The DC-190 Furnace and Carrier Gas. Confirm 200 mL/min readout. Even though soils and the Boat Module do not need the DC-190 Furnace and carrier gas on, the instrument software does.
- Allow time for all to come to correct temperature. The LED's for each

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will turn from red to green when ready.

11.3.2. Confirm that the desired analysis mode is properly selected. The TC and BOAT LED's on the DC-190 should be lit.

11.3.3. Condition the boat.

11.4. Initial Calibration

11.4.1. Prior to calibrating, reset the software to original conditions by pressing "Calibrate", 7, 1. Press "Yes" when prompted. This will reset the calibration factor to 1.0. Be sure to then make sure that the sample size (50 uL) and concentration (10,000) of the calibration stock standard are entered correctly before starting a new calibration. Press "Boat" then "1" to change ug/g to mg/L. Press "3" to print.

11.4.2. Standard Analysis - A single point calibration technique is used because the infrared detector response has been linearized. It is then verified with the other standards. The single-point calibration standard is run in duplicate, at least.

11.4.2.1. Weigh 100 mg of Ottawa Sand into the Pt Boat.

11.4.2.2. Add 1+1 HNO_3 dropwise until no effervescence is visible.

11.4.2.3. Place the boat in the oven at 70-75 °C for 10-15 minutes.

11.4.2.4. Remove the boat from the oven, place it in the hatch. Add 50 uL of the 10,000 mg/L Standard Stock to the PT Boat. Close the hatch (the sparger will bubble if the hatch is closed properly. Always watch for this every time the hatch is closed.)

11.4.2.5. Press the START button on the DC-190. Enter the "sample ID." The "Wait" LED will light momentarily while the instrument checks the baseline.

11.4.2.6. When the "Inject" LED lights, advance the boat into the furnace at the rate of about 2 inches per second.

11.4.2.7. The "Acquire" light will turn on. The analysis will take about 6-8 minutes to finish, at which point the printer will print out the result, and the screen will ask "Continue Y/N?" Press the "Yes" button to do another.

11.4.2.8. Repeat the Standard Analysis steps for the replicate standard. If a third replicate is desired, press "Yes" at the end of the analysis. (If an outlier needs to be removed from the calibration, see page 6-26 of the DC-190 Ops Manual. At least 2 replicates must be run for the initial calibration.) Press "No" to end the initial calibration.

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11.4.3. Software Calibration

11.4.3.1. Press "Calibrate" to review the calibration menu. Verify that the sample size and standard concentration are correct.

11.4.3.2. Press "5" to update the Calibration Factor. This will be calculated, displayed on the menu, and printed out.

11.4.4. If necessary, set instrument blank - Perform the standard analysis steps except adding 50 uL of UPDI instead of standard. After 2 replicates of the blank, press "No", "Calibrate" and "6" to update the system blank.

11.4.5. Change the concentration units from mg/L to ug/g by pressing "Boat" then "1".

11.5. Run the curve check standards to verify linear range. The instrument calculated results of the curve check standards should be within 10% of the true value and 20% for the lowest curve check standard. If they are not, repeat the calibration

11.6. Run the ICV, ICB, and a LCS to verify the curve. See Section 12 for Limits and Corrective Action.

11.7. **Daily Sample Analysis and Continuing Calibration Verification**

The calibration is confirmed with every run by the CCV, CCB, and LCS. Daily, every run is started with a CCV, CCB, and LCS and is closed with a CCV, CCB, as well as a CCV/CCB set every 10 samples and an LCS every 20. The instrument only needs to be recalibrated when these QC become unacceptable, or at least once a year.

11.7.1. Complete the initial power up steps

11.7.2. Condition Pt boat.

11.7.3. Sample preparation

11.7.3.1. Thoroughly mix sample and place a small aliquot (~2 g) in a drying tin.

11.7.3.2. Allow to dry overnight in a hood at room temperature or dry at 70 ± 2ºC for one hour.

11.7.3.3. Grind the sample with the bottom of a small glass test tube to break up aggregates.

11.7.3.4. Discard rocks.

11.7.3.5. Weigh 100 mg (or less depending on anticipated concentration) of sample

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or Ottawa Sand into the Pt Boat.

11.7.3.6. For TOC - Add 1+1 HNO_3 dropwise until no effervescence is visible. For TIC, separate aliquots of the sample will be analyzed – one with the acid added (TOC) and one with no acid added (TC). TIC will be the difference between the two results.

11.7.3.7. If acid has been added, place the boat in the oven at 70-75 °C for 10-15 minutes. Remove the boat from the oven.

11.7.4. Sample analysis

11.7.4.1. Spike at this time if needed.

11.7.4.2. The system is set up using the "Range Extender". This setup creates back-pressure. The release of pressure when the sample hatch is opened can cause the carrier gas to back-flow through the mist-trap and into the sparger, thus forcing the sparge liquid into the combustion tube/cobalt oxide. To prevent this, the following procedure should be followed:

11.7.4.2.1. **Before Opening the Hatch**: remove one end of the line from the sparger to the mist-trap.

11.7.4.2.2. Open the hatch and insert the boat. Close the hatch.

11.7.4.2.3. **After Closing the Hatch**: replace the end of the line from the sparger to the mist-trap. The sparger will bubble if the hatch is closed properly. Always watch for this every time the hatch is closed.

11.7.4.3. Press the START button on the DC-190. Enter the "sample ID" and the mg of sample. The "Wait" LED will light momentarily while the instrument checks the baseline.

11.7.4.4. When the "Inject" LED lights, advance the boat into the furnace at the rate of about 2 inches per second.

11.7.4.5. The "Acquire" light will turn on. The analysis will take about 6-8 minutes to finish, at which point the printer will print out the result, and the screen will ask "Continue Y/N?" Press the "No" button to analyze another sample.

11.7.4.6. Samples analyzed by 9060 are to be analyzed in quadruplicate. Lloyd Kahn samples are analyzed only once. Samples for TIC are analyzed once for TOC (with acid) and once for TC (without acid).

12. QA/QC REQUIREMENTS

*See DoD Summary for DoD Specific Criteria

12.1. QC Samples and Acceptance Criteria

12.1.1. LCS for TOC - An LCS must be analyzed with each batch of 20 or fewer samples. Results must be within the limits in Appendix C of the Quality Assurance Manual.

12.1.2. LCS for TIC - Analyze the inorganic carbon LCS per batch. Results should be within 75-125% of the true value until enough points are available that calculated limits may be established.

12.1.3. ICV-Analze immediately after calibration. Result must be 85-115% of the true value. If not, recalibrate unless there is an assignable cause for the ICV failure. Acceptable ICV must be achieved before sample analysis.

12.1.4. A CCV must be analyzed following every tenth injection and at the end of the run. Recovery must be within 15% of the value (85-115%).

12.1.5. A CCB must be analyzed following every CCV. The result must be below the PQL*.

12.1.6. Duplicates- One sample per 20 samples, must be analyzed in duplicate. If one or both of sample or duplicate is < 5X RL, the control limit is ± RL.

12.1.7. Matrix Spikes- One spike sample must be analyzed per 20 samples. The matrix spike recovery must within the limits in Appendix C of the Quality Assurance Manual.

12.1.8. Matrix Spike Duplicates – One sample for every 10 samples must be spiked and analyzed in duplicate for 9060. The % recovery limits and corrective action are the same as the matrix spike and the RPD limits and corrective action are the same as the duplicate.

12.2. Corrective Action

12.2.1. CCV, CCB, LCS

12.2.1.1. Check for gas leaks, sufficient reagent, and other instrument problems.
12.2.1.2. Remake working standard and reanalyze.
12.2.1.3. Recalibrate if QC is still out of control.
12.2.1.4. When instrument is back in control, reanalyze samples bound by out of control QC.

12.2.2. Duplicates

12.2.2.1. Check for gas leaks, sufficient reagent, and other instrument problems.

12.2.2.2. If the RPD of the duplicates is out of limits, repeat the sample and

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duplicate unless there is assignable matrix interference, historical failures, or lack of volume. If an out of control duplicate is not repeated, note the reason on the data quality checklist. If, at the time when the problem is discovered, the sample exceeds twice the holding time, discuss with supervisor or Project Manager prior to repeating the samples. Report all of the replicates and explain in the checklist for the case narrative.

12.2.3. Matrix spike

12.2.3.1. Check for gas leaks, sufficient reagent, and other instrument problems

12.2.3.2. If there are no such problems and recovery of the MS fails on a sample, the batch is considered acceptable as long as the LCS meets acceptance criteria. It is recommended that the MS/MSD be reprepared and reanalyzed to confirm the outliers, however it is not required due to probable matrix interferences.

13. DATA REDUCTION AND REPORTING

13.1. Calculations used by the DC-190 system

13.1.1. The equations used internally for determining a calibrated result:

$$Y = (Fx - b)/V$$

Where:
- Y = Concentration (calibrated) of sample
- x = Peak with background subtracted. Normally invisible to the user. The displayed value, y, may be made to equal x by setting F,b, and V to the appropriate values (1, 0, and 1, respectively)
- F = "Calibration Factor". This is the slope of the linear fit line.
- b = Intercept. This is an internal parameter which is invisible to the user.
- SB = "System" Blank = b/V
- V = Sample volume (or mass)

The quantities F and SB are the ones displayed on the calibration menu and are the ones which can be edited directly.

13.1.2. The Calibration Factor and Blank are calculated by:

$$F_n = F_o(C_s/Y_s)$$
$$b_n = b_o(F_n/F_o)$$

Where:
- C_s = Concentration of the standard
- o = Old value
- n = New value
- s = Value of the standard

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Both F_n and b_n are re-calculated each time either the Calibration Factor or the System Blank are updated. It should be noted that if the old value b_o is already 0, the new value b_n and therefore SB will also be 0. This provides a means to have the system effectively do a one point calibration update when it calculates a new Calibration Factor. These equations may also be used to manually calculate the values and enter them on the "Calibration" menu directly.

13.2. The instrument calculates the ug/g C in the sample from the linearized response and the sample volume using the above equations. As long as the user enters the calibration correctly and the sample volume correctly, the instrument will print the final result with no further manipulation.

13.3. The result from the instrument is a dry weight result and is not to be further adjusted in LIMS.

13.4. Data must be reviewed by the analyst and a peer (supervisor or qualified analyst) using a Data Quality Checklist before the results are validated and reported to the client. Further data review policies and procedures are discussed in ADM-DREV.

13.5. TIC=TC-TIC. If TOC>TC, it is OK to report if TOC is within 10%RPD of TC.

14. METHOD PERFORMANCE

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

Demonstration of Capability is performed upon instrument set-up , whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office

15. WASTE MANAGEMENT AND POLLUTION PREVENTION

- It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

- The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

- Excess, unused sample and testing byproducts are disposed following the procedures in SMO- SPLDIS.

16. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

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If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18. REFERENCES

18.1. "Total Organic Carbon in Soils" Lloyd Kahn EPA 1988.

18.2. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods*, USEPA SW-846.

18.3. OI Analytical, *Model 1010 Wet Oxidation TOC Analyzer Operator's Manual*, Revision 9.1, June 2001

19. TRAINING OUTLINE

19.1. Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies and procedures in ADM-TRANDOC.

19.2. Observe Sample Preparation and Analysis, including instrument operation and maintenance. Follow Training Plan Form.

19.3. Participate in the methodology, documentation, and data reduction with guidance.

19.4. Demonstrate Competency by performing the analysis independently. Analyze a known proficiency or standard four times for Initial Demonstration of Capability. If recovery is within acceptable limits, complete training plan form and IDC certificate. Turn paperwork in to QA for acceptance and filing.

19.5. Continuing Demonstration of Capability will be performed annually using an internal unknown, a PE, or a new four replicate study.

20. METHOD MODIFICATIONS

Method 9060 is for waters and wastes but is useful for soils as described here.

21. INSTRUMENT-SPECIFIC ADDENDUM

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The instrument manual is located next to the instrument. Refer to this manual for instrument setting or specifications not described in this SOP.

22. ATTACHMENTS

Benchsheet

23. CHANGES FROM PREVIOUS REVISION

- Eliminated most wording about High Level vs. Low Level since the range extender is always used at this time.
- Added TIC throughout
- Changed references to TOC to Carbon, where applicable, to be inclusive for the addition of TIC to this SOP
- 2.1 – Added section about pre-treatment of sample
- 4 – Added to be cautious of contamination by carry-over
- 5 – Changed wording of Safety section for consistency with other SOPs
- 8 – Expanded upon wording of Maintenance Log
- 9 – Rearranged to put Reagents together and Standards together
- 9- Eliminated 100 ug/g standard
- 9 – Added Standards Prep General Info and Disclaimers
- 12 – Modified wording of corrective actions for consistency
- 14 – Added ADM-Trandoc for consistency with other SOPs
- 15 – Modified for consistency with other SOPs
- 18 – Added reference to TOC Analyzer Manual (used to verify method for TIC)

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COLUMBIA ANALYTICAL SERVICES
1 Mustard St., Rochester, NY

Analysis Type: TOC-LK (EPA 9060) TC-LK (EPA 9060 Modified)

Analyst: _____ Date: _____

Pipet ID: _____ Start Time: _____

Curve Date: 6/7/05

Corr. Coeff.: 0.99977

Position #	Sub. #	Order #	Sample Amt. (mg)	Result (ug/g)	Comments
1	Std: 50 uL of 10,000 ppm Std.		Calibration	Source: TOC1-132D	
2	Std: 50 uL of 10,000 ppm Std.		Calibration	Source: TOC1-132D	
3	ICV	TV=5000	100	4949	99.0%
4	ICB		100	10.96	
5	LCS	TV=2000	100	1845	92.3%
6	8000	Curve Check	100	8205	102.6%
7	5000	Curve Check	100	5229	105%
8	3000	Curve Check	100	3134	104%
9	1000	Curve Check	100	910.6	91.1%
10	500	Curve Check	100	421.3	84%
11	300	Curve Check	100	299.4	99.8%
12	ERA CHECK	TV=12700	100	11820	93%
1	CCV	TV=5000	100		
2	CCB		100		
3	LCS	TV=2000	100		
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"Time Out Error": Baseline did not return after ignition to within 0.250 mV of the pre-ignition baseline

Note: For TOC analysis, samples are treated with 1:1 HNO_3 to remove any inorganic carbon. This is not done for TC analysis. The TIC value is determined by subtracting the TOC value from the TC value.

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*DOD SUMMARY

For work for the Department of Defense – the DOD Quality Systems Manual must be followed. The DOD Manual is based on the NELAC Standards with some additional requirements. The following are the requirements which are different or additional to routine analysis and must be followed for DOD work:

- The ICB/CCB/Method Blank must not have any hits above ½ the reporting limit.
- Reporting Limits –"the lowest standard of the calibration establishes the [reporting limit], but it must be greater than or equal to 3 times the [MDL]". DoD QSM, V3, Appendix B-Glossary

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STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

SOP No.: MET-200.7/6010B

Revision: 9

July 12, 2006

Approved by: _____ _____
Department Supervisor Date

_____ _____
Laboratory Manager Date

_____ _____
QA Coordinator Date

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1 Mustard Street, Suite 250
Rochester, New York 14609

Annual review of this SOP has been performed and the SOP still reflects current practice.
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1. SCOPE AND APPLICATION

This SOP uses EPA methods 200.7 and 6010B for the determination of trace metals in solution by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The method is applicable to all of the elements listed in Table 1. All matrices, including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. The Method Reporting Limits (MRL) are listed in Table 1. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated.

2. SUMMARY OF METHOD

Sample preparation and digestion is found in the appropriate digestion SOP. Examples include MET-3005A, MET-3010A, MET-3020A, MET-3050B, MET-TCLP, MET-TZHE, MET-SPLP, MET-SPLPZHE, and MET-NIOSH.

This method describes multiple elemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. 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. In one mode of analysis the position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 should also be recognized and appropriate corrections made.

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3. DEFINITIONS

3.1. Initial Calibration - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the detector to the element.

3.2. Calibration Blank - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument.

3.3. Calibration Standard (CAL) - A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration

3.4. Dissolved Analyte - The concentration of analyte in an aqueous sample that will pass through a 0.45 µm membrane filter assembly prior to sample acidification.

3.5. Instrument Detection Limit (IDL) - The concentration equivalent to a signal due to the analyte which is equal to three times the standard deviation of a series of 21 replicate measurements of a low standard's signal at the same wavelength.

3.6. Initial/Continuing Calibration Verification Solution (ICV/CCV) - A solution of method analytes from a separate source as the initial calibration standards, used to validate the curve.

3.7. Internal Standard - Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component.

3.8. Laboratory Duplicates - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates and indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.9. Laboratory Control Sample (LCS) – A matrix blank spiked with all of the elements of client interest. This is used to document laboratory accuracy.

3.10. Matrix Spike - An aliquot of an environmental sample to which known quantities of all of the elements of client interest are added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes

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bias to the analytical results in a given client matrix.

3.11. Preparation Blank (PB) or Method Blank (MB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

3.12. Linear Range - The concentration range over which the instrument response to an analyte is linear.

3.13. Method Detection Limit (MDL) or Limit of Detection (LoD) - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.14. Plasma Solution - A solution that is used to determine the optimum height above the work coil for viewing the plasma.

3.15. Interference Check Solution (ICS) - A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known interelement spectral interferences with respect to a defined set of method criteria.

3.16. Method Reporting Limit Standard (MRL) or Practical Quantitation Limit (PQL) or Limit of Quantitation (LoQ) – The minimum amount of a target analyte that can be measured and reported quantitatively.

3.17. MRL Standard - Standard prepared with a known concentration of elements to check accuracy at the low end of the curve.

3.18. HLCCV1 – A standard prepared at the bench at a high concentration to encompass the range of the samples being analyzed. This standard is used to assess accuracy at the high end of the calibration curve.

3.19. HLCCV2 – A standard prepared slightly higher than the calibration range for metals.

3.20. Batch – a group of no more than 20 field samples digested or analyzed together on the same day with the same reagents. See ADM-BATCH for more detail.

3.21. Matrix - the predominant material, component, or substrate (e.g., surface water, drinking water, etc.) of which the sample to be analyzed is composed.

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3.22. Relative Percent Difference (RPD) – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.23. Interelement Correction Factors (IECs) - factors that the instrument uses to compensate for spectral overlap when analyzing samples with complex spectra.

4. INTERFERENCES

There are several types of interferences by the ICPs: Spectral interferences can be from an overlap of spectral lines, background points or background from line emissions of high concentration elements. Physical interferences are effects associated with the sample introduction process, example high dissolved solids buildup on the nebulizer tip. Chemical interferences caused by the sample matrix itself. IECs aid in eliminating some of these interferences. IECs are interelement correction factors that the instrument uses to compensate for spectral overlap when analyzing samples with complex spectra. The following is the text from Method 6010B Section 3.0. Similar text is found in Method 200.7. Not all of the items may be applicable to this laboratory's instruments or procedures.

> Spectral interferences are caused by background emission from continuous 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.
>
> 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 (interelement 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 spectral interferences are handled by including spectra on interfering species in the algorithm.
>
> 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

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off-line interelement 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 concentration near the upper analytical range limit.

Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement 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 interfering effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement 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 wavelengths are given 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.

When using interelement 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 As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Interelement 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. Interelement 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. Interelement 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.

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The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous 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 (Table 2) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

Users of sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.

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.

When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within the 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All interelement 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.

When interelement corrections are not used, verification of absence of interferences is required.

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One method is to use a computer software routine for comparing the determinative data to limits files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is > 20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

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. The test described in Section 8.5.1 will help determine if a physical interference is present.

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

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 element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by

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aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

The dashes in Table 2 indicate that no measurable interferences were observed even at higher interfering concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

5. SAFETY

All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

Hydrochloric and Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g.,

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pressurized hydrogen) should be clearly labeled.

Refer to the Safety Manual for further discussion of general safety procedures and information.

High Voltage - The power unit supplies high voltage to the RF generator which is used to form the plasma. The unit should never be opened. Exposure to high voltage can cause injury or death.

UV Light - The plasma when lit is a very intense light, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available.

6. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

6.1. Solid samples require no preservation prior to analysis other than storage at 0-6°C. Sample containers may be glass or plastic. When the laboratory provides the sample containers, the containers are glass soil jars with Teflon-lined lids. Samples are analyzed within 6 months of collection.

6.2. For aqueous samples, glass or plastic sample containers are acceptable. When the laboratory provides the sample containers, the containers are 500 mL plastic bottles. Sample volume should be acid preserved with (1+1) nitric acid to pH <2. Samples are held at room temperature (although refrigeration is acceptable also). Samples are analyzed within 6 months of sample collection.

6.3. For the determination of the dissolved elements, the sample must be filtered through a 0.45 µm pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. (Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus should be used when the determinations of boron and silica are critical.) Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) nitric acid immediately following filtration to pH <2.

Note: When the nature of the sample is either unknown or is known to be hazardous, acidification should be done in a well ventilated area or fume hood.

6.4. Samples received by the ICP lab as digestates contain nitric and hydrochloric acid. Digestates are stored at room temperature in plastic B-cups or Hot Block Digestion cups.

6.5. Following analysis, digestates are stored until all results have been reviewed. Digestates are diluted and disposed of through the sewer system in approximately 90 days after receipt of

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sample.

6.6. For more information about custody, sample handling, and storage procedures, see SMO-GEN.

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7. APPARATUS AND EQUIPMENT

7.1. See Appendix A of the Quality Assurance Manual for configuration of specific components, serial numbers and receipt of the major components of the instruments.

7.2. Inductively coupled argon plasma emission spectrometer (ICP) equipped with the following:

7.2.1. Computer-controlled emission spectrometer with background correction.

7.2.2. Mass flow controller for argon nebulizer gas supply.

7.2.3. Peristaltic pump.

7.2.4. Autosampler.

7.2.5. Argon gas supply - high purity.

7.3. Volumetric flasks, class A .

8. PREVENTIVE MAINTENANCE

Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

Typical preventive maintenance measures include, but are not limited to, the following items:

- Cleaning the pump tubing as needed
- Empty waste container, as needed
- Cleaning the nebulizer, spray chamber, and torch, as needed
- Replace water and vacuum filters, as needed

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9. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1. Trace metals grade chemicals shall be used in all tests. Each lot of acid used is to be analyzed to demonstrate that it is free of interference before use. See ADM-CTMN.

9.1.1. Hydrochloric acid (conc.), HCl, and Nitric acid (conc), HNO_3. Purchased commercially. Store at room temperature. Expires three years from receipt or upon manufacturer's indications, whichever is sooner.

9.1.2. Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriately sized beaker. Store at room temperature. Expires one year from preparation.

9.1.3. Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriately sized beaker. Store at room temperature. Expires one year from preparation.

9.2. Reagent Water. All references to water in the method refer to DI Type II water unless otherwise specified. Reagent water will be interference free.

9.3. **<u>Standards Preparation General Information and Disclaimers</u>**

9.3.1. All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mLs or may be made by adding 4 mL of 50 mg/L to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

9.3.2. Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

9.3.3. All Standards must be traceable using the CAS lot system (ADM-DATANTRY). The manufacturer, lot number, standard /reagent name, concentration, date received and expiration date are recorded in a reagent log.

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9.3.4. All standards are prepared from NIST traceable stock standard solutions. Manufacturers expiration dates are used to determine viability of standards. Preparatory procedures for standards and QC solutions vary between instruments due to the working ranges. All preparatory information for the QA/QC samples are provided in Appendix I.

9.4. Mixed Calibration Standards are prepared by combining appropriate volumes of the stock solutions in volumetric flasks. Matrix match with the appropriate acid and dilute to 100ml with water. Calibration standards should be verified using a second source quality control sample (LCS, ICV, or CCV). Calibration standards should be stored at room temperature in glass volumetric flasks with a shelf-life of 7 days.

9.5. Initial and Continuing Calibration Verification (ICV and CCV) Standards are prepared by combining compatible analytes at concentrations equivalent to the midpoint of their respective calibration curves. The ICV and CCV standards should be prepared from a separate source independent from that used in the calibration standards. ICV / CCV standards should be stored at room temperature in glass volumetric flasks with a shelf-life of 48 hours.

9.6. Internal Standard - An internal standard solution consisting of Yttrium and Cesium. The Optima #1 uses a 100 mg/L solution of each element. The Optima #2 and #3 uses a 10 mg/L solution of each element. The apparent concentration is 1.00 mg/L Yttrium. The Yttrium intensity is used by the instrument to ratio the analyte intensity signals for both calibration and quantitation. Cesium is used only as a stabilizer. Store the prepared solutions at room temperature for up to 6 months.

9.7. HLCCV1- The highest calibration standard with the same storage and expiration as the calibration standards.

9.8. HLCCV2 – A standard prepared to verify the linear range of the curve. See Attachment I. Store at room temperature for up to 14 days.

9.9. MRL Standards (low level calibration check standards) are prepared to contain known concentrations of elements at or near the Method Reporting Limit. MRL standards should be stored in plastic containers with a shelf-life of 6 months.

9.10. Interference Check Solutions A and AB are prepared to contain known concentrations of interfering analytes that will provide an adequate test of the correction factors. ICSA / ICSAB standards should be stored in plastic containers with a shelf-life of 6 months.

9.11. Laboratory Control Sample and Matrix Spike are purchased as custom mixes stored in plastic containers with a shelf-life of 6 months at the concentrations recommended in the method.

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The soil LCS is purchased as a soil reference material to be used as a sample. Each sample, up to 50 ml, is spiked with 0.5 ml of spike solution. The LCS and MS will contain all of the target analytes. If a target is not in the LCS/MS custom mix, the target will be added separately to the LCS and MS.

9.12. Blanks

9.12.1. Method Blanks (all matrices) – process DI water as a sample and carry through the entire preparation and analysis procedure. The MB must contain all the reagents in the same volumes as used in the preparation of samples.

9.12.2. The Calibration Blank is prepared by acidifying reagent water to the same concentrations of acid found in the standards and samples.

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

11. PROCEDURE

11.1. All samples are digested prior to analysis. Refer to the following Metals Digestion SOPs:

MET-3005A	Metals Digestion, Waters, Total Recoverable and Dissolved for ICP
MET-3010A	Metals Digestion, Waters for ICP
MET-3020A	Metals Digestion, Waters for GFAA
MET-3050B	Metals Digestion, Soils, Sediments and Sludges for ICP and GFAA
MET-CLP	Metals Digestion, Waters and Soils for CLP

11.2. Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 45 minutes of operation prior to calibration). Operating conditions - The analyst should follow the instructions provided in Table 3.

11.3. Before using this procedure to analyze samples, there must be data available documenting initial demonstration of performance. The required data documents the selection criteria of background correction points; linear ranges, and the upper limits of those ranges; the method and instrument detection limits; and the determination and verification of

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interelement correction equations or other routines for correcting spectral interferences. This data must be generated using the same instrument, operating conditions and calibration routine to be used for sample analysis. These documented data must be kept on file and be available for review by the data user or auditor. The limits and on-going frequency of these performance demonstrations are provided in Section 12.

11.4. Be sure the analyst has a current Demonstration of Capability (see section 19).

11.5. Turn on power supply for the instrument, computer, printer and light the plasma. Allow instrument to warm-up for 45-60 minutes before operation. The cooling water and the argon are on when the instrument are on.

11.6. Profile the instrument on a daily basis, or when maintenance is done to align it optically for both horizontal and vertical optimization in either mode. The vendor recommends aspirating a 10 ppm source of manganese. Choose the Tools menu/Spectrometer Control/Optimize X&Y. The instrument automatically adjusts the torch viewing position for maximum intensity.

11.7. Addition of Internal standard -

11.7.1. Pour 40 mL of the calibration blank, the 3 calibration standards, ICV/CCV standards, MRL, ICSA, and ICSAB into separate 50 mL centrifuge tubes and add 0.80 mL of the 100 mg/L internal standard solution. All other samples, preparation blanks and laboratory control samples are poured up to 10 mL in a 15 mL centrifuge tube and 0.20 mL of internal standard solution is added. This will give an apparent concentration of 1.00 mg/L Yttrium.

11.7.2. Internal standards can be added via pump and mixing block. This technique uses a solution of 10 mg/L Y and 10 mg/L Cs.

11.7.3. Place the tubes on the autosampler and program the software to analyze according to the analytical sequence.

11.8. Analytical Sequence

11.8.1. Initial Calibration; ICV; ICB; MRL; ICSA; ICSAB; CCV; CCB;
10 environmental samples (including PBs and LCSs); CCV; CCB; repeat to the end of the run…..
Last 10 samples; CCV; CCB, MRL, ICSA, ICSAB; HLCCV1; HLCCV2; CCV; CCB.
For more information, refer to ADM-BATCH for Batches and Sequences.

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11.8.2. Rinse the system with the blank solution before the analysis of each sample. The rinse time will be at least one minute, depending upon the instrument. Each laboratory may establish a reduction in this rinse time through a suitable demonstration.

11.9. Initial Calibration- follow ADM-ICAL. If these instructions conflict with ADM-ICAL, follow the instructions in this SOP.

11.9.1. Number of Calibration standards – 3 standards and a calibration blank are analyzed with each analytical batch or sequence. The low standard must be at or below the MRL. The high standard will define the upper limit of the linear range for the curve. The calibration blank should not be included as a zero standard in the curve.

11.9.2. Initial Calibration Curve Calculation – the intensity is plotted against concentration using the linear equation of a line:

$$y=mx+b$$

To avoid bias at the low end of the curve, the curve is forced through zero. Forcing the curve through zero may favor the low end of the curve therefore a low-level CCV (CRDL or MRL standards) and high level CCV (HLCCV1 and HLCCV2) are analyzed to verify both ends of the curve (see analytical sequence).

11.9.3. Acceptance Criteria – the correlation coefficient must be 0.995 or greater for target analytes of interest and the ICV must meet limits (see section 12).

11.9.4. Corrective Action – if the ICAL does not meet acceptance criteria, correct the problem and recalibrate. The curve must be acceptable before sample analysis begins.

11.10. Sample Analysis and Evaluation

11.10.1. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

11.10.2. Samples which exceed the linear range of the instrument must be diluted and reanalyzed according to ADM-DIL.

*See DOD Summary Attachment for DOD Specific Criteria

11.10.3 Evaluate QC according to section 12. Repeat samples associated with non-compliant QC whenever possible.

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*See DOD Summary Attachment for DOD Specific Criteria

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12. QA/QC REQUIREMENTS

12.1. Instrument values are based on duplicate readings. Precision between the emission readings shall not exceed 20 %RSD. If RSD values exceed 20%, reanalyze the sample.

12.2. Preparation Blanks

- Frequency -at least one PB with each batch of 20 or fewer samples of the same matrix.
- Limits - PB values must not exceed the MRL*.
- Corrective Action - Fresh aliquots of the samples must be prepared and analyzed again for affected analytes after the source of the contamination has been corrected and acceptable PB values have been obtained. If detections are greater than the MRL, the batch needs to be redigested if sample concentration is less than 10 times the concentration found in the prep blank. If the sample concentration is less than the MRL the sample does not require redigestion.

12.3. HLCCV1 – High standard used in curve

- Frequency - analyzed once during daily analysis.
- Limits - Should agree within 10% of the true value.
- Corrective Action - If HLCCV1 is > 10% different the analysis is judged to be out of control and the source of the problem should be identified and resolved before continuing analysis.

12.4. HLCCV2 – standard slightly higher than calibration for some metals.

- Frequency - Analyzed once during daily analysis.
- Limits - Should agree within 10% of the true value.
- Corrective Action - If out of control, client data above the HLCCV1 should be re-analyzed.

12.5. ICV –

- Frequency - must immediately follow each calibration.
- Limits - ±5% for Method 200.7 and ±10% for Method 6010B.
- Corrective Action – correct the problem and recalibrate the instrument.

*See DOD Summary Attachment for DOD Specific Criteria

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12.6. CCV

- Frequency - after every tenth sample, and at the end of the sample run
- Limits - ±10%
- Corrective Action – correct the problem and analyze another CCV. If second CCV fails, recalibrate. Reanalyze all samples since the last successful CCV.

12.7. CCB

- Frequency – after every ICV and CCV (at the beginning and end of the run and after every 10 samples)
- Limits - The results of the calibration blank (CCB) must be less than the MRL*.
- Corrective Action - If not, terminate the analysis, correct the problem, recalibrate, and reanalyze the samples affected.

12.8. MS –

- Frequency - one per matrix batch (max. 20 samples) or one per 10 for Method 200.7.
- Limits - within ± 25%* of the actual value or within the documented historical acceptance limits for each matrix. All elements of client interest are evaluated. Sample concentrations greater than four times the spike concentration are not valid and shall not be evaluated.
- Corrective Action - If the matrix spike does not meet these criteria, analyze a Post Digestion Spike. DOD requires project-specific DQOs be examined or contact the client for additional measures. See Table B-6.

12.9. DUP –

- Frequency - one per matrix batch (max. 20 samples) or one per 10 for Method 200.7
- Limits - A control limit of ± 20% RPD shall be used for original and duplicate samples greater than or equal to 5X the CRDL. A control limit of ± the CRDL shall be used if either the sample or duplicate value is less than 5 times the CRDL. CRDL values are given in Table 1.
- Corrective Action – If the DUP does not meet criteria, data will be reported with a qualifier.

*See DOD Summary Attachment for DOD Specific Criteria

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12.10. Laboratory Control Sample –

- Frequency – one per matrix batch (max. 20 samples).
- Limits – all elements of client interest are evaluated.
  - Waters - Results should be within ± 15% of the true value for method 200.7 and ± 20% for 6010B.
  - Soils – Results must be within the limits indicated on the Certificate of Analysis for the soil reference.
- Corrective Action - Outlying recoveries may indicate loss of analyte due to digestion procedures or laboratory contamination. If an LCS is found to be out of the specified limits, recalibrate and reanalyze. If the LCS remains out of control with a low bias, redigestion of the entire batch should occur. If the LCS remains out of control with a high bias, redigestion of all positive results (greater than the MRL) should occur.

12.11. MRL standard (low level calibration check standard) - A standard at near* the MRL is analyzed

- Frequency - at the beginning and end of each analytical run but not before the ICV.
- Limits- There are no limits in the 200.7 or 6010B methods, but the CAS guideline used is +/- 50% of the true value*.
- Corrective Action - If the limits are not met the analysis is stopped and the instrument is recalibrated.

12.12. Interference Check Samples-

12.12.1. ICSA

- Frequency - at the beginning and end of each analytical batch.
- Limits – less than MRL (less than 2 times MRL for metals with MRL < 10mg/L)*.
- Corrective Action – terminate analysis; locate and correct problem; reanalyze ICS

12.12.2. ICSAB –

- Frequency – immediately after ICSA
- Limits - The analyte recoveries for the AB solution must fall within 20% of the true value
- Corrective Action - recalibrate and reanalyze unless analytes are not detected in the associated samples or interfering elements are not present at the ICSA level.

*See DOD Summary Attachment for DOD Specific Criteria

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12.13. Serial Dilution Test –

- Frequency – one each prep batch or when a new or unusual matrix is encountered. Only applicable to samples with a concentration greater than 50 times the MDL. If no samples in the prep batch are >50xMDL, analyze a post-digestion spike.
- Limits - a 5-fold dilution should agree within ± 10% of the original determination.
- Corrective Action – if the results are not within limits, flag the data with a qualifier*.

12.14. Post Digestion Spike Addition (for 6010B analyses): The spike addition should produce a minimum level of 10 times and a maximum of 100 times the IDL

- Frequency – When a dilution test fails, or when no samples have a concentration 50 times the MDL or if a matrix spike does not yield acceptable results.
- Limits - should be recovered to within 75% to 125% of the known value.
- Corrective action - If the spike is not recovered within the specified limits, a matrix effect has been confirmed and the data should be flagged.

12.15. Instrument Performance

12.15.1. InterElement Correction Factors (IEC)

- Procedure – A calibration curve is analyzed as per instrument specifications. Once completed, individual standards for Al, Ca, Fe, Mg are analyzed. The instrument software then creates an IEC table.
- Frequency - annually, or as needed.
- Limits and corrective action – The ICSA check standard routinely confirms the IECs. If the ICSA is persistently problematic, re-establish IECs.

12.15.2. Linear Ranges (LR)

- Procedure – A standard is prepared at the linear range level and analyzed.
- Frequency – biannually
- Limits - must be ±5% of true value.
- Corrective Action – If the linear range is too high, lower the linear range and repeat the study.

12.15.3. Instrument Detection Limits (IDL)

- Procedure – Analyze 7 replicates of a low level standard. Repeat twice more for a total of 21 replicates over 3 non-consecutive days.
- Frequency - with initial set-up, after significant change and quarterly thereafter.
- Limits – The calculated IDL must be less than the PQL*.

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- Corrective Action – if the calculated IDL is not less than the limit, correct the problem and perform a new IDL study.

12.15.4 Method Detection Limits (MDL)

- Procedure – according to 40CFR and ADM-MDL.
- Frequency – annually or quarterly MDL verification checks
- Limits – must meet the MDL study criteria as stated in ADM-MDL
- Corrective Action – see ADM-MDL.

13. DATA REDUCTION AND REPORTING

13.1. Calculations: If dilutions were performed, the appropriate factors must be applied to sample values. All results should be reported with up to three significant figures. Refer to ADM-DREV for guidance in data review and acceptability. See Appendix D of the Quality Assurance Manual for flag definitions. See attached Table B-6 for DOD flagging requirements. The equations for determining Matrix Spike Recovery, LCS Recovery, and Duplicate Precision (RPD) are found in the Quality Assurance Manual.

13.1.1. Sample Calculation (water)

$$\text{Conc. (mg/L)} = \frac{\text{Instrument Reading (mg/L) x Final digestion volume (L)}}{\text{Initial volume (L)}}$$

13.1.2. Sample Calculation (soils)

$$\text{Conc. (mg/g)} = \frac{\text{Instrument Reading (mg/L) x Final digestion volume (L)}}{\text{Initial mass (g) x Percent Solids expressed as a decimal}}$$

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14. METHOD PERFORMANCE

Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit. See Table 2 for approximate wavelengths. See ADM-MDL for more information

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office*.

Demonstration of Capability is performed upon instrument set-up , whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office.

15. WASTE MANAGEMENT AND POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when disposed of properly.

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual..

Samples with analyte concentrations exceeding TCLP regulatory limits are disposed of as hazardous waste, see SOP *SMO-SPLDIS*.

16. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures

*See DOD Summary Attachment for DOD Specific Criteria

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in Section 15 of the Quality Assurance Manual.

18. REFERENCES

18.1. *Test Methods For Evaluating Solid Waste, Physical/Chemical Methods*. USEPA SW-846, 3rd Edition, December 1996.

18.2. *Methods For the Determination of Metals in Environmental Samples Supplement I*. USEPA/600/R-94/111, May 1994.

18.3. *D*OD Quality Systems Manual – Version 3, January 2006.

19. TRAINING OUTLINE

19.1. Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2. Observe Sample Preparation and Analysis. Follow Metals Analysis Training Plan Form (found on the Rochester CASLAB Intranet website).

19.3. Participate in the methodology, documentation, and data reduction with guidance.

19.4. Demonstrate Competency by performing the analysis independently. Analyze four replicates of a known mid-range standard. If recovery is within acceptable limits, complete Training Plan Form and IDC certificate and file with QA. Continuing demonstration (CDC) will be performed annually using a Proficiency, an internal unknown, or a new 4 replicate study.

20. METHOD MODIFICATIONS

This lab is using a 3 point curve for initial calibration. The methods only require one point.

21. INSTRUMENT-SPECIFIC ADDENDUM

- Perkin Elmer Optima 3000XL (ICP #1)
- Perkin Elmer Optima 3000XL (ICP #2)
- Perkin Elmer 5300DV (ICP#3)
- For operation: Refer to Manufacturer Operation Manuals located in ICP lab.

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22. ATTACHMENTS

- Table 1 List of Analytes and Practical Quantitation Limits
- Table 2 Potential Interferences
- Table 3 Recommended Wavelengths and Instrument Specifications
- Table B-6 DOD specific Requirements from DOD QSM Version 3 January 2006
- Appendix I Standard Preparation Instructions

*See DOD Summary Attachment for DOD Specific Criteria

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23. CHANGES FROM PREVIOUS REVISION

- Added DOD Summary attachment and DOD * and footnote
- 1-reworded to include methods 200.7 and 6010B.
- 2-Added SOP references for the digestions
- 3-Added definitions for Initial Calibration, Matrix, RPD, and IECs. Changed the definition for IDL.
- 4-Added text from 6010B for interferences
- 5-Added wording for safety for consistency with other SOPs
- 6-Added details about the containers used when the bottle set is sent by the lab
- 8-Expanded upon the maintenance log section for consistency with other SOPs
- 9-Added reference to ADM-CTMN
- 9-Added Standard Prep General Information and Disclaimers
- 9-Added prep of Internal Standard solution, HLCCV1, HLCCV2.
- 9-Added that the soil LCS is purchased as a reference material
- 9-Added that the LCS and MS will contain all targets
- 9-Eliminated Certificate of Analysis attachment for soil LCS
- 9-Expanded upon MB – added that the matrix is DI
- 9-Eliminated reagent receiving log section and referenced ADM-DATANTRY in General Info section
- 11-Added need to check for DOC
- 11-Reworded many areas for clarity
- 11-Added ICAL section
- 11-Eliminated MDL discussion from this section – moved to 14
- 12-Greatly expanded upon and formatted to include Frequency, Limits, and Corrective Action for each QC element.
- 13-Expanded upon references to other SOPs for data and calculation information-eliminated calculations for MS recovery and RPD (referenced QAM)
- 14+15-reworded for consistency with other SOPs
- 18-Added reference to DOD QSM
- 21-Added ICP#3
- 22-Added Table 2 and B6

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*DOD SUMMARY

For work for the Department of Defense – the DOD Quality Systems Manual must be followed. The DOD Manual is based on the NELAC Standards with some additional requirements. The exact wording is in Table B6. The following are the requirements which are different or additional to routine analysis and must be followed for DOD work:

- The Second Source Calibration Verification (ICV) must have a recovery of 80-120% of the true value.
- The CCB must be less than twice the MDL.
- The Method Blank must be less than ½ the reporting limit (<RL for common laboratory contaminants).
- Apply J flag to all hits between MDL and MRL.
- Reporting Limits – "the lowest standard of the calibration establishes the [reporting limit], but it must be greater than or equal to 3 times the [MDL]". DoD QSM, V3, Appendix B-Glossary
- The limits for LCS and MS are 80-120%. All targets are spiked and evaluated.
- The IDL must be less than the MDL.
- Low Level Check Standard (MRL standard) must be spiked at or below the reporting limit and it must have a recovery of 80-120% of the true value when analyzed at the beginning of the run.
- ICS-A absolute value of concentration for all non-spiked analytes must be <2 times MDL (unless they are a verified trace impurity from one of the spiked analytes).
- Serial Dilution Test Corrective Action – If the limits are not met, perform a post digestion spike addition.

*See DOD Summary Attachment for DOD Specific Criteria

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TABLE 1

Analyte	*MRLs Water-Optima mg/L*	*MRLs Soil ug/g*	*Typical IDL ug/L*
Silver	0.010	1.00	0.632
Aluminum	0.100	10.0	6.57
Arsenic	0.0100	50.0	6.89
Boron	0.200	20.0	37.3
Barium	0.0200	2.00	12.2
Beryllium	0.0050	0.500	0.26
Calcium	1.00	100.0	167
Cadmium	0.0050	0.500	0.489
Cobalt	0.0500	5.00	3.03
Chromium	0.0100	1.0	1.81
Copper	0.0200	2.00	3.02
Iron	0.100	5.00	44.1
Potassium	2.00	100	857
Magnesium	1.00	100	124
Manganese	0.0100	1.0	1.78
Molybdenum	0.0250	2.50	3.08
Sodium	1.00	100	193
Nickel	0.0400	4.00	3.92
Lead	0.00500	5.00	1.29
Antimony	0.0600	10.0	3.72
Selenium	0.00500	50.0	12.5
Tin	0.500	100	15.8
Titanium	0.0500	5.00	3.15
Thallium	0.0100	30.0	7.77
Vanadium	0.0500	5.00	2.74
Zinc	0.0200	1.0	2.47

*See DOD Summary Attachment for DOD Specific Criteria

TABLE 2
POTENTIAL INTERFERENCES
ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM
INTERFERENCE AT THE 100-mg/L LEVEL[c]

Analyte	Wavelength (nm)	Interferant[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 mg/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

[c] Interferences will be affected by background choice and other interferences may be present.

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Table 3

Recommended Wavelengths and Instrument Specifications

Suggested wavelengths are listed below:

Analyte	Wavelength
Ag Silver	328.068
Al Aluminum	308.215
B Boron	249.773
Ba Barium	233.527
Be Beryllium	234.861
Ca Calcium	430.253
Cd Cadmium	226.502
Co Cobalt	228.616
Cr Chromium	267.716
Cu Copper	324.754
Fe Iron	238.863
Mg Magnesium	279.079
Mn Manganese	257.610
Mo Molybdenum	202.030
Na Sodium	330.237
Ni Nickel	231.604
Pb Lead	220.353
Sb Antimony	206.833
Sn Tin	189.933
Ti Titanium	334.941
V Vanadium	292.402
Zn Zinc	206.191
Y Yttrium	371.030

Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. Because of differences among various makes and models of spectrometers, specific instrument operating conditions cannot be provided. The instrument operating conditions herein are recommended based upon manufacturer's instrument manuals.

Current Method Operating Conditions are as follows, these conditions may vary to optimize the instrument for different analyses:

Parameter	Radial Plasma	Axial Plasma
Resolution	Fixed	Fixed
Purge Gas Flow	Normal	Normal
Read Time (min/max sec.)	5/20	5/50
Replicates	2	2
Plasma (L/min)	15	15
Aux. (L/min)	0.5	0.3
Nebulizer Flow (L/min)	0.72	0.56
Power (watts)	1300	1450

*See DOD Summary Attachment for DOD Specific Criteria

Viewing Height (mm) 15 15

Table B-6 DOD specific Requirements from DOD QSM Version 3 January 2006

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	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).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18).	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
Instrument detection limit (IDL) study (ICP only)	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Linear dynamic range or high-level check standard (ICP only)	Every 6 months	Within ± 10% of expected value	NA	NA	

*See DOD Summary Attachment for DOD Specific Criteria

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes (ICAL) **(ICP: minimum one high standard and a calibration blank; GFAA: minimum three standards and a calibration blank; CVAA: minimum 5 standards and a calibration blank)**	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case r ≥ 0.995. GFAA: r ≥ 0.995 CVAA: r ≥ 0.995	Correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	ICP: within ± 10% of expected value GFAA: within ± 20% of expected value CVAA: within ± 20% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±20% of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch	No analytes detected > ½ RL For common laboratory contaminants, no analytes detected > RL	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid ICS.
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ± 10% of the original determination	ICP: Perform post-digestion spike (PDS) addition GFAA: Perform recovery test CVAA: Perform matrix spike	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x MDL (ICP) or > 25 x MDL (GFAA and CVAA).
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples < 50 x MDL	Recovery within 75-125% of expected result.	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	The spike addition should produce a level between 10 - 100 x MDL.

TABLE B-6. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) ATOMIC EMISSION SPECTROMETRY AND ATOMIC ABSORPTION SPECTROPHOTOMETRY (AA) (METHODS 6010 AND 7000 SERIES) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples < 25 x MDL	Recovery within 85-115% of expected result.	Run samples by method of standard addition (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range.	
Method of standard additions (MSA) or Internal standard calibration	When matrix interference is suspected	NA	NA	NA	Document use of MSA in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	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 J-flag 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.
MSD or sample duplicate	One per preparatory batch per matrix	RPD ≤ 20% (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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ	

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APPENDIX I

PREPARATION PROCEDURES FOR STANDARDS AND QC

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2 CALIBRATION STANDARD #1 (Standard is prepared weekly or as necessary)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Single Metals	AL		10000	0.010	1000	0.100	2%HNO3		A				
	BA		100	0.200		0.0200	5%HCl		B				
	CU		1000	0.020		0.0200			C				
	MN		1000	0.010		0.0100			D				
	MO		1000	0.025		0.0250			E				
	SB		1000	0.060		0.0600			F				
Cal	AS		100	0.100		0.0100			G				
Std 4	CD		50			0.0050			H				
	SE		50			0.0050			I				
	PB		50			0.0050			J				
	TL		100			0.0100			K				
									L				
									M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				
									U				
									V				
									W				
									X				
									Y				
									Z				

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2- CALIBRATION STANDARD #4 / HLCCV1 (Standard is prepared weekly or as necessary)
(CALIBRATION STANDARD #2 IS A 1/100 DILUTION OF THIS STANDARD)
(CALIBRATION STANDARD #3 IS A 1/5 DILUTION OF THIS STANDARD)

	Meta l	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Cal Std 1	CA		5000	2.00	200	50.0	2%HNO3		A				
	MG		5000			50.0	5%HCl		B				
	K		5000			50.0			C				
	NA		5000			50.0			D				
Cal Std 2	AG		100	2.00		1.00			E				
	CR		100			1.00			F				
	MN		150			1.50			G				
	NI		400			4.00			H				
	ZN		200			2.00			I				
Cal Std 3	AL		2000	2.00		20.0			J				
	BA		2000			20.0			K				
	BE		50			0.500			L				
	CO		500			5.00			M				
	CU		250			2.50			N				
	FE		1000			10.0			O				
	V		500			5.00			P				
Cal Std 4	AS		100	4.00		2.00			Q				
	CD		50			1.00			R				
	PB		50			1.00			S				
	SE		50			1.00			T				
	TL		100			2.00			U				
Single Metals	SB		1000	2.00		10.0			V				
	SN		1000	2.00		10.0			W				
	B		1000	1.00		5.00			X				
	MO		1000	1.00		5.00			Y				
	TI		1000	1.00		5.00			Z				

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2 ICV/CCV STANDARD (Standard is prepared daily.)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Pipet ID
Cal Std 1	CA		5000	1.00	200	25.0	2%HNO3		A			
	MG		5000			25.0	5%HCl		B			
	K		5000			25.0			C			
	NA		5000			25.0			D			
Cal Std 2	AG		100	1.00		0.500			E			
	CR		100			0.500			F			
	MN		150			0.750			G			
	NI		400			2.00			H			
	ZN		200			1.00			I			
Cal Std 3	AL		2000	1.00		10.0			J			
	BA		2000			10.0			K			
	BE		50			0.250			L			
	CO		500			2.50			M			
	CU		250			1.25			N			
	FE		1000			5.00			O			
	V		500			2.50			P			
Cal Std 4	AS		100	2.00		1.00			Q			
	CD		50			0.500			R			
	PB		50			0.500			S			
	SE		50			0.500			T			
	TL		100			1.00			U			
Single Elements	SB		1000	1.00		5.00			V			
	SN		1000	1.00		5.00			W			
	B		1000	0.500		2.50			X			
	MO		1000	0.500		2.50			Y			
	TI		1000	0.500		2.50			Z			

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2- HLCCV2 (Standard is prepared weekly or as necessary)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Cal Std 2	AG		100	2.00	100	2.00	2%HNO3		A				
	CR		100			Below	5%HCl		B				
	MN		150			Below			C				
	NI		400			8.00			D				
	ZN		200			4.00			E				
Cal Std 3	AL		2000	2.00		Below			F				
	BA		2000			40.0			G				
	BE		50			1.00			H				
	CO		500			10.0			I				
	CU		250			5.00			J				
	FE		1000			Below			K				
	V		500			10.0			L				
Cal Std 4	AS		100	4.00		4.00			M				
	CD		50			2.00			N				
	PB		50			Below			O				
	SE		50			2.00			P				
	TL		100			4.00			Q				
Single Metals	MO		1000	1.00		10.0			R				
	PB		1000	0.800		10.0			S				
	AL		10000	4.60		500			T				
	CA		10000	5.00		500			U				
	CR		1000	0.800		10.0			V				
	FE		10000	1.80		200			W				
	MG		10000	5.00		500			X				
	MN		1000	0.700		10.0			Y				
									Z				

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2 MRL STANDARD

	Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Exp. Date	Pipet ID
Cal #1	Ca		5000	0.200	1000	1.00	5% HCL		A				
	Mg		5000			1.00	2%HNO3		B				
	Na		5000			1.00			C				
									D				
Cal #2	Cr		100	0.10		0.0100			E				
	Ag		100			0.0100			F				
	Mn		150			0.0150			G				
	Zn		200			0.0200			H				
	Ni		400			0.0400			I				
Cal #3	Al		2000	0.10		0.200			J				
	Ba		2000			0.200			K				
	Fe		1000			0.100			L				
	Co		500			0.050			M				
	V		500			0.050			N				
	Cu		250			0.025			O				
	Be		50			0.00500			P				
Cal #4	Cd , Pb , Se		50	0.20		0.0100			Q				
	As, Tl		100			0.0200			R				
PQL #2	B		200	1.00		0.200			T				
	Mo		25			0.0250			U				
	Sn		500			0.500			V				
	Ti		50			0.050			W				
Single Stds	Sb		1000	0.060		0.060			X				
									Y				
									Z				

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2 ASP CRDL STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
CRDL STD		Multi	0.500	500	Multi	5% HCL 2%HNO3		A				
AG		20			0.0200			B				
AS		20			0.0200			C				
BE		10			0.0100			D				
CD		10			0.0100			E				
CR		20			0.0200			F				
CO		100			0.100			G				
CU		50			0.0500			H				
MN		30			0.0300			I				
NI		80			0.0800			J				
PB		6			0.00600			K				
SB		120			0.120			L				
SE		10			0.0100			M				
TL		20			0.0200			N				
V		100			0.100			O				
ZN		40			0.0400			P				
								Q				

*See DOD Summary Attachment for DOD Specific Criteria

ICSA STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Int. A Sol'n		Multi	100	1000	Multi	5% HCL		A				
AL		5000			500	2%HNO3		B				
CA		5000			500			C				
FE		2000			200			D				
MG		5000			500			E				
								F				
								G				
								H				
								I				
								J				
								K				
								L				
								M				
								N				
								O				
								P				
								Q				
								R				
								S				
								T				
								U				
								V				

*See DOD Summary Attachment for DOD Specific Criteria

AXIAL OPTIMA #2 ICSAB STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Int. A Sol'n		Multi	50	500	Multi			A				
AL		5000			500			B				
CA		5000			500			C				
FE		2000			200			D				
MG		5000			500			E				
Int. B Sol'n		Multi	5		Multi			F				
AG		20			0.200			G				
BA		50			0.500			H				
BE		50			0.500			I				
CD		100			1.00			J				
CO		50			0.500			K				
CR		50			0.500			L				
CU		50			0.500			M				
MN		50			0.500			N				
NI		100			1.00			O				
PB		5			0.0500			P				
V		50			0.500			Q				
ZN		100			1.00			R				
AS		10			0.100			S				
SB		60			0.600			T				
SE		5			0.0500			U				
TL		10			0.100			V				

*See DOD Summary Attachment for DOD Specific Criteria

OPTIMA #2 ON-LINE INTERNAL STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Y		10000	2.0	2000	10.0	2% HNO3		A				
Cs		10000	2.0	2000	10.0	5% HCL		B				
								C				
								D				
								E				
								F				
								G				
								H				
								I				
								J				
								K				
								L				
								M				
								N				
								O				
								P				
								Q				
								R				
								S				

*See DOD Summary Attachment for DOD Specific Criteria

MISCELLANEOUS STANDARDS

Type Of Std	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot#	Expiration Date	Pipet ID
									A				
									B				
									C				
									D				
									E				
									F				
									G				
									H				
									I				
									J				
									K				
									L				
									M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				
									U				
									V				
									W				

*See DOD Summary Attachment for DOD Specific Criteria

RADIAL OPTIMA #1 CALIBRATION STANDARD #1 (Standard is prepared weekly or as necessary)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
PQL Std 1	BE		5	0.100	100	0.0050	2%HNO3		A				
	CO		50			0.0500	5%HCl		B				
	V		50			0.0500			C				
	SB		60			0.0600			D				
	AG		10			0.0100			E				
	ZN		20			0.0200			F				
	FE		100			0.100			G				
	NI		40			0.0400			H				
	CR		10			0.0100			I				
PQL Std 2	B		200	0.100		0.200			J				
	MO		25			0.0250			K				
	SN		500			0.500			L				
	TI		50			0.0500			M				
Single std	SR		1000	0.010		0.100			N				
									O				
									P				
									Q				
									R				
									S				
									T				
									U				
									V				
									W				
									X				
									Y				
									Z				

RADIAL OPTIMA #1 CALIBRATION STANDARD #2 (Standard is prepared weekly or as necessary)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
CAL STD 1	CA		5000	0.100	1000	0.500	2%HNO3		A				
	K		5000			Below	5%HCl		B				
	MG		5000			0.500			C				
	NA		5000			0.500			D				
Single std	AL		10000	0.01		0.100			E				
	BA		100	0.20		0.020			F				
	CU		1000	0.02		0.020			G				
	MN		1000	0.01		0.010			H				
	K		10000	0.15		2.00			I				
	CD		1000	0.01		0.010			J				
	PB		1000	0.05		0.050			K				
	AS		1000	0.20		0.200			L				
	SE		1000	0.20		0.200			M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				
									U				
									V				
									W				
									X				
									Y				
									Z				

RADIAL OPTIMA #1- CALIBRATION STANDARD #4 / HLCCV1 (Standard is prepared weekly or as necessary)
(CALIBRATION STANDARD #3 IS A 1/5 DILUTION OF THIS STANDARD)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Cal Std 1	CA		5000	4.00	200	100	2%HNO3		A				
	MG		5000			100	5%HCl		B				
	K		5000			100			C				
	NA		5000			100			D				
Cal Std 2	AG		100	2.00		1.00			E				
	CR		100			1.00			F				
	MN		150			1.50			G				
	NI		400			4.00			H				
	ZN		200			2.00			I				
Cal Std 3	AL		2000	2.00		20.0			J				
	BA		2000			20.0			K				
	BE		50			0.500			L				
	CO		500			5.00			M				
	CU		250			2.50			N				
	FE		1000			10.0			O				
	V		500			5.00			P				
Cal Std 4	AS		100	8.00		4.00			Q				
	CD		50			2.00			R				
	PB		50			2.00			S				
	SE		50			2.00			T				
	TL		100			4.00			U				
Single Metals	SB		1000	2.00		10.0			V				
	SN		1000	2.00		10.0			W				
	B		1000	1.00		5.00			X				
	MO		1000	1.00		5.00			Y				
	TI		1000	1.00		5.00			Z				
	SR		1000	1.00		5.00			AA				

RADIAL OPTIMA #1 ICV/CCV STANDARD (Standard is prepared daily.)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Pipet ID
Cal Std 1	CA		5000	2.00	200	50.0	2%HNO3		A			
	MG		5000			50.0	5%HCl		B			
	K		5000			50.0			C			
	NA		5000			50.0			D			
Cal Std 2	AG		100	1.00		0.500			E			
	CR		100			0.500			F			
	MN		150			0.750			G			
	NI		400			2.00			H			
	ZN		200			1.00			I			
Cal Std 3	AL		2000	1.00		10.0			J			
	BA		2000			10.0			K			
	BE		50			0.250			L			
	CO		500			2.50			M			
	CU		250			1.25			N			
	FE		1000			5.00			O			
	V		500			2.50			P			
Cal Std 4	AS		100	4.00		2.00			Q			
	CD		50			1.00			R			
	PB		50			1.00			S			
	SE		50			1.00			T			
	TL		100			2.00			U			
Single Elements	SB		1000	1.00		5.00			V			
	SN		1000	1.00		5.00			W			
	B		1000	0.500		2.50			X			
	MO		1000	0.500		2.50			Y			
	TI		1000	0.500		2.50			Z			
	SR		1000	0.500		2.50			AA			

RADIAL OPTIMA #1 - HLCCV2 (Standard is prepared weekly or as necessary.)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Single Metals	CA		10000	5.00	100	500	2%HNO3		A				
	MG		10000	5.00		500	5%HCl		B				
	K		10000	3.00		300			C				
	NA		10000	4.00		400			D				
Cal Std 2	AG		100	2.00		2.00			E				
	CR		100			Below			F				
	MN		150			Below			G				
	NI		400			8.00			H				
	ZN		200			4.00			I				
Cal Std 3	AL		2000	2.00		Below			J				
	BA		2000			40.0			K				
	BE		50			1.00			L				
	CO		500			10.0			M				
	CU		250			5.00			N				
	FE		1000			Below			O				
	V		500			10.0			P				
Single Metals	MO		1000	1.00		10.0			Q				
	CR		1000	0.800		10.0			R				
	TI		1000	1.00		10.0			S				
Cal Std 4	AS,TL		100	8.00		8.00			T				
	CD,SE		50			4.00			U				
	PB		50			4.00			V				
Single Metals	AL		10000	4.60		500			W				
	FE		10000	3.80		400			X				
	MN		1000	0.700		10.0			Y				
									Z				

*See DOD Summary Attachment for DOD Specific Criteria

RADIAL OPTIMA #1 MRL STANDARD

	Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Exp. Date	Pipet ID
Cal #1	Ca		5000	0.200	1000	1.00	5% HCL 2%HNO3		A				
	K		5000			1.00			B				
	Mg		5000			1.00			C				
	Na		5000			1.00			D				
Cal #2	Cr		100	0.10		0.0100			E				
	Ag		100			0.0100			F				
	Mn		150			0.0150			G				
	Zn		200			0.0200			H				
	Ni		400			0.0400			I				
Cal #3	Al		2000	0.10		0.200			J				
	Ba		2000			0.200			K				
	Fe		1000			0.100			L				
	Co		500			0.050			M				
	V		500			0.050			N				
	Cu		250			0.025			O				
	Be		50			0.00500			P				
Cal #4	Cd , Pb , Se		50	0.10		BELOW			Q				
	As		100			BELOW			R				
PQL #2	B		200	1.00		0.200			T				
	Mo		25			0.0250			U				
	Sn		500			0.500			V				
	Ti		50			0.050			W				
Single Stds	Sb		1000	0.060		0.060			X				
	Sr		1000	0.100		0.100			Y				
	As		1000	0.500		0.510			Z				
	Se		1000	0.500		0.505							
	Pb		1000	0.050		0.055							
	Cd		1000	0.050		0.055							

*See DOD Summary Attachment for DOD Specific Criteria

RADIAL OPTIMA #1 ICSAB STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Int. A Sol'n		Multi	100	1000	Multi			A				
AL		5000			500			B				
CA		5000			500			C				
FE		2000			200			D				
MG		5000			500			E				
Int. B Sol'n		Multi	10.0		Multi			F				
AG		100			1.00			G				
BA		50			0.500			H				
BE		50			0.500			I				
CD		100			1.00			J				
CO		50			0.500			K				
CR		50			0.500			L				
CU		50			0.500			M				
MN		50			0.500			N				
NI		100			1.00			O				
PB		100			1.00			P				
V		50			0.500			Q				
ZN		100			1.00			R				
								S				
								T				
								U				

*See DOD Summary Attachment for DOD Specific Criteria

RADIAL OPTIMA #1 INTERNAL STANDARD

Element	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	ID Letter	Nitric Acid Lot #	Hydrochloric Acid Lot #	Expiration Date	Pipet ID
Y		1000	50	500	100	2% HNO3		A				
Cs		1000	50	500	100	5% HCL		B				
								C				
								D				
								E				
								F				
								G				
								H				
								I				
								J				
								K				
								L				
								M				
								N				
								O				
								P				
								Q				
								R				
								S				

*See DOD Summary Attachment for DOD Specific Criteria

MISCELLANEOUS STANDARDS

Type Of Std	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst/ Date	Letter ID	Nitric Acid Lot#	Hydrochloric Acid Lot#	Expiration Date	Pipet ID
									A				
									B				
									C				
									D				
									E				
									F				
									G				
									H				
									I				
									J				
									K				
									L				
									M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				
									U				
									V				
									W				

*See DOD Summary Attachment for DOD Specific Criteria

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STANDARD OPERATING PROCEDURE

for

METALS DIGESTION,
SOILS, SEDIMENTS, AND SLUDGE FOR ICP-AES, ICP-MS, GFAA ANALYSIS and ICP-MS WATERS

SOP No.: MET-3050B

Revision: 3

January 15, 2003

Approved by: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Manager Date

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1 Mustard Street, Suite 250
Rochester, New York 14609

Annual review of this SOP has been performed and the SOP still reflects current practice.
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1 SCOPE AND APPLICATION

Method 3050 is an acid digestion procedure used to prepare matrices such as soils, sludges, or sediments for analysis by ICP or graphite furnace atomic absorption. It is also used for ICP-MS waters.

2 METHOD SUMMARY

A representative aliquot of sample is digested in nitric acid and hydrogen peroxide. Hydrochloric acid is used as a final reflux acid for ICP analyses. Nitric Acid is used as the final reflux acid for most Graphite Furnace analyses.

3 DEFINITIONS

3.1 **Laboratory Duplicates** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates and indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.2 **Laboratory Control Sample Soil (LCSS)** - An aliquot of a soil to which known quantities of the method analytes are added by an outside vendor. The LCSS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.3 **Matrix Spike** - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results.

3.4 **Preparation Blank (PB)** - An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

3.5 **Digestion Batch** - A digestion batch is no more than 20 samples of the same matrix digested as a unit per day.

4 INTERFERENCES

4.1 See appropriate analysis SOP for applicable interferences

5 SAFETY

Nitric and Hydrochloric acids are extremely corrosive. Care should be taken while working with these chemicals. Personal protective equipment including safety glasses (with side shields), gloves, and lab coat shall be worn when handling samples or reagents.

6 SAMPLE COLLECTION, PRESERVATION AND STORAGE

For non-aqueous samples, glass or plastic sample containers are acceptable. Samples are analyzed within 6 months of sample collection. Additional sample handling policies and procedures are in SMO-GEN.

7 APPARATUS AND EQUIPMENT

7.1 250 and 100 mL beakers
7.2 Ribbed watch glasses
7.3 Hot plates
7.4 Graduated cylinders
7.5 Eppendorf Pipettors
7.6 Funnels
7.7 Mortar and pestle
7.8 Tongue depressors
7.9 Filter paper
7.10 Hot Block Digestor with ETR-3200 Controller by Environmental Express, LTD.
7.11 Graduated block digestor ribbed watch glasses
7.12 Block Digestor Filters.
7.13 CPI MOD Block Digestor

8 PREVENTIVE MAINTENANCE

8.1 All hoods in the Metals Prep Lab are wiped down once a week with DI water. The tops of all digestion hot plates are wiped down daily.

9 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1 Reagent water - ASTM Type II deionized water. Reagent water must be interference free.

9.2 Concentrated nitric acid (Baker Instra-Analyzed 69-70%): Acid should be demonstrated to be free of impurities at levels which would interfere with sample determinations. Store at room temperature in the dark. Expires per manufacturer's indications or one year from receipt, whichever is sooner.

9.3 Concentrated hydrochloric acid (Baker Instra-Analyzed 36.5-38%): Acid should be demonstrated to be free of impurities at levels which would interfere with sample determinations. Store at room temperature. Expires per manufacturer's indications or one year from receipt, whichever is sooner.

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9.4 Hydrogen peroxide (30%) - H_2O_2. Purchased commercially. Should be demonstrated to be free of impurities at levels which would interfere with sample determinations. Store at room temperature. Expires upon manufacturer's indications or 1 year from receipt, whichever is sooner.

9.5 ERA Soil Laboratory Control Sample (LCSS) - Concentrations and Performance Acceptance Limits distributed through vendor. Store at room temperature. Expires upon manufacturer's indications or 1 year from receipt, whichever is sooner.

9.6 Metals spiking solutions – Purchased commercially. See Table 1. Store at room temperature. Stocks expires upon manufacturer's indications or 1 year from receipt, whichever is sooner. Solutions prepared from stocks expire 6 months from preparation. All target elements needed for client samples in the batch are added to the LCS and MS.

9.7 LCS – for ICP-MS waters – add appropriate spiking solutions to DI.

9.8 MS – Add appropriate spiking solutions to client sample.

9.9 Method Blank – Analyze DI as a sample.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

11 PROCEDURES

11.1 HOT PLATE

11.1.1 Mix the sample thoroughly to achieve homogeneity using a tongue depressor or the mortar and pestle.

11.1.2 Weigh (to the nearest 0.01g) 1.00g to 1.50g of sample into a 250 or 100 mL beaker. For sludges and sediments that have a high moisture content, use more sample. The goal is to use about 1g of dry weight sample. At this point add the appropriate spiking solutions (see Table 1) directly onto the designated spike sample prior to addition of reagents.

11.1.3 Unless specified by project or state requirements, the addition of acid should be as follows: Add 10ml of 1:1 HNO_3, cover with a ribbed watch glass and reflux for 15 minutes. The sample temperature should be 90-95°C. Allow the sample to cool,

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then add 5ml of concentrated HNO_3, cover and reflux for 30 minutes. Repeat the addition of 5ml of HNO_3 and reflux to 5 mLs. Do not allow the sample to go to dryness. CAUTION: Do not boil. Antimony is easily lost by volatilization.

11.1.4 Cool the sample and add 2ml of DI and 3ml of 30% H_2O_2. Cover and heat to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive effervescence. Heat until effervescence subsides and cool the beaker.

11.1.5 If the effervescence does not subside, add 3 mLs of hydrogen peroxide with warming to each of the samples (including blanks and LCSs) in the batch. If necessary, continue to add 30% H_2O_2 in 1ml aliquots with warming until the effervescence is minimal, or until the general sample appearance is unchanged. Do not add more than 10ml of 30% H_2O_2.

11.1.6 If the sample is being prepared for analysis by ICP, add 10 mL 1:1 HCL. If the sample is being prepared for analysis by Graphite Furnace no HCl is added.

11.1.7 Cover and reflux the ICP samples for 15 minutes without boiling. Allow to cool.

11.1.8 Prepare filters by rinsing with 1:1 nitric acid and DI.

11.1.9 All samples are diluted to 100 mLs with DI. Quantitatively transfer the digestate to a graduated cylinder by pouring the sample through a prepared filter into the cylinder and rinsing the beaker and watch glass with DI into the filter. Rinse the filter with DI. Bring to volume with DI.

11.2 HOT BLOCK DIGESTOR

11.2.1 Set the temperature on the Block Digestor to a temperature that brings the sample temperature to 90-95°C without boiling.

11.2.2 The Hot Block is on a timer which can be set to turn on and off whenever necessary. To set timer press the timer button and choose the days M-F (Monday through Friday). Then choose the hour and minutes to start and stop the Block Digestor.

11.2.3 Label graduated hot block digestor sample cups with appropriate sample IDs for digestion.

11.2.4 Mix the sample thoroughly to achieve homogeneity using a tongue depressor or the mortar and pestle.

11.2.5 Weigh (to the nearest 0.01g) 1.00g to 1.50g of sample into labeled digestor sample cup. For sludges and sediments that have a high moisture content, use more sample. The goal is to use about 1g of dry weight sample. At this point add the appropriate

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spiking solutions (see Table 1) directly onto the designated spike sample prior to addition of reagents.

11.2.6 Unless specified by project or state requirements, the addition of acid should be as follows: Add 10ml of 1:1 HNO_3 and for ICP only add 1.5 mL of 1:1 HCl, cover with reflux cap and reflux for 15 minutes. The sample temperature should be 90-95°C. Allow the sample to cool, then add 5ml of concentrated HNO_3, cover and reflux for 30 minutes. Repeat the addition of 5ml of HNO_3 and reflux to 5 mLs. Do not allow the sample to go to dryness. CAUTION: Do not boil. Antimony is easily lost by volatilization.

11.2.7 Cool the sample and add 2ml of DI and 3ml of 30% H_2O_2. Cover and heat to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive effervescence. Heat until effervescence subsides and cool the sample cup.

11.2.8 If the effervescence does not subside, add 3 mLs of hydrogen peroxide with warming to each of the samples (including blanks and LCSs) in the batch. If necessary, continue to add 30% H_2O_2 in 1ml aliquots with warming until the effervescence is minimal, or until the general sample appearance is unchanged. Do not add more than 10ml of 30% H_2O_2.

11.2.9 If the sample is being prepared for analysis by ICP, add 10 mL 1:1 HCL. If the sample is being prepared for analysis by Graphite Furnace no HCl is added.

11.2.10 Cover and reflux the ICP samples for 15 minutes without boiling. Allow to cool.

11.2.11 Prepare filters by rinsing with 1:1 nitric acid and DI.

11.2.12 All samples are diluted to 100 mLs with DI. Filter if particulates are suspended. If particulates have settled, no filtering is necessary. Filtering is usually required if analyzed on the same day as digestion. If filtering, quantitatively transfer the digestate to a graduated cylinder by pouring the sample through a prepared filter into the cylinder and rinsing the hot block cup and reflux cap with DI into the filter. Rinse the filter with DI. If not filtering, quantitately transfer digestate to a graduated cylinder rinsing hot block cup with DI. Bring to volume with DI. Pour into a labeled B-cup.

12 QA/QC REQUIREMENTS

12.1 Each day, digest one laboratory control sample (LCS) per digestion batch, or per 20 samples, or per EPA SDG group, whichever is more frequent. Use the appropriate solid laboratory control sample (LCSS) for soils analysis.

12.2 Each day, digest one blank per digestion batch, or per 20 samples, or per EPA SDG group, whichever is more frequent. Use D.I. water and follow the digestion procedures.

12.3 Each day, prepare one duplicate and one spiked sample with each digestion batch, or per twenty samples, or per EPA SDG group, whichever is more frequent. At times, specific samples will be assigned as duplicates of spikes depending on client requirements.

12.4 Matrix spikes are prepared by adding the appropriate volume of spiking solution (See Table 1).

12.5 See appropriate analysis SOP for applicable QC limits and corrective action.

13 DATA REDUCTION AND REPORTING

13.1 Digestion logs are used to record all sample volumes, spike volumes, etc. The Manufacturer's lot number for the reagents used are added to the digestion log (see attached digestion log benchsheet).

13.2 Reporting and method performance is discussed in the appropriate analysis SOP. Data review is discussed in ADM-DREV.

14 METHOD PERFORMANCE

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

15 WASTE MANAGEMENT AND POLLUTION PREVENTION

15.1 Reagents are prepared upon an as-needed basis in small quantities. Minimum sample volumes are used during analysis.

15.2 Acidic waste is poured down the drain with copious amounts of water.

15.3 Samples with analyte concentrations exceeding TCLP regulatory limits are disposed of as hazardous waste. Others are dumped down the drain with plenty of water. See SMO-SPLDIS.

16 CORRECTIVE ACTION FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17 CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

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If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18 REFERENCES

"Test Methods For Evaluating Solid Waste, Physical/Chemical Methods". EPA SW846, Third Edition, December 1996.

19 TRAINING OUTLINE

19.1 Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2 Observe Sample Preparation.

19.3 Participate in the methodology, documentation, and data reduction with guidance.

19.4 Complete a Training Plan Form for the procedure.

19.5 Show Initial Demonstration of Capability (IDC) by independently preparing and digesting four LCSs, or equivalent, according to the test method either concurrently or over a period of days. If recovery is within acceptable limits, complete IDC certification form, and Training Plan forms and file with QA. Continued capability shall be demonstrated annually using PE results, a single blind, or a new 4 replicate study.

20 METHOD MODIFICATIONS

None

21 INSTRUMENT-SPECIFIC ADDENDUM

Not Applicable

22 ATTACHMENTS

Table 1 Spike Concentrations
Digestion Log Benchsheets
SW846 Method 3050 Flow Chart

23 CHANGES FROM PREVIOUS REVISION

- Added Hot Block digestion procedures (11) and associated items to Apparatus and Equipment (7)
- Added sections 14, 16, 17, and 20 for NELAP compliance

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- Changed the amount of time to reflux sample from 10-15 minutes to just 15 minutes after the first addition of acid (11).

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Table 1 Spiking Concentrations for LCS and MS Samples

SPIKE SOLUTION A			1.00ml Spk A	to Final Vol of 100ml
Metal	***Conc. (ug/mL)***		***Metal***	***Conc. (ug/mL)***
AL	200		**NI**	50
AS	4		**SE**	1
BA	200		**AG**	5
BE	5		**TL**	200
CD	5		**V**	50
CR	20		**ZN**	50
CO	50		**B**	100
CU	25		**CA**	200
FE	100		**MG**	200
PB	50		**NA**	2000
MN	50		**K**	2000

SPIKE SOLUTION B			1.00ml Spk B	to Final Vol of 100ml
Metal	***Conc. (ug/mL)***		***Metal***	***Conc. (ug/mL)***
SB	50		**TI**	50
MO	50		**-**	**-**

INDIVIDUAL METALS	0.10ml Spk. to Final Volume of 100ml		**INDIVIDUAL METALS**	0.5ml Spk. to Final Volume of 100ml
Metal	***Conc. (ug/mL)***		***Metal***	***Conc. (ug/mL)***
SE	1000		**SN**	1000

SPIKE #4 **Furnace Spike**			1.00ml Spk #4	to Final Vol of 100ml
Metal	***Conc. (ug/mL)***		***Metal***	***Conc. (ug/mL)***
AS	4		**SB**	10
PB	2		**TL**	5
SE	1		**CU**	0.5

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CAS-Rochester ICP Soil Digestion Log

6010B/846 // 200.7/136 // ASP/CLP4.1

Batch ID:

Analyst: _____ **Date:** _____

Spike Witness / Lot Approval: _____

Prep Method: SW846 3050 // CLP

Batch Temp:

Digest: Initial // Redigest of: _____

Report Type: Routine // ASP // Pkg5

	Submission / Order #	Initial Wgt. (g)	Final Vol (ml)	Initial Color / Texture	Final Color / Clarity	Metals	Spike Vol (ml)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							

Spiking Standards / Reagent Lot #:

Spike A,B: _____ Spike #4: _____
TCLP Spk: _____ TCLP Ba: _____
Se Std: _____ Sn Std: _____
HNO3: _____ HCL: _____
H2O2: _____ LCSS: _____

Color / Clarity Key:
Color: **C** = Colorless ; **Y** = Yellow ; **B** = Brown
BL = Black ; **G** = Grey ; **W** = White
Clarity: **CDY** = Cloudy ; **CLR** = Clear ; **OP** = Opaque
Texture: **F** = Fine ; **M** = Medium ; **CS** = Coarse ; **NAQ** = Non Aqueous

01

Comments: _____

CAS-Rochester Furnace Soil Digest Log

Analyst:_____ **Date:**_____ **Spike Witness:**_____ **Approval:**_____

Method: SW846 3050 // CLP Digest: Initial Digestion // Redigestion of _____

Report: Routine // ASP // Pkg.5 **6010B/846 // 200.7/136 // ASP/CLP**

	Client/ Order #	Initial wgt(g)	Final vol(ml)	Initial Color/Clarity	Final Color/Clarity	Metals	Spike vol(ml)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							

Spiking Standards Lot # or Prep Date:
Spike #4_____
Surrogent Lot #:
LCSS_____ HNO3_____ H2O2_____

Comments/Problems:_____

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SW846 Method 3050
Flow Chart

Non-CLP Soils, Sediments and Sludges

Mix sample. Weigh 1.0-1.5g of sample. Add 10mL (1:1) HNO3 and for ICP only add 1.5 mL 1:1 HCl, mix to a slurry and cover with a watch glass.

↓

Gently reflux 15min.

↓

Cool

Add 5mL conc. HNO3 and reflux for 30 min. Repeat.

↓

Evaporate to 5mL.

↓

Add 2mL DI and 3mL 30% H2O2. Warm gently to start effervescence.

↓

If efferv. doesn't subside, add 3mL portion of 30% H2O2 (followed by warming). Add 1 mL portions until efferv subsides. Don't add more than a total of 10mL 30% H2O2.

↓

Type of Digest

Furnace

↓

Evaporate to approximately 5mL

ICP

↓

Add 5mL conc. HCL and 10mL DI Reflux 15min.

↓

Filter into grad cylinder, Collecting DI rinses of beaker and watch glass

↓

Dilute to 100mL with DI water.

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STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (ICP-MS); EPA METHOD 6020

SOP No.: MET-6020

Revision: 3

July 24, 2006

Approved by: _____ _____
Supervisor Date

_____ _____
QA Manager Date

_____ _____
Laboratory Manager Date

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1 Mustard Street, Suite 250
Rochester, NY 14609

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NUMBER: _____
Initials: _____ Date: _____

*See DOD Summary for DOD specific criteria.

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1 SCOPE AND APPLICATION

This SOP uses EPA SW846 Method 6020 for the determination of the concentrations of certain elements in water, soil, aqueous and non-aqueous wastes, and sediment samples. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

2 SUMMARY OF METHOD

2.1 Prior to analysis, samples must be digested using appropriate sample preparation methods. These methods may include SW 846 Methods 3020A or 3050B as described in the appropriate SOPs. The digestate is analyzed for the elements of interest using ICP spectrometry.

2.2 Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. The analytes and method reporting limits (MRLs) are listed in Table 1.

3 DEFINITIONS

3.1 **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2 **Independent Calibration Verification (ICV)** (also called Second Source Calibration Verification) - ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.3 **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

3.4 **Duplicate Sample** (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

*See DOD Summary for DOD specific criteria.

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3.5 **Laboratory Control Sample (LCS)** - An aliquot of to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.6 **Preparation Blank** (also called Method Blank)- The prep blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.7 **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.8 **Instrument Blank (ICB/CCB)** - The instrument blank (also called initial or continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the ICB/CCB is to determine the levels of contamination associated with the instrumental analysis.

3.9 **Batch** – A group of no more than 20 samples prepared together or analyzed together on the same day with the same reagents.

3.10 **Calibration Blank** - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to calibrate the ICP instrument.

3.11 **Instrument Detection Limit (IDL)** - The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 7 replicate measurements of the calibration blank signal at the same wavelength.

3.12 **Internal Standard** - Pure analyte(s) added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component

3.13 **Linear Range** - The concentration range over which the instrument response to an analyte is linear.

3.14 **Method Detection Limit (MDL)** - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.15 **Interference Check Solution (ICS)** - A solution of selected method analytes of higher concentrations which is used to evaluate the procedural routine for correcting known interelement spectral interferences with respect to a defined set of method criteria.

*See DOD Summary for DOD specific criteria.

3.16 **Tune** – The analysis of a standard element to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis.

4 INTERFERENCES

4.1 Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isotope.

4.2 Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Refer to Method 6020 for further discussion.

4.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Total solid levels below 2000 mg/L have been recommended to minimize solid deposition on the nebulizer tip. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

4.4 Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate these interferences.

5 SAFETY

5.1 All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2 Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3 Hydrochloric and Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring

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acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4 The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

5.5 High Voltage - The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

5.6 UV Light - The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

5.7 Refer to the Safety Manual for further discussion of general safety procedures and information.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Solid samples require no preservation prior to analysis other than storage at 0-6°C. Sample containers may be glass or plastic. When the laboratory provides the sample containers, the containers are glass soil jars with Teflon-lined lids.

6.2 For aqueous samples, glass or plastic sample containers are acceptable. When the laboratory provides the sample containers, the containers are 500 mL plastic bottles. Sample volume should be acid preserved with (1+1) nitric acid to pH <2. Samples are held at room temperature (although refrigeration is acceptable also).

6.3 For the determination of the dissolved elements, the sample must be filtered through a 0.45 µm pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. (Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus should be used when the determinations of boron and silica are critical.) Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) nitric acid immediately following filtration to pH <2

Note: When the nature of the sample is either unknown or is known to be hazardous, acidification should be done in a well ventilated area or fume hood

6.4 Samples are generally received in the ICP-MS laboratory as 1% Nitric Acid digestates. Digestates are stored at room temperature in hotblock cups. There is no specific holding time from digestion to analysis. Client samples must be analyzed within 6 months of collection.

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6.5 Following analysis, digestates are stored until all results have been reviewed. Digestates are diluted and disposed of through the sewer system in approximately 90 days after receipt of sample.

6.6 For more information about custody, sample handling, and storage procedures, see SMO-GEN.

7 APPARATUS & EQUIPMENT

7.1 Instrument: Perkin Elmer Elan 9000 ICP-MS System – The system has a mass range from at least 6 to 240 amu and a data system which allows corrections for isobaric interferences and the application of the internal standard technique.

7.2 Peristaltic Pump and pump tubing

7.3 15 and 50 mL autosampler tubes

7.4 Pipettes (Eppindorf)

*See DOD Summary for DOD specific criteria.

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8 PREVENTIVE MAINTENANCE

8.1 Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. CAS maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The data file name of the first acceptable run after maintenance is to be documented in the maintenance log.

8.2 Preventive maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Other maintenance or repairs may, or may not require factory service, depending on the nature of the task.

- cone removal and cleaning
- removal and cleaning of ICP glassware and fittings
- checking air filters and cleaning if necessary
- checking the rotary pump oil and adding or changing if necessary
- removal and cleaning of extraction lens
- removal and cleaning of ion lens stack

9 STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

9.1 **Standards Preparation General Information and Disclaimers**

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mLs or may be made by adding 4 mL of 50 mg/L to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

*See DOD Summary for DOD specific criteria.

9.2 Argon – 99.99% purity

9.3 Trace metals grade chemicals shall be used in all tests. Each lot of acid used is to be analyzed to demonstrate that it is free of interference before use. See ADM-CTMN. All acids are stored at room temperature. Purchased concentrated acids expire 3 years from receipt.

9.3.1 Hydrochloric acid (conc.), HCl. Purchased commercially

9.3.2 Nitric acid (conc.), HNO_3. Purchased commercially.

9.3.3 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriately sized beaker. Expires one year from preparation.

9.3.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL water and dilute to 1 liter in an appropriately sized beaker. Expires one year from preparation.

9.4 Reagent Water. All references to water in the method refer to DI Type II water unless otherwise specified. Reagent water will be interference free.

9.5 The elements, concentrations and additional preparatory information for the following standards are provided in Appendix I. They are all stored in plastic at room temperature.

9.5.1 Mixed Calibration Standards are prepared by combining appropriate volumes of the stock solutions in volumetric flasks. Matrix match with the appropriate acid and dilute to 100ml with water. Calibration standards should be verified using a second source quality control sample (LCS, ICV, or CCV). Calibration standards expire in 1 month.

9.5.2 Initial and Continuing Calibration Verification (ICV and CCV) Standards are prepared by combining compatible analytes at concentrations equivalent to the midpoint of their respective calibration curves. The ICV and CCV standards should be prepared from a separate source independent from that used in the calibration standards. ICV / CCV standards expire in 48 hours.

9.5.3 MRL Standards are prepared to contain known concentrations of elements at or near* the Method Reporting Limit. MRL standards expire in 1 month.

9.5.4 Interference Check Solutions A and AB are prepared to contain known concentrations of interfering analytes that will provide an adequate test of the correction factors. ICSA / ICSAB standards expire in 1 week.

9.5.5 Laboratory Control Sample and Matrix Spike are purchased as custom mixes at the concentrations recommended in the method. Solutions expire per manufacturer recommended expiration date. Each LCS sample, up to 100 mL DI, is spiked with 0.2 ml of spike solution. All target analytes are spiked in the LCS and MS.

*See DOD Summary for DOD specific criteria.

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9.5.6 Internal Standards Solutions-high purity grade solutions are purchased and diluted to final concentration of 1000-5000 ppb. Diluted solutions expire in 6 months. Each 5 ml aliquot of blanks, standards, and samples are spiked with 50 uL of internal standard solution prior to analysis.

9.6 Blanks

9.6.1 Method Blanks must contain all the reagents and in the same volumes as used in the preparation of samples. The method blanks are DI samples carried through the complete procedure and contain the same acid concentration in the final solution as the samples.

9.6.2 The Calibration Blank is prepared by acidifying reagent water to the same concentrations of acid found in the standards and samples.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

11 PROCEDURE

11.1 Digest samples with the appropriate digestion method. For waters, use MET-CLP-DIG. For soils, use MET-3050. Digestates originating from soil samples are diluted prior to instrumental analysis to avoid interferences and allow the analysis to achieve maximum sensitivity which results in optimum Reporting Limits.

11.2 Refer to method 6020 (Section 7.0) and the instrument manuals for detailed instruction on implementation of the following procedures preceding an analytical run. All of the following are done daily prior to initial calibration unless otherwise specified.

11.3 Initiate plasma and allow a warm up of at least 30 minutes. The tuning procedures may be carried out during warm-up.

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11.4 Open the EPA Tune Method and optimize the following parameter using the 10 ppb tuning solution containing Be, Mg, Co, Rh, In, Ba, Ce, Pb

11.4.1 Set RF power to desired level

- 1100-1200 watts for clean water
- 1300-1400 watts for soil and sediment digests.

11.4.2 Optimize the nebulizer argon flow

11.4.3 Optimize the static lens voltage

11.4.4 Save the optimization file

11.5 Open the EPA Lens Calibration method and perform the autolens calibration using 10 ppb tuning solution. This is done daily – it is performed as needed.

11.5.1 Clear old calibration

11.5.2 Get analytes

11.5.3 Optimize (Takes about 6 minutes)

11.5.4 Save file

11.6 Open the EPA 6020 Dual Detector calibration method and aspirate a solution twice the calibration range. All elements plus internal standard elements should be present.

Note: This procedure is required when detector voltages or a new detector is installed. CAS does periodically

11.6.1 Clear old calibration

11.6.2 Get analytes

11.6.3 Optimize (Takes about 20 minutes)

11.6.4 Save file

*See DOD Summary for DOD specific criteria.

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11.7 Open the EPA Tune Method

11.7.1 Aspirate 10 ppb tuning solution

11.7.2 Click on Tune Mass Spec button in Tuning Window

11.7.3 Check mass calibration. Measured mass must be within 0.05 amu of actual mass. The resolution must be <0.9 amu full width at 10% peak height. Save and print tune. Put one copy of the tune with the analytical run and another copy in the tune binder.

11.8 Open EPA Daily Method

11.8.1 Aspirate the 10 ppb tuning solution

11.8.2 Click on the Analyze button to acquire

11.8.3 Check that the RSDs for the five replicates are less than 5%

11.8.4 Monitor daily performance measures as recommended by Perkin Elmer for Rh sensitivity, background, % double charged, and % oxide levels.

- Rh>150000 cps for 10 ppb
- Background @ mass 200<30cps
- % double charged <4%
- % oxides <4%

Oxides and double charged levels can be reduced by slightly decreasing the nebulizer flow rate.

11.8.5 If the tune and Daily does not meet criteria, retune the instrument and reanalyze tuning solution. The tune and Daily must pass before proceeding.

*See DOD Summary for DOD specific criteria.

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11.9 Analytical Run

11.9.1 Open desired method. Enter sample information. Enter pump control speeds for all samples and save sample file. Re-open the sample file (this must be done for batch QC to run properly) and highlight the row numbers to be analyzed. Select "analyze batch."

11.9.2 Calibration is done daily using 3 standards and a blank for each analyte. Following the calibration, the sequence of the run shall be as follows:

- ICV (QC Std 1)
- ICB (QC Std 2)
- MRL (QC Std 3)
- ICSA (QC Std 4)
- ICSAB (QC Std 5)
- CCV (QC Std 6)
- CCB (QC Std 7)
- Analyze 10 samples (including Method Blanks (Prep. Blank), LCSs, MS/Duplicate, and Serial dilutions)
- CCV (QC Std 6)
- CCB (QC Std 7)

- Repeat sequence as required to complete analytical run, analyzing CCVs/CCBs every 10 samples and MRL after each batch. The sequence is closed with a CCV/CCB.

- Include within each batch the batch QC including LCS, Prep Blank, DUP/MS and Serial Dilution Test

- Dilute and reanalyze samples which are above the linear range of the instrument or measure an alternate, less-abundant isotope (if calibrated)

12 QA/QC REQUIREMENTS

12.1 **Initial Calibration** – performed daily. The correlation coefficient must be greater than 0.995 for each analyte. If the correlation is less than 0.995, recalibrate the instrument prior to analyzing any samples.

12.2 **Continuing Calibration Verification Standard (CCV)** Analyzed after every 10 samples and at the end of the analytical sequence. The results of the CCV must agree within ±10% of the expected value. If the control limits are exceeded, correct the problem and reanalyze the CCV. If that fails, recalibrate the instrument. Repeat any samples bound by the out of control CCV.

*See DOD Summary for DOD specific criteria.

12.3 **Continuing Calibration Blank (CCB)** Analyzed after every 10 samples (immediately following the CCV. The CCB must be less than the reporting limit*. If the control limits are exceeded, correct the problem and reanalyze the CCB. If that fails, recalibrate the instrument. Repeat any samples bound by the out of control CCB. If CCB results are exceeded, these associated data shall be flagged.

12.4 **Initial Calibration Verification Standard (ICV)** Analyzed immediately after the calibration using a second source standard to verify the standards in the calibration curve. The results for each analyte must agree within 10% of the expected value. If not, the analyses should be terminated and instrument recalibrated. Investigate the sources and preparation procedures of the standards.

12.5 **Method Reporting Limit (MRL) Standard** A standard at or near the MRL is analyzed at the beginning and end of each analytical run but not before the ICV. There are no limits in Method 6020, but the CAS guideline used is +/- 20% of the true value*. If the limits are not met the analysis is stopped and the instrument is recalibrated.

12.6 **DUP -** analyzed at a frequency of 5% or one per batch, whichever is greater. The relative percent difference will be calculated using the equations listed in Section 9 of the QA Manual. The control limits are listed in Appendix C of the Quality Assurance Manual. Client specific QC recoveries may supercede the limits listed in the QA manual. If the control limits are exceeded, the data will be reported with a qualifier.

12.7 **MS** - analyzed at a frequency of 5% or one per batch, whichever is greater. The matrix spike recovery and relative percent difference will be calculated using the equations listed in Section 9 of the QA Manual. The control limits are listed in Appendix C of the Quality Assurance Manual. Client specific QC recoveries may supercede the limits listed in the QA manual. If the control limits are exceeded, analyze a post-digestion spike. DOD requires project-specific DQOs be examined or contact the client for additional measures. See Table B-6.

12.8 **LCS** is analyzed at a frequency of one per preparation batch, not to exceed 20 samples. The % recovery must be within the limits in Appendix C of the Quality Assurance Manual. Client specific QC recoveries may supercede these limits. If the control limits are exceeded, the associated batch of samples will be redigested and reanalyzed for the out of control elements or the data is to be flagged. Exception – if the LCS recovery is too high, elements which are less than the reporting limit may be reported.

12.9 **IDLs** are performed quarterly*. Analyze 7 replicates of a low level standard. Repeat twice more for a total of 21 replicates over 3 non-consecutive days. These will be calculated and made available to the ICP-MS operator. The IDL must be less than the MRL*. If the IDL does not meet limits, correct the problem and reconduct the IDL study or raise the reporting limits.

*See DOD Summary for DOD specific criteria.

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12.10 **Linear Range Study** – performed every 6 months. A high level check standard must be within 10% of the expected value. If it is not, repeat the linear range study or reduce the linear range and analyze a new high level check standard.

12.11 **MDLs** are determined annually. See Section 14.

12.12 **Preparation Blank or Method Blank (PB)** Preparation Blanks must be analyzed with each batch of 20 or fewer samples of the same matrix. PB values must not exceed the MRL*. Fresh aliquots of the samples must be prepared and analyzed again for affected analytes after the source of the contamination has been corrected and acceptable PB values have been obtained. If detections are greater than the limit, the batch needs to be redigested if sample concentration is less than 10 times the concentration found in the prep blank. If the sample concentration is less than the reporting limit*, the sample does not require redigestion. Affected sample data shall be flagged or the samples shall be redigested.

12.13 **Internal standards**– The intensity of each internal standard in the CCV, CCB must fall within 80-120%. If this is not met, analysis should be terminated, problem corrected, instrument recalibrated, and samples reanalyzed. The intensity of each internal standard in samples must fall within 30-120% of the intensity of the internal standard in the initial calibration. If they do not in the samples, verify that the instrument is not drifting by evaluating the internal standards in the CCBs and dilute the sample five fold and reanalyze with the proper addition of internal standards. Repeat this procedure until the internal standard intensities fall within the limits. If they do not for the standards, terminate the analysis.

12.14 **Dilution Test (serial dilution)** – One dilution test must be performed for each batch of 20 or fewer samples. To perform the Dilution Test, choose a sample which has a concentration within the linear range of the instrument and dilute it 1/5. The result of the dilution must agree within 10% of the original sample determination when concentrations exceed 50X IDL*. If it does not,*an interference effect must be suspected and should be noted in the case narrative.

12.15 **Post digestion spike** – Perform when the MS fails*. The Post Digestion Spike recovery should be 75-125% of the true value. If it is not the sample must be diluted and reanalyzed to compensate for the matrix effect. Results must agree within 10% of the original determination. The use of standard-addition may also be used to compensate for this effect.

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12.16 **Interference Check Standard (ICS) –**

- Frequency - Solutions A and AB are analyzed at the beginning of each analytical run or every 12 hours, which ever is more frequent.
- Limits –
  - ICS-A - the absolute value of concentration for all non-spiked analytes must be less than 2 times the MDL (unless they are a verified trace impurity from one of the spiked analytes).
  - ICS-AB - The analytes in ICS-AB should recover within 20% of the expected value. If the analytes are not present, monitor concentration for possible interferences.
- Corrective action – Terminate analysis, locate and correct the problem, reanalyze ICS, reanalyze all affected samples. If corrective action fails, qualify the associated sample data.

13 DATA REDUCTION, REPORTING, AND REVIEW

13.1 Calculations

Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = \frac{C^{*}\ x\ Dilution\ Factor\ x\ FinalDigestateVolume(ml)}{InitialVolumeDigested(ml)}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

Solid samples are reported in ug/Kg:

$$ug/Kg(Sample) = C^{*}\ x\ PostDigestionDilutionFactor\ x\ \frac{DigestionVol.(ml)}{Samplewt.(g)}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

13.2 Common isobaric interferences are corrected using equations equivalent to those listed in EPA Methods 6020 and 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA

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Method 6020 and 200.8 for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3 Data is reviewed according to ADM-DREV.

14 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

Demonstration of Capability is performed upon instrument set-up, whenever a new analyst begins independent analysis and annually thereafter according to ADM-TRANDOC and Section 19. This documentation of Method Performance is retained by the Quality Assurance office.

15 WASTE MANAGEMENT AND POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of reagents used to perform this method wherever technically sound, feasibly possible and within method requirements. Standards are prepared in volumes consistent with the laboratory use in order to minimize the volume of expired standards to be disposed.

16 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17 CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

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18 REFERENCES

18.1 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update II, Method 6020, Revision 0, September 1994.

18.2 Method 200.8 - Determination of Trace Elements in Waters and Wastes By Inductively Coupled Plasma-Mass Spectrometry, USEPA-EMSL, Revision 5.4, 1994.

18.3 Perkin Elmer Instrument Manuals

19 TRAINING OUTLINE

19.1 Read current SOP. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2 Observe Sample Preparation and Analysis. Follow Training Plan Form.

19.3 Participate in the methodology, documentation, and data reduction with guidance.

19.4 Perform Initial Demonstration of Capability by performing the analysis independently and analyzing a known standard four times. If recovery is within acceptable limits, complete Training Plan Form and IDC certificate and file with QA. Continuing proficiency shall be demonstrated annually using an outside PE source, an internal unknown, or a new 4 replicate study.

20 METHOD MODIFICATIONS

20.1 The concentration of interfering elements in the ICSA and ICSAB solutions are spiked at levels recommended by the manufacturer due to instrument sensitivity.

20.2 Section 9.8.4 of the method states that the CCB must be controlled to <3 times the IDL. The IDL, which are calculated from the analysis of reagent blank, are in most cases too low to be used as control criteria for analyzing environmental samples. The effects of sample matrices on the baseline values can be great in relation to the IDLs. The CCBs are controlled to the CAS RL* (thus minimizing any negative impact on the data) and in cases where the CCB value is greater than the RL the effected data is evaluated for usability (i.e. are the sample levels greater than 20 times the level in the CCB.).

21 INSTRUMENT ADDENDUM

None

*See DOD Summary for DOD specific criteria.

22 ATTACHMENTS

- DOD Summary
- Table 1 Analytes and Reporting Levels
- Appendix I Standard Preparation Instructions

23 CHANGES FROM PREVIOUS REVISION

- Added DOD Summary, Table B-6, and DOD requirements throughout.
- 3-Added definition for Tune
- 5-Modified Safety for consistency with other SOPs
- 6-Added info for collection, preservation and storage of field samples instead of only the digestates. Moved statement about diluting soil digestates to section 11. Added reference to SMO-GEN
- 8-expanded upon Maintenance Log for consistency
- 9-added Standards Prep and General Information section
- 9-noted that all targets are spiked in LCS and MS.
- 9-noted that DI is used for LCS and MB
- 11 – added that all of the startup procedures are done daily unless otherwise indicated
- 11.9.2 Added that the analytical sequence is closed with a CCV/CCB
- 12-Added CCB frequency and limit
- 12-Change the limits for ICSA (they were incorrectly listed in the SOP- there is no change for the lab)
- 12-Added tuning limit of <0.9 amu full width at 10% peak height and added corrective action
- 12-Added LDR limits
- 12.8-Added exception for LCS high recovery (with < results)
- 12-added IDL procedure instead of referencing 6020. The method in 6020 uses blanks.
- 12-added that the PDS is analyzed when MS fails.

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*DOD SUMMARY

For work for the Department of Defense – the DOD Quality Systems Manual must be followed. The DOD Manual is based on the NELAC Standards with some additional requirements. The exact wording is in Table B6. The following are the requirements which are different or additional to routine analysis and must be followed for DOD work:

- The Second Source Calibration Verification (ICV) must have a recovery of 80-120% of the true value.
- The CCB must be less than twice the MDL.
- The Method Blank must be less than ½ the reporting limit (<RL for common laboratory contaminants).
- Apply J flag to all hits between MDL and MRL.
- Reporting Limits – "the lowest standard of the calibration establishes the [reporting limit], but it must be greater than or equal to 3 times the [MDL]". DoD QSM, V3, Appendix B-Glossary
- The limits for LCS and MS are 80-120%. All targets are spiked and evaluated.
- The IDL must be less than the MDL. The IDL study is only required by DoD at set-up and after significant change.
- Low Level Check Standard (MRL standard) must be spiked at or below the reporting limit and it must have a recovery of 80-120% of the true value when analyzed at the beginning of the run.
- ICS-A absolute value of concentration for all non-spiked analytes must be <2 times MDL (unless they are a verified trace impurity from one of the spiked analytes).
- Serial Dilution Test
    - Only for samples >100 times MDL
    - Corrective Action – If the limits are not met, perform a post digestion spike addition.
- Post digestion spike – Perform when dilution test fails or analyte concentration for all samples <100 times MDL.
- Method of Standard Additions – use when matrix interference is suspected. Document use in the case narrative.

*See DOD Summary for DOD specific criteria.

TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method (see Appendix C)	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).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-18)	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument noise level.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study (see box D-18).	NA	Samples cannot be analyzed without a valid MDL.
IDL study	At initial set-up and after significant change	Detection limits established shall be ≤ MDL.	NA	NA	Samples cannot be analyzed without a valid IDL.
Tuning	Prior to initial calibration	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analytes	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) **(minimum one high standard and a calibration blank)**	Initial calibration prior to sample analysis	If more than one calibration standard is used, r ≥ 0.995	Correct problem, then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each ICAL, prior to beginning a sample run	Value of second source for all analytes within ± 10% of expected value (initial source)	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Low-level calibration check standard	Daily, after one-point initial calibration	Within ± 20% of expected value	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ± 10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration verification.	Flagging criteria are not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Linear dynamic range or high-level check standard	Every 6 months	Within ±10% of expected value	NA	NA	
Method blank	One per preparatory batch	No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected > RL.	Correct problem, then see criteria in box D-5. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 x MDL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning ofan analytical run	ICS-A: Absolute value of concentration for all non-spiked analytes < 2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ±20% of expected value	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all affected samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	
LCS containing all analytes required to be reported	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-7 and Appendix DoD-D	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. (See full explanation in Appendix DoD-D.)	If corrective action fails, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	

TABLE B-7. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETRY (METHOD 6020) (CONTINUED)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Dilution test	Each preparatory batch	Five-fold dilution must agree within ± 10% of the original measurement	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 100 x MDL.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples < 100 x MDL	Recovery within 75-125% of expected results	Run samples by method of standard additions (MSA) or see flagging criteria.	Apply J-flag to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition.	
Method of standard additions (MSA)	When matrix interference is suspected	NA	NA	NA	Document use in the case narrative.
MS	One MS per preparatory batch per matrix (see box D-15)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	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 J-flag 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.
MSD or sample duplicate	One per preparatory batch per matrix	RPD < 20% (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 J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Internal standards (IS)	Every sample	IS intensity within 30-120% of intensity of the IS in the initial calibration	Perform corrective action as described in Method 6020 (8.3).	Flagging criteria are not appropriate.	
Results reported between LOD and LOQ	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

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TABLE 1

Analyte	MRL (ug/L)	MRL (ug/g)
Aluminum	10	2.0
Antimony	1	0.20
Arsenic	1	0.10
Barium	1	0.10
Beryllium	1	0.10
Cadmium	1	0.10
Chromium	1	0.10
Cobalt	1	0.50
Copper	1	4.0
Lead	1	0.10
Manganese	1	0.10
Molybdenum	1	0.20
Nickel	1	0.10
Selenium	1	0.20
Silver	1	0.10
Thallium	1	0.10
Vanadium	1	0.10
Zinc	10	4.0

*See DOD Summary for DOD specific criteria.

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APPENDIX I

STANDARD PREPARATION INSTRUCTIONS

*See DOD Summary for DOD specific criteria.

ICP-MS CALIBRATION STANDARD #1
(PREPARED MONTHLY OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Cal Std 1	SB		20000	0.250	500	10			A				
	MO								B				
Cal Std 2	AL			0.250					C				
	AS								D				
	BA								E				
	BE								F				
	CD								G				
	CR								H				
	CO								I				
	CU								J				
	PB								K				
	MN								L				
	NI								M				
	AG								N				
	TL								O				
	V								P				
	ZN								Q				
	SE								R				
~~Cal Std 3~~	~~B~~			~~0.250~~					S				
	~~SN~~								T				

Sn 12510

:P-MS CALIBRA N STANDARD #2
REPARED MONTHLY OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
al Std 1	SB		20000	0.500	500	20			A				
	MO								B				
al Std 2	AL			0.500					C				
	AS								D				
	BA								E				
	BE								F				
	CD								G				
	CR								H				
	CO								I				
	CU								J				
	PB								K				
	MN								L				
	NI								M				
	AG								N				
	TL								O				
	V								P				
	ZN								Q				
	SE								R				
~~al Std 3~~	~~B~~			~~0.500~~					S				
	~~SN~~								T				

S5612503

CP-MS CALIBRATION STANDARD #3
PREPARED MONTHLY OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Cal Std 1	SB		20000	2.50	500	100			A				
	MO								B				
Cal Std 2	AL			2.50					C				
	AS								D				
	BA								E				
	BE								F				
	CD								G				
	CR								H				
	CO								I				
	CU								J				
	PB								K				
	MN								L				
	NI								M				
	AG								N				
	TL								O				
	V								P				
	ZN								Q				
	SE								R				
~~Cal Std 3~~	~~B~~ SD 8/25/03			~~2.50~~					S				
	~~SN~~								T				

CP-MS ICV / CCV ANDARD
PREPARED DAILY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Cal Std 1	AL		10000	0.500	100	50			A				
	SB								B				
	AS								C				
	BE								D				
	CD								E				
	CR								F				
	CO								G				
	CU								H				
	PB								I				
	MN								J				
	MO								K				
	NI								L				
	TL								M				
	V								N				
	ZN								O				
	SE								P				
Cal Std 2	BA			0.500					Q				
	AG								R				
~~Cal Std 3~~	~~B~~			~~0.500~~					S				
	~~SN~~								T				

SD 9/25/05

ICP-MS MRL STANDARD
(PREPARED MONTHLY OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Cal Std 1	SB		20000	0.025	500	1.0			A				
	MO					1.0			B				
Cal Std 2	AL			0.025		below			C				
	AS					1.0			D				
	BA								E				
	BE								F				
	CD								G				
	CR								H				
	CO								I				
	CU								J				
	PB								K				
	MN								L				
	NI								M				
	AG								N				
	TL								O				
	V								P				
	ZN					below			Q				
	SE					1.0			R				
Indiv. Std	AL		10000	0.450		10.0			S				
	ZN		10000	0.150		4.0			T				

P-MS ICSA SOLU N
EPARED WEEKL R AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
CSA	CL-		10000	2.00	100	200000			A				
	AL		1000			20000			B				
	CA								C				
	FE								D				
	MG								E				
	P								F				
	K								G				
	NA								H				
	S								I				
	C		2000			40000			J				
	MO		20			400			K				
	TI		20			400			L				
									M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				

P-MS ICSAB SOLUTION
REPARED WEEKLY OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
CSA	CL-		10000	2.00	100	200000			A				
	AL,CA		1000			20000			B				
	FE,MG								C				
	P,K								D				
	NA,S								E				
	C		2000			40000			F				
	MO,TI		20			400			G				
									H				
									I				
									J				
									K				
									L				
									M				
ICSAB	NI		2	1.00		20.0			N				
	MN		2						O				
	CR		2						P				
	AS		2						Q				
	CO,CU		2						R				
	AG,CD		2						S				
	ZN		2						T				

'-MS INTERNAL STANDARD SOLUTION
EPARED EVERY 6 MONTHS OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppb)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppb)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
STD	BI		10000	50.0	500	1000			A				
	HO								B				
	IN								C				
	LI +6								D				
	RH								E				
	SC					Below			F				
	TB					1000			G				
	Y					1000			H				
ingle	SC		100000	5.00		2000			I				
lement	GE		100000	25		5000			J				
									K				
									L				
									M				
									N				
									O				
									P				
									Q				
									R				
									S				
									T				

P-MS MRL AL,ZN STANDARD SOLUTION
REPARED EVERY MONTH OR AS NECESSARY)

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Single Std	AL		10000	0.010	10	10			A				
									B				
									C				
									D				
									E				
									F				
									G				
									H				

	Metal	CAS Lot #	Conc. (ppm)	Vol. (mls)	Final Vol. (mls)	Final Conc. (ppm)	Matrix	Analyst / Date	Letter ID	Nitric Acid Lot #	HCL Lot #	Expiration Date	Pipet ID
Single Std	ZN		1000	~~0.010~~ 0.100	10	10			I				
									J				
									K				
									L				
									M				
									N				
									O				
									P				

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STANDARD OPERATING PROCEDURE

for

DETERMINATION OF MERCURY IN SOLID OR SEMISOLID WASTE BY COLD VAPOR ATOMIC ABSORPTION SPECTROMETRY

SOP No.: MET-7471A/245.5

Revision: 4

December 9, 2005

Approved by: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Director Date

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One Mustard St, Suite 250
Rochester, NY 14609

Annual review of this SOP has been performed
and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

*See DOD Summary Attachment for DOD Specific Criteria

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1. SCOPE AND APPLICATION

1.1. This SOP uses Methods EPA 245.5 and SW-846 7471A for the determination of mercury in soils, sediments, bottom deposits and sludge-type materials. These are cold-vapor atomic absorption procedures. The range of the method is 0.0033 mg/kg to 0.167 mg/kg. The range may be extended above or below the normal range by increasing or decreasing the sample size. Higher concentrations may be determined by dilution of digestate.

2. METHOD SUMMARY

2.1. A known portion of a soil sample is digested in a hotblock in diluted potassium permanganate-potassium persulfate solutions and oxidized for 30 minutes at 95°C. Mercury in the water portion of the digested sample is reduced with stannous chloride to elemental mercury and measured by the conventional cold vapor atomic absorption technique.

3. DEFINITIONS

3.1. **Initial Calibration -** analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the detector to the element.

3.2. **Calibration Blank** - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to auto-zero the instrument.

3.3. **Calibration Standard** - A solution prepared from the dilution of stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.4. **Laboratory Duplicates** - Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Duplicate sample results indicate precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.5. **Laboratory Control Sample (LCS)** - An aliquot of an ERA soil standard sample to which a known quantity of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate measurements.

3.6. **Matrix Spike (MS)** - An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix

*See DOD Summary Attachment for DOD Specific Criteria

must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.7. **Preparation Blank (PB) or Method Blank (MB)** - An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if the method analyte or other interferences are present in the laboratory environment, reagents, or apparatus. This is also called Laboratory Reagent Blank (LRB) in Method 245.5.

3.8. **Independent (or Initial) Calibration Verification (ICV also known as Reference Check) -** A standard from a different source as the calibration standards used to verify the calibration curve. This is also known as the initial IPC (Instrument Performance Check) Solution in Method 245.5.

3.9. **Continuing Calibration Verification (CCV) -** A standard from the same source as the calibration standards used to verify the curve with each daily run and throughout the run at specified intervals. This is also known as the IPC (Instrument Performance Check) Solution in Method 245.5.

3.10. **Method Reporting Limit Standard (MRL) or CRDL** – Standard prepared with a known concentration of elements to check accuracy at the low end of the curve.

3.11. **Linear Dynamic Range (LDR)** - The concentration range over which the instrument response to an analyte is linear.

3.12. **Method Detection Limit (MDL)** - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

3.13. **Standard Addition** - The addition of a known amount of analyte to the sample in order to determine the relative response of the detector to an analyte within the sample matrix. The relative response is then used to assess either an operative matrix effect or the sample analyte concentration.

3.14. **Batch** – Unit of samples prepared together on the same day, not to exceed 20 samples.

3.15. **Relative Percent Difference (RPD)** – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.16. **Method Reporting Limit (MRL):** The minimum amount of a target analyte that can be measured and reported quantitatively. The MRL is equivalent to Practical Quantitation Level (PQL) and Estimated Quantitation Level (EQL). Typically, the MRL is calculated

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*See DOD Summary Attachment for DOD Specific Criteria

as five times the MDL (although this is a rule of thumb and not intended to be a strict policy of establishing the MRL for a compound).

3.17. **Dissolved Analyte** - The concentration of analyte in an aqueous sample that will pass through a 0.45 µm membrane filter assembly prior to sample acidification.

4. INTERFERENCES

4.1. Interferences have been reported for waters containing sulfide, chloride, copper and tellurium. Organic compounds, which have broad band UV absorbance (around 253.7 nm), are confirmed interferences. The concentration levels for interferants are difficult to define.

4.2. Low level mercury sample preparation, digestion, and analysis may be subject to environmental contamination if preformed in areas with high ambient backgrounds where mercury was previously employed as an analytical reagent in analyses such as total kjeldahl nitrogen (TKN) or chemical oxygen demand (COD).

5. SAFETY

All appropriate safety precautions for handling reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

Mercury compounds are highly toxic if swallowed, inhaled or absorbed through the skin.

Sulfuric and Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

Acidification should only be done in a fume hood due to the potential release of toxic gases such as cyanide or sulfide.

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases should be clearly labeled.

Refer to the Safety Manual for further discussion of general safety procedures and information.

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6. SAMPLE CONTAINERS, COLLECTION, PRESERVATIONS, AND STORAGE

6.1. Because of the extreme sensitivity of the analytical procedure and the presence of mercury in a laboratory environment, care must be taken to avoid extraneous contamination. Sampling devices, sample containers and plastic items should be determined to be free of mercury; the sample should not be exposed to any condition in the laboratory that may result in contamination from airborne mercury vapor.

6.2. Samples are maintained at 0-6°C from receipt until analysis.

6.3. The sample should be analyzed within 28 days of collection. Once digested, samples are analyzed as soon as possible.

6.4. Sample Receiving procedures are discussed in SOP, SMO-GEN.

7. APPARATUS AND EQUIPMENT

7.1. Atomic Absorption Spectrophotometer equipped with a vapor generator, quartz absorption cell and mercury hollow cathode lamp. The details may be found in Appendix A of the Quality Assurance Manual – including model numbers, serial numbers, and computer hardware and software.

7.2. 100 mL Hot Block cups

7.3. Hot block capable of maintaining a sample temperature of 90-95ºC. See Appendix A of the Quality Assurance Manual for more details.

7.4. Pipettes and graduated cylinders.

*See DOD Summary Attachment for DOD Specific Criteria

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8. PREVENTIVE MAINTENANCE

Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

Typical preventive maintenance measures include, but are not limited to, the following items:

- Check gases and tubing, daily
- Check optic tubes and filter membrane for moisture

Troubleshooting – see Instrument manual or maintenance log for help with solving specific analytical or instrument problems.

9. STANDARDS AND REAGENTS

9.1. Each lot of acid used is to be analyzed to demonstrate that it is free of interference before use. See ADM-CTMN.

9.2. **Standards Preparation General Information and Disclaimers**

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mLs or may be made by adding 4 mL of 50 mg/L to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to targets and detection levels needed. The curves will always be at least 5 points. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

*See DOD Summary Attachment for DOD Specific Criteria

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All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

9.3. Mercury stock solution (1,000 mg/L) - Purchased. Store at room temperature. Dispose per manufacturer's expiration date.

9.4. Intermediate Stock Solution (10 mg/L) - Prepare a 1/100 dilution of the 1000mg/L Stock Solution in a volumetric flask and dilute with DI water. Acidify with 0.5ml of concentrated HNO_3. Store at room temperature for up to 1 week.

9.5. Working Solution (100 µg/L) - Prepare a 1/100 dilution of the 10mg/L Intermediate Stock Solution in a volumetric flask and dilute with DI water. Acidify with 0.5 of concentrated HNO_3. Prepare fresh each day the analysis is performed.

9.6. Calibration Standards

Concentration (µg/L)	100 µg/L working solution added (mL)	Final Volume with reagent water (mL)
0	0	10
0.2	0.2	10
0.5	0.5	10
1.0	1.0	10
2.0	2.0	10
5.0	5.0	10
10.0	10.0	10

Prepare in a 100 mL hotblock cup. Add 5 mL aqua regia. Mix thoroughly and digest as a sample. Prepare fresh each day the analysis is performed.

9.7. ASTM Type II water.

9.8. Concentrated Nitric Acid - Metals Grade. Store at room temperature. Expires upon manufacturer's indications or 3 years from receipt if no other indication is given.

9.9. Concentrated Sulfuric Acid - Metals Grade. Store at room temperature. Expires upon manufacturer's indications or 3 years from receipt if no other indication is given.

9.10. Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

9.11. 5% w/v Potassium Permanganate Solution - Dissolve 50 g of $KMnO_4$ in 1 L of reagent water. Store at room temperature for up to 6 months.

*See DOD Summary Attachment for DOD Specific Criteria

9.12. 12% w/v Sodium chloride-hydroxylamine chloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride (NH_2OH*HCl) in 1 L reagent water. (Hydroxylamine sulfate $(NH_2OH)_2$ H_2SO_4 may be used in place of hydroxylamine hydrochloride. Store at room temperature for up to 6 months.

9.13. 1.1% Stannous chloride + 3% HCl solution - Add 11.0 g of $SnCl_2 \bullet 2H_2O$ to 1L of 3% HCl. Prepare fresh each use.

9.14. The calibration blanks (ICB and CCB) must contain all reagents in the same concentrations and in the same volume as used in preparing the calibration solutions.

9.15. The preparation blank (PB) is prepared in the same manner as the calibration blank and is carried through the entire preparation scheme with each batch of samples to be analyzed.

9.16. LCS – purchased commercially from ERA with a certified value. The LCS must be carried through the entire sample preparation scheme with each batch of samples to be analyzed.

9.17. ICV/CCV – (3.0 µg/L) - Prepare an intermediate stock solution and working solution of 10 mg/L and 100 µg/L as described above using a different stock source. Transfer 3.0 ml of 100 µg/L solution (prepared daily) to a 100 ml hotblock cup. Add 7 mL reagent water and 5 mL aqua regia. Digest as a sample.

9.18. MS –Prepared by fortifying a 25 ml sample with 0.6 g sample with 1.0 mL of 100 µg/L CCV standard in 100 mL hotblock cup. The MS must be carried through the entire analytical procedure.

9.19. CRDL Standard – This is the same as the 0.2 µg/L standard. It need not be prepared separately.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

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*See DOD Summary Attachment for DOD Specific Criteria

11. PROCEDURES

11.1. Be sure the analyst has a current Demonstration of Capability and the system has a current MDL.

11.2. Prepare calibration standards and QC samples as per Section 9.

11.3. For client samples, weigh 0.6g portion of a representative sample (approx. 0.2g portions from three areas of the sample) and place in the bottom of a 100 mL hotblock cup. Add 5 ml of reagent water and 5 ml of aqua regia.

11.4. Heat in the hotblock for 2 minutes at 95°C. Cool. Add 45 ml reagent water to calibration standards and CCVs. Add 50 ml of reagent water to samples. To all cups add 15 ml of potassium permanganate solution. Loosely cap all containers. Mix thoroughly and place in the hotblock for 30 minutes at 95°C.

Note: Samples with high organic content may required additional permanganate. Shake and add additional permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Ensure that equal amounts of permanganate are added to all samples, standards and blanks.

11.5. Cool and add 6.0 ml of sodium chloride/hydroxylamine hydrochloride solution. Add 50 ml of reagent water and the samples are now ready to be analyzed.

11.6. The final step of adding the stannous chloride solution to the sample volume to release the elemental mercury is done automatically by the vapor generator.

11.7. Analyze the standards and samples using the Perkin Elmer Flow Injection Mercury System. See Attachment 2 for Operation Instructions and Instrument Specifications.

11.8. Initial Calibration

11.8.1. Analyze the initial calibration standards. See Operations Manual for details on operating the instrument. A blank and 5 standards are required by both methods.

11.8.2. Construct a calibration curve using linear regression. This method of quantitation uses the equation of a line ($y=mx+b$). The correlation coefficient for each calibration must be ≥0.995.

11.8.3. Analyze the ICV. The ICV must be within 5% of the true value for Method 245.5 and within 10% of the true value for method 7471A. If the ICV is out of control, stop the run, investigate the source of the problem, and recalibrate. Obtain a compliant ICV before sample analysis begins.

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*See DOD Summary Attachment for DOD Specific Criteria

11.8.4. ICB – The initial calibration blank must be less than the MRL*. Do not proceed with sample analysis until an acceptable ICB is achieved. Recalibrate if necessary.

11.9. Analyze the samples and QC samples.

11.10. Sample concentrations exceeding the Linear Range require sample dilution. Dilutions should be performed so that the instrument concentration will fall in the mid-range of the calibration curve. See ADM-DIL

12. QA/QC REQUIREMENTS

12.1. Method Detection Limit (MDL) - A mercury MDL must be determined annually using 7 replicates of a fortified blank solution at a concentration of 2-3 times the estimated detection limit. Practical Quantitation Limits (PQLs) are calculated from the MDL by multiplying the MDL by a factor of at least 3. The PQLs are generally used as CAS Reporting Limits. To determine the MDL, refer to CAS SOP *ADM-MDL*.

12.2. PB - Analyze at least one PB with each batch of 20 or fewer samples of the same matrix. PB values must not exceed the MRL*. If the PB is out of control, fresh aliquots of the samples must be reprepared and analyzed again for affected analytes after the source of the contamination has been corrected and acceptable PB values have been obtained. Samples greater than 10 times the blank contamination may be reported.

12.3. LCS – Analyze at least one LCS with each batch of 20 or fewer samples of the same matrix. Assess laboratory performance against the values on the vendor's certificate of analysis for the solid standard. The control limit range is specific for each lot. If the recovery of mercury falls outside the required control limits, the analysis is judged to be out of control, and the source of the problem should be identified and resolved before continuing analysis. Redigestion and analysis is required until acceptable LCS recovery is performed.

12.4. ICV is discussed in the ICAL section in the Procedure (section 11).

12.5. CCV – Analyze one after every tenth sample and at the end of the sample run. Continuing Calibration Verification standards must confirm the calibration within ±10% throughout the analyses. Refer to Appendix C of the Quality Assurance Manual. If the recovery of mercury falls outside the required control limits, the analysis is judged to be out of control, and the source of the problem should be identified and resolved before continuing analysis. Reanalysis of any sample(s) associated with the outlying CCV standards is required. All samples must be bracketed with acceptable ICV and CCV standards.

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*See DOD Summary Attachment for DOD Specific Criteria

12.6. Calibration Blank – analyze one calibration blank after the ICV, after every 10 samples and at the end of the run. The calibration blank must be less than the MRL*. If it is not, correct the problem and reanalyze all samples bound by the out of control blank.

12.7. MS – Analyze one MS sample for every 10 client samples. Refer to Appendix C of the Quality Assurance Manual for recovery limits. If the recovery of mercury falls outside the required control limits, and the LCS is in control, assume matrix interference and flag the associated data.

12.8. DUP – Analyze one client in duplicate for every 20 client samples. Refer to Appendix C of the Quality Assurance Manual for RPD limits*. If the RPD value exceeds limits, reanalyze the pair if deemed appropriate. The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately

12.9. CRDL Standard – must be analyzed before client samples and the end of the analytical run. The CRDL standard must be within 50-150% of the true value unless otherwise stated in project specific data quality objectives. If it is not, correct the problem and obtain an acceptable value or qualify the associated data.

12.10. Dilution Test and MSA – See DOD Summary*

13. DATA REDUCTION AND REPORTING

13.1. Calculations:

13.1.1. From the prepared calibration curve compute sample values by comparing response with the standard curve. Calculate the mercury concentration in the sample by the formula:

mg/Kg = Vol. (ml)/sample Wt(g) **x** 1mg/1000ug **x** 1L/1000ml **x** 1000g/1Kg **x** C **x** dilution

C = concentration of Hg in digestate, in ug/L

13.2. Reporting

13.2.1. Record all sample masses and dilution factors in the Mercury Preparation and Run Log.

13.2.2. Record all concentrations determined at the instrument and calculate the final results in mg/Kg. Print out a LIMS run report and attach to the instrument print out.

13.2.3. Data Review shall be in accordance to SOP ADM-DREV.

13.2.4. DOD – flag all values between MDL and MRL.

*See DOD Summary Attachment for DOD Specific Criteria

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*See DOD Summary Attachment for DOD Specific Criteria

14. METHOD PERFORMANCE

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

Demonstration of Capability is performed upon instrument set-up, whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office

Accuracy and Precision Data is available in the referenced methods.

15. WASTE MANAGEMENT AND POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when disposed of properly.

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

Samples with mercury concentrations exceeding 0.2 ppm are disposed of as hazardous waste, see CAS SOP *SMO-SPLDIS*.

16. CORRECTIVE ACTION FOR OUT of CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT of CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

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*See DOD Summary Attachment for DOD Specific Criteria

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18. REFERENCES

18.1. *Test Methods For Evaluating Solid Waste, Physical/Chemical Methods*. USEPA SW-846, 3rd Edition, September 1994.

18.2. *Methods For the Determination of Metals in Environmental Samples Supplement I.* USEPA/600/R-94/111, May 1994

18.3. EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0.

18.4. Analytical Services Protocol (ASP), New York State Department of Environmental Conservation, December 1995.

19. TRAINING OUTLINE

19.1. Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2. Observe Sample Preparation and Analysis. Follow Training Plan Form (May be found on the Rochester CASLAB Intranet at P:\INTRANET\QAQC\TRAINING\QAforms.HTM.)

19.3. Participate in the methodology, documentation, and data reduction with guidance.

19.4. Perform Initial Demonstration of Capability by performing the analysis independently and analyzing a known standard four times. If recovery is within acceptable limits, complete Training Plan Form and IDC certificate and file with QA. Continuing proficiency shall be demonstrated annually using an outside PE source, an internal unknown, or a new 4 replicate study.

20. METHOD MODIFICATIONS

None.

21. INSTRUMENT-SPECIFIC ADDENDUM

21.1. Perkin Elmer Flow Injection Mercury System (FIMS)

21.2. Refer to Manufacturer Operation Manuals located near FIMS instrument.

*See DOD Summary Attachment for DOD Specific Criteria

22. ATTACHMENTS

Analytical Preparation Log
Operation Instructions and Instrument Specifications
DOD Summary

23. CHANGES FROM PREVIOUS REVISION

- Added sections 14, 16, 17, and 20 for NELAC compliance.
- Incorporated the previous addendum into the SOP.
- Eliminated statements referring to BOD bottles or water bath – all samples are now processed in a hotblock. Volumes of sample and reagents used for digestion all modified throughout.
- Added DOD Summary for requirements for all DOD work.
- 1 – Reworded for consistency with other SOPs
- 3 – Added Definitions for ICAL, ICV, CCV, MRL/CRDL standard, RPD, MRL, and Dissolved Analyte. Equated 245.5 terminology with our terminology
- 5, 15, 19 – Modified for consistency with other SOPs
- 7 – Referenced Appendix A of the QAM for instrument details
- 8-Expanded upon the requirements of the maintenance log. Added troubleshooting.
- 9-Added reference to ADM-CTMN. Added Standards Prep Info and Disclaimers.
- 9-Put the calibration standards into a table
- 9-Added storage and expiration where needed.
- 11-Added need to check for a current MDL and DOC.
- 11-Reworded and reorganized entire section for clarity and consistency with other SOPs.
- 11-Added ICAL section - moved ICAL requirements from Section 12 to Section 11.
- 12-Added Calibration Blank, DUP, CRDL, Dilution Test and MSA
- 12-Changed PB frequency from 1/10 to 1/20 since 1/10 is not required by the methods.

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*See DOD Summary Attachment for DOD Specific Criteria

Columbia Analytical Services

Analysis For: Hg **EPA Method: 7470 / 7471 / 245.1 / 245.5** **Analyst:** _____

Bath Temp: _____ **Report: Routine / ASP / Pkg.5** Date Prepped: _____

Time In: _____ **File Name:** _____ Date Analyzed: _____

Time Out: _____

	Client / Submission #	Sample Number	Initial Wgt/Volume (g/ml)	Final Volume (ml)			Client / Submission #	Sample Number	Initial Wgt/Volume (g/ml)	Final Volume (ml)
1						31				
2						32				
3						33				
4						34				
5						35				
6						36				
7						37				
8						38				
9						39				
10						40				
11						41				
12						42				
13						43				
14						44				
15						45				
16						46				
						47				
						48				
19						49				
20						50				
21						51				
22								Std 0.2*	0.20ml of 0.1ppm	100
23								Std 0.5*	0.50ml of 0.1ppm	100
24								Std 1.0*	1.00ml of 0.1ppm	100
25								Std 2.0*	2.00ml of 0.1ppm	100
26								Std 5.0*	5.00ml of 0.1ppm	100
27								Std 10.0*	10.0ml of 0.1ppm	100
28								ICV/CCV**	3.00ml of 0.1ppm	100
29								LCSW/MS**	1.00ml of 0.1ppm	100
30								CRDL*	0.20ml of 0.1ppm	100

Lot # of Reagents Used:

HNO3: _____ H2SO4: _____ HCL: _____ K2S2O8: _____ KMnO4: _____

SnCl2: _____ NaCl: _____ NH2OH-HCL: _____ LCSS CAS Lot#: _____

LCSS ERA Lot#: _____

*Source Standard: (Vendor/Lot #)_____ **ICV Standard: (Vendor/Lot #)_____

*(10ppm stock Lot#: _____) **(10ppm stock Lot#: _____)

*(0.1ppm working std Lot#: _____) **(0.1ppm working std Lot#: _____)

Comments/Problems: _____

Attachment 2

Operation Instructions and Instrument Specifications

The Flow Injection Mercury (FIM) Instrument is preset to analyze mercury with the following specifications. Adjustments to these specifications are not recommended:

Spectrophotometer

Technique	AA
Integration Time (s)	20
Data Processing	Peak Height, Smoothing 0.5 sec or 19 points
Lamp	HCL or EDL
Slit (nm)	0.7
Wavelength (nm)	253.7

Reagents

Carrier Solution	3% HCl
Reducing Agent	1.1% $SnCl_2$ in 3%HCl
Sample Solution	Hg^{2+} in acidified solution
Carrier Gas	Argon, Inlet pressure 320-400 kPa. Flows: 40-250 ml/min,

Operation Instructions:

1. Turn on the instrument and allow it to warm up for approximately 30-45 minutes.
2. Prepare the $SNCl_2$ and 3%HCl reagents.
3. Tighten the tubing and pump and insert the lines into the reagents.
4. Insert the gas/liquid separator membrane after the mixing block.
5. Pour up samples and standards into centrifuge tubes on the autosampler. The standards for the initial calibration curve shall be set from low to high to avoid carry-over into low-level standards.
6. Engage the pump and allow all reagents to circulate throughout the tubing. Air bubbles should be forming in the waste line due to the gas/liquid separator membrane.
7. Start the analytical run.
8. Monitor the peaks occasionally to ensure adequate baseline and bell-shaped peaks are formed.
9. Monitor run QC for acceptable performance and document data review on data review checklist.

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*See DOD Summary Attachment for DOD Specific Criteria

*DOD SUMMARY

For work for the Department of Defense – the DOD Quality Systems Manual must be followed. The DOD Manual is based on the NELAC Standards with some additional requirements. The following are the requirements which are different or additional to routine analysis and must be followed for DOD work:

- The Prep Blank result must not be above ½ the reporting limit.
- The Calibration Blanks must be < 2 times MDL.
- Dilution Test – must be performed with each batch. Five-fold dilution must agree within 10% of the original determination. Only applicable for samples with concentrations >25 x MDL. If dilution test fails, perform matrix spike.
- Use the Method of Standard Addition (MSA) when matrix interference is suspected.
- Apply J flag to all hits between MDL and MRL.
- Reporting Limits – "the lowest standard of the calibration curve establishes the [reporting limit], but it must be greater than or equal to 3 times the [MDL]. DoD QSM, V3, Appendix B-Glossary
- Volumetric glassware must be checked annually and be within 3% of known value.
- The RPD between the sample and the duplicate must be less than 20%.

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*See DOD Summary Attachment for DOD Specific Criteria

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STANDARD OPERATING PROCEDURE

for

PCBs by GC/MS

SOP No.: SOC-680

Revision: 2

April 6, 2004

Approved by: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____
Laboratory Manager Date

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1 Mustard Street, Suite 250
Rochester, NY 14609

Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER:_____
Initials:_____Date:_____

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1. SCOPE AND APPLICATION

1.1 Method 680 provides a procedure for mass spectrometric determination of polychlorinated biphenyls (PCBs) in water. This method is applicable to samples containing PCBs as single congeners or as complex mixtures, such as commercial Aroclors. PCBs are identified and measured as isomer groups (i.e. by level of chlorination). The existence of 209 possible PCB congeners makes impractical the listing of the CAS registry number (CASRN) for each potential method analyte. Because PCBs are identified and measured as isomer groups, the non-specific CASRN for each level of chlorination is used to describe the method analyte. Reporting Limits are found in Appendix C of the Quality Assurance Manual.

Analyte	Formula	CASRN
Monochlorobiphenyls	$C_{12}H_9Cl$	27323-18-8
Dichlorobiphenyls	$C_{12}H_8Cl_2$	25512-42-9
Trichlorobiphenyls	$C_{12}H_7Cl_3$	25323-68-6
Tetrachlorobiphenyls	$C_{12}H_6Cl_4$	26914-33-0
Pentachlorobiphenyls	$C_{12}H_5Cl_5$	25429-29-2
Hexachlorobiphenyls	$C_{12}H_4Cl_6$	26601-64-9
Heptachlorobiphenyls	$C_{12}H_3Cl_7$	28655-71-2
Octachlorobiphenyls	$C_{12}H_2Cl_8$	31472-83-0
Nonachlorobiphenyls	$C_{12}HCl_9$	53742-07-7
Decachlorobiphenyl	$C_{12}Cl_{10}$	2051-24-3

2 METHOD SUMMARY

2.1 A one liter water sample is placed in a separatory funnel and extracted with methylene chloride. The extract is dried and concentrated to a final volume of 0.5 ml. Sample extract components are separated with capillary column gas chromatography (GC) and identified and measured with low resolution, electron ionization mass spectrometry (MS). An interfaced data system is used to control data acquisition and to store, retrieve, and manipulate mass spectral data.

2.2 Two surrogates are added to each sample before sample preparation; these compounds are 4,4'-DDT and gamma-BHC. Two internal standards, chrysene-d_{12} and phenanthrene-d_{10} are added to each sample extract before GC/MS analysis and are used to calibrate MS response.

2.3 Surrogate compound, 4,4'-DDT, shall be used to determine retention time for starting ion set #4.

2.4 PCBs are identified and measured as isomer groups (by level of chlorination). A concentration is measured for each PCB isomer group; total PCB concentration in each sample extract is obtained by summing isomer group concentrations.

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2.5 Nine selected PCB congeners are used as calibration standards, and one internal standard, chrysene-d_{12} is used to calibrate MS response to PCBs, unless sample conditions require the use of the second standard, phenanthrene-d_{10}.

3 DEFINITIONS

3.1 **Concentration Calibration Solution** - A solution of method analytes used to calibrate the mass spectrometer response.

3.2 **Congener Number** - Throughout this method, individual PCBs are described with the number assigned by Ballschmiter and Zell. These numbers are also used to describe PCB congeners in catalogs produced by manufacturers.

3.3 **Analysis Window** – Samples are analyzed in a set referred to as a "window". The window begins with the injection of the tune verification standard. Standards, required QC samples, and samples may be run for 12 hours in this window. A new window must be opened to continue analysis.

3.4 **Retention Time Window:** the time period established within which a target analyte is qualitatively determined to be present in the sample

3.5 **Matrix -** the predominant material, component, or substrate (e.g., surface water, drinking water, etc.) of which the sample to be analyzed is composed.

3.6 **Internal Standard** - A pure compound added to a sample extract in known amounts and used to calibrate concentration measurements of other compounds that are sample components. The internal standard must be a compound that is not a sample component.

3.7 **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.8 **Batch** – a group of no more than 20 samples extracted together on the same day.

3.9 **Sample -** a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

3.10 **Method Detection Limit (MDL):** a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero

3.11 **QA/QC Samples** -

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3.11.1 **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.11.2 **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.11.3 **Laboratory Control Sample (LCS)** - An aliquot of analyte-free water or other blank matrix to which known quantities of analytes of interest are added in the laboratory. The LCS is analyzed the same as a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements.

3.12 **Standard Curve** - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.13 **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration. This standard is also known as the Laboratory Performance Check Solution.

3.14 **Independent Calibration Verification Standard (ICV)** - A mid-level standard injected into the instrument after the calibration curve – prepared by a different analyst as the calibration standards - is used to verify the validity of the initial calibration.

4 INTERFERENCES

4.1 Interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing equipment. Laboratory reagent blanks are analyzed routinely to demonstrate that these materials are free of interferences under the analytical conditions used for samples.

4.2 To minimize interferences, glassware should be meticulously cleaned. As soon as possible after use, rinse glassware with the last solvent used. Then wash with detergent in hot water and rinse with tap water followed by distilled water and a final rinse with acetone and methylene chloride.

4.3 Interference can be caused by the presence of much greater quantities of other sample components that overload the capillary column. Capillary column GC retention times

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and the compound-specific characteristics of mass spectra eliminate many interferences that formerly were of concern with PCB determinations with electron capture detection. The approach and identification criteria used in this method for PCBs eliminate interference by most chlorinated compounds other than PCBs. With the isomer group approach, coeluting PCBs that contain the same number of chlorines are identified and measured together. Therefore, coeluting PCBs are a problem only if they contain a different number of chlorine atoms.

4.4 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical and sample should be treated as a potential health hazard. Exposure should be reduced to the lowest possible level. The laboratory maintains a compilation of Material Safety Data Sheets in binders the conference room. Always wear safety glasses or a shield for eye protection, and protective clothing, and observe proper mixing when working with these reagents.

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases should be clearly labeled.

Refer to the Safety Manual for further discussion of general safety procedures and information.

6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Sample collection containers are purchased certified clean glass and have screw-top covers with teflon liners.

6.2 Water samples must be iced or refrigerated at 0-6 °C from time of collection until extraction. If samples will not be extracted within 72 hours after collection, use either sodium hydroxide or sulfuric acid to adjust sample pH to within a range of 5-9. Record the volume of acid or base used.

6.3 Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction

6.4 Further sample handling, storage, and custody procedures are found in SMO-GEN.

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7 APPARATUS AND MATERIALS

7.1 Gas Chromatograph/Mass Spectrometer System

7.1.1 Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

7.1.2 Column: RTX-5 SILMS with Integra guard - 30 m x 0.25 mm ID x 0.50 µm film thickness silicone-coated fused-silica capillary column.

7.1.3 SIM Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. To ensure sufficient precision of mass spectral data, the required MS scan rate must allow acquisition of at least five full-range mass spectra or five data points for each monitored ion while a sample component elutes from the GC. The MS must produce a mass spectrum meeting all criteria for ≤50ng of decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet.

7.1.4 Operating Conditions of GC/MS

Initial Temperature: 50°C
Initial Time: 1.0 min.
Injection Volume: 2 ul

Level	Rate (C/min)	Final Temperature (C)	Final Time (min.)
1	9.0	120	0.0
2	15.0	320	3.78

7.1.5 GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.6 Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

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7.1.7 SIM processing - For SIM data acquisition, the data system must be equipped with software capable of acquiring data for multiple groups of ions, and the system must allow automated and rapid changes of the set of ions being monitored. The SIM program must be capable of acquiring data for five groups of ≤20 ions each. The times spent monitoring ions during sample analysis must be the same as the times used when calibration solutions were analyzed.

7.2 Glassware

7.2.1 Volumetric Flasks.
7.2.2 Microsyringes.
7.2.3 Two-liter Separatory Funnels.
7.2.4 Glass Funnels with glass wool plugs.
7.2.5 Concentrator Tubes (10 ml graduated Kuderna-Danish design with ground glass stoppers).
7.2.6 Evaporative Flask (500 ml Kuderna-Danish design).
7.2.7 Snyder Columns.
7.2.8 Boiling Chips.
7.2.9 Water Bath.
7.2.10 10-15 ml Amber Glass Vials.

8 PREVENTIVE MAINTENANCE

8.1 Glass Insert/Sleeve –Indicators are breakdown of DDT and loss of sensitivity of other compounds. The insert should always be replaced before a new calibration and the o-ring should be replaced with the insert.

8.2 Septum - Replaced approximately every 150 injections. A retention time shift or poor baseline chromatography is a common symptom.

8.3 Autosampler syringe - Cleaned daily and after samples with a particularly bad matrix. The syringe should be rinsed with CH_2Cl_2; the plunger is rinsed, wiped with a Kimwipe, and rinsed again. The syringe should be replaced with a new one if loose action is noted or inconsistent action is evident (uniform fluctuation in response, area counts) or obvious wear.

8.4 Injection Port –applicable to split-splitless injectors only- The port and the port/column interface should be cleaned as needed (when replacing the insert is ineffective and in conjunction with column pruning). Port should be reamed with a wire brush and swabbed with CH_2Cl_2. Disc and washer can be sonicated in methylene chloride and then silanized, but should be replaced if wear is evident. The capillary inlet should also be cleaned in CH_2Cl_2 and the septum nut is sonicated in CH_2Cl_2 also.

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8.5 Column Pruning - Cut back one revolution as needed. Indicators are same as insert replacement and is another remedy if insert replacement does not yield the desired results. After initial cut, a graphite ferrule should first be installed and then another short length of column removed (cut should be carefully examined for smoothness - no jagged edges). The column end should extend 6mm past the ferrule when installed (use white out or septum marking method for reference).

8.6 Column Replacement - As needed, usually every 2-3 months for moderate sample volumes. Symptoms are ineffectiveness of insert replacement, injector cleaning, or column pruning to rectify a problem. All ferrules replaced with column change.

8.7 Syringe Rinse Solvent - Supply is replenished as needed. Old solvent is discarded and vial rinsed before refilling. Rinse waste and sample waste vials are replaced when residue appears.

8.8 Carrier Gas - Tank is changed before pressure reaches approximately 200psi to avoid impurities that may be delivered by a nearly empty tank. Leak check with snoop after change.

8.9 System baking - When the system is to be idle oven temperature should be set to 100°C to avoid water vapor condensation in column. Baking of injector, column, interface may be done for short periods of time to eliminate contaminants due to sample matrix residuals. Temperature should not exceed recommended column temperature limits.

8.10 Source Cleaning - Performed on an as-needed basis. Source is cleaned/replaced when persistent difficulty in meeting and maintaining acceptable DFTPP tune checks is encountered. This is indicated by : 1.) reduced high-mass sensitivity (DFTPP m/z 365<1% of 198 or PFTBA m/z 502<0.5%) 2.) High EM setting resulting in excessive signal and inconsistent response in both tune file and instrument methods. A dirty source can sometimes be diagnosed by jagged lens-ramp plots in addition to tune difficulties. The procedure for removing and cleaning the ion source is followed as in the instrument Manual.

8.11 Electron Multiplier - Preventive Maintenance or replacement as needed. Method of cleaning and conditioning a new EM can be found in accompanying literature from the manufacturer (Channeltron/Galileo). Cleaning is usually necessary when source replacement is not having a significant effect on instrument sensitivity. Replacement of the EM is necessary when cleaning does not yield the desired results, or when there is a catastrophic event (outlined in literature).

8.12 Leak Detection - A manual tune should be monitored at set intervals for peak characteristics of air leakage (abundance of ions corresponding to N_2, H_2O, O_2), or an Ion gauge reading taken regularly can show changes in vacuum. Proper Ion gauge readings should be in the 10^{-5} - 10^{-6} torr range.

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8.13 Internal Standard Syringe - Rinsed with methylene chloride before and after use. The Teflon tip is checked for wear regularly and the tip, plunger or entire syringe is replaced when necessary.

8.14 Splitless Liner Preparation - Remove dirty glass wool plug. Solvent rinse splitless liner with methylene chloride. Air dry and pack with glass wool to an approximate 5mm thickness. The glass wool packing should not be very dense. Immerse prepared liners in Sylon solution for deactivation. Rinse with methanol and air dry.

8.15 Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

9 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1 Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences. Purity is checked as per ADM-CTMN. Store at room temperature for up to 3 years from receipt.

9.2 Sodium Sulfate - Granular, anhydrous. Purify and store as per EXT-SUL.

9.3 **Standards Preparation General Information and Disclaimers**

9.3.1 All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 ppb standard may be made by adding 1 uL of 200 ppb to 10 mLs or may be made by adding 4 uL of 50 ppb to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

9.3.2 The initial calibration curves given are typical, but also subject to variation due to targets and detection levels needed. The curves will always be at least 5 points. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

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9.3.3 Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

9.3.4 All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

9.3.5 Stock standard solutions-are purchased as certified solutions and expire per manufacturer recommendations. Protect all standards from light. Samples, sample extracts and standards must be stored separately. All standards are stored at 0-6 °C.

9.4 Stock calibration standard – Ultra 680 PCB Calibration Mix or equivalent. The concentrations of each congener in the purchased stock are given in Table 2. Expires upon manufacturer's indications or one year from receipt.

9.5 Stock Retention Time Standard – Ultra 680 PCB Retention Time Mix or equivalent. The concentrations of each congener in the purchased standard are given in Table 2. Expires upon manufacturer's indications or one year from receipt.

Knowledge of the retention times of certain congeners is necessary to determine when to acquire data with each ion set. Two concentration calibration congeners also serve as retention time congeners; the first eluting Cl_1-PCB indicates the time when data acquisition must have been initiated for ion set #1, and the Cl_{10}-PCB indicates when all PCBs have eluted. The three additional PCB congeners in this Retention Time Standard (Cl_4#77, CL_5#104, and CL_9#208) are used to establish times to initiate data acquisition with other ion sets.

9.6 Stock surrogate standards – Purchased commercially. Expires upon manufacturer's indications or one year from receipt.

9.6.1 4,4'-DDT (5000 mg/L)
9.6.2 Gamma-BHC (1000 mg/L)

9.7 Intermediate surrogate standards (100 mg/L) – Expires in one year. This solution is used to spike the calibration standards and to make the Working Surrogate Standard.

9.7.1 Dilute 1.0 mL stock 4,4'-DDT (5000 mg/L) to 50 mL with methylene chloride.

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9.7.2 Dilute 1.0 mL stock gamma-BHC (1000 mg/L) to 10 mL with methylene chloride.

9.8 Working Surrogate Standard (1 ppm) . Dilute 0.5 ml of each 100 ppm stock standard to 50 ml in a volumetric flask with methanol. Use 0.5 ml to spike performance check solutions, blanks, spikes, and samples prior to extraction. Take into account all dilutions of sample extracts. If surrogates are diluted more than 10 times, report as "NA" (diluted below calibration).

9.9 Intermediate Calibration Standard (5-25 ppm) - Dilute 1.0 ml of each of the following to 10 mL with methylene chloride: Stock Calibration Standard, Stock Retention Time standard, 100 ppm 4,4'-DDT, and 100 ppm gamma-BHC (intermediate surrogate standards). This standard is the CAL 5 and its composition and concentration is given in Table 2.

9.10 Working Calibration Standards - The nine individual PCB congeners listed in Table 1 are used as concentration calibration compounds for PCB determinations. One isomer at each level of chlorination is used as the concentration calibration standard for all other isomers at that level of chlorination, except that decachlorobiphenyl (Cl_{10}) is used for both Cl_9 and Cl_{10} isomer groups. A minimum of five calibration standards should be prepared. Each 1 mL aliquot of calibration standard should be spiked with 10 uL of the internal standard solution prior to analysis. The working calibration standards should be prepared weekly. Prepare the working calibration standards as follows:

Calibration Standard	Volume of Intermed. Cal Std.	Final Volume (in Methylene Chloride)	Final Conc (ppm)*
0	200ulof Cal Std 1	1 ml	0.02-0.10
0.5	0.010ml	1 ml	0.05-0.25
1.0	0.020ml	1 ml	0.1-0.5
1.5	0.050ml	1 ml	0.25-1.25
2.0	0.100ml	1 ml	0.5-2.5
3.0	0.200	1 ml	1.0-5.0

*See Table 2 for actual concentrations of each congener, internal standard, and surrogate compound in each calibration standard.

9.11 Stock Decafluorotriphenylphosphine Tune Standard (2500 mg/L DFTPP) – purchased commercially. Expires upon manufacturer's indications or one year from receipt.

9.12 Working Tune Standard - DFTPP standard (25 ppm) - Dilute 50 uL stock DFTPP solution to 5 mL in a volumetric flask with methylene chloride. Expires 6 months.

9.13 Stock Internal Standards – purchased commercially. Expires as per manufacturer's indications or one year from receipt.

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9.13.1 Phenanthrene-d_{10} (1000 mg/L)
9.13.2 Chrysene-d_{12} (2000 mg/L)

9.14 Working Internal Standard Solution (75 ppm) - Dilute 0.75 ml of stock phenanthrene-d_{10} and 0.375 ml of stock chrysene-d_{12} to 10 ml with methylene chloride. Each 1 mL sample extract undergoing analysis should be spiked with 10 uL of the internal standard solution, resulting in a concentration of 0.75 ppm of each internal standard.

9.15 ICV Working Standard (0.1-0.5 ppm) - Dilute 0.1 ml of Intermediate Calibration Standard to 5.0 ml with methylene chloride. It has the same concentrations as CAL 1 in Table 2. This standard is the ICV. Expires in 6 months.

9.16 LCS/MS Midstock Solution (1-5 ppm) – Dilute 1 mL of Ultra Concentraed Calibration Mixture in hexane to 50 mL with acetone. Expires in 6 months.

9.17 LCS/MS Spiking Solution – (0.5-2.5 ppm) –Dilute 10 mL of stock LCS/MS Spiking solution (1-5 ppm) to 20 mL with acetone. Expires in 6 months.

9.18 CCV – Also known as Laboratory Performance Check Solution - A calibration standard (Calibration Standard 1) is used as the laboratory performance check solution to verify response factors and to demonstrate adequate GC resolution and MS performance.

9.19 MB – One liter of reagent water spiked with surrogate and extracted as a sample.

10 RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

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11 PROCEDURE

11.1 Be sure the system has a current MDL and the analyst has current training documentation (IDC/CDC).

11.2 Sample Extraction:

11.2.1 Mark the meniscus on the side of the samples bottle for later determination of sample volume. Pour entire sample into a 2-L separatory funnel.

11.2.2 Add 0.5 ml of surrogate compound solution.

11.2.3 Add 60 ml of methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 minutes with periodic venting to release excess pressure. Wait at least 10 minutes to allow the organic layer to separate from the water phase.

11.2.4 Collect the methylene chloride extract in a 250 mL erlenmeyer flask and add a second 60 ml volume of methylene chloride to the sample and repeat the extraction procedure a second and third time. Emulsions may form during the extraction and it is up to the discretion and experience of the analyst as to what methods may be used to break them up. Centrifugation, filtering through glass wool, are all means of dealing with emulsions.

11.3 Sample concentration by K-D

11.3.1 Assemble Kurderna-Danish (K-D) apparatus. Attach 10 mL concentrator tube to 500 mL evaporator flask, and add several small glass beads.

11.3.2 Pour the combined extract into a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate. Rinse the erlenmeyer flask with a 20-30 ml portion of methylene chloride and add the rinse to the drying column. Collect the combined extract in the K-D concentrator.

11.3.3 Add sample to evaporator flask and connect Snyder column to top. Place on steam bath (60-65°C) so the concentrator tube is partially immersed in water.

11.3.4 Allow the sample to concentrate to approximately 1 ml (approximately 15-20 minutes.). Remove K-D flask from steam bath and allow to cool for at least 10 minutes.

11.3.5 Remove Snyder column and rinse down inside of evaporator flask and joint with 1-2 ml methylene chloride. Allow solvent to drain down into the concentrator tube. Using nitrogen gas blowdown apparatus, concentrate the extract to 0.5 ml.

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If the extract is to be stored for more than two days, transfer it to a teflon-sealed screw-cap vial.

11.4 Sample concentration by **LabConco RapidVap Nitrogen Evaporation System**

11.4.1 Add extract to a 600 mL Sample tube

11.4.2 Turn on power switch

11.4.3 Set parameters – Input the % Speed, Temperature, Time and Number of Samples to be concentrated. (See users manual for Time, Temperature and Speed Setting Guidelines per volume per solvent.)

11.4.4 Turn on Nitrogen gas.

11.4.5 Load sample tubes into RapidVap chamber. Close lid and swing both latched over the lid and tighten knobs.

11.4.6 Press Run to begin concentration. Be sure to check the samples at the beginning of the program to ensure samples are not rotating so quickly that they are splashing out of the sample tube. If this occurs press the Stop button and adjust the speed. To resume press Run.

11.4.7 Allow samples to evaporate to a final volume of ~ 1 mL. When the time in the program is complete the instrument will beep and stop. Check sample volume. If additional time is needed reset program and press Run. Once desired final volume is reached, concentrate further by Nitrogen Blowdown to 0.5 mL.

11.5 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000 ml graduated cylinder. Record the sample volume to the nearest 5 ml.

11.6 CALIBRATION

Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required intermittently throughout sample analyses as dictated by results of continuing calibration checks. After initial calibration is successfully performed, a continuing calibration check is required at the beginning and end of each 12-hour period during which analyses are performed.

Optimize conditions for analyte separation and sensitivity. Once optimized, the same conditions must be used for the analysis of all standards, samples, and batch QC samples.

11.6.1 Initial Calibration - SIM

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11.6.1.1 Tuning - Inject a 2-uL aliquot of the 25 ng/uL DFTPP solution and acquire a mass spectrum that includes data for m/z 35-500. The mass spectrum of DFTPP is acquired as follows: Three scans (the apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Alternatively, the scan preceding the apex and the scan following the apex can be used. When background subtraction is required, subtraction is accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. No part of the DFTPP peak may be used to background subtract. If the spectrum does not meet all criteria (Table 3), the MS must be hardware tuned to meet all criteria before proceeding with calibration.

11.6.1.2 Acquire at least six points for each ion during elution of each GC peak. Total cycle time should be ≥0.5 seconds and <1.5 seconds.

Note: When acquiring SIM data, GC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times; if not, ions will not be monitored at the appropriate times.

11.6.1.3 SIM data acquisition is provided using five ion sets (≤20 ions each) shown in Tables 4 and 5.

11.6.1.4 The time (scan number) for initiation of data acquisition with each ion set must be carefully determined from the retention times (scan numbers) of the retention time congeners.

11.6.1.5 Begin data acquisition with Ion Set #1 before elution of PCB congener #1, the first eluting Cl_1-PCB. Stop acquisition with Ion set #1 and begin acquisition with Ion Set #2 just (approximately 10 seconds) <u>before</u> elution of PCB congener #104, the first eluting Cl_5-PCB. Stop acquisition with Ion Set #2 and begin acquisition with Ion Set #3 just (approximately 10 seconds) <u>after</u> elution of PCB congener #77, the last eluting Cl_4-PCB. Stop acquisition with Ion Set #3 and begin acquisition with Ion Set #4 just (approximately 10 seconds) <u>after</u> elution of 4,4'-DDT. The fifth Ion Set beginning data acquisition with that set just (approximately 10 seconds) <u>before</u> elution of PCB congener #208, the first eluting Cl_9-PCB.

11.6.1.6 Performance criteria SIM PCB Data

GC separation - Baseline separation of PCB congener #87 from congener #154 and #77, which coelute.

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11.6.1.7 Response Factor Reproducibility

For each PCB calibration congener and surrogate compound, calculate and mean RF from analyses of each of the five CALS. The RSD should not exceed 20%. Alternatively, the analytes may be calibrated by method of linear regression. If the correlation coefficient <0.990 the calibration may not be used to quantitate samples and corrective action must be taken to eliminate the problem prior to a new calibration.

11.6.1.8 SIM Analyte Retention Time Reproducibility

PCB determinations - Absolute retention times of PCB congeners #77 and #104 should not vary by more than ±10 seconds from one analysis to the next. (Retention time reproducibility is not as critical for congeners #1 and #209 as for #77 and #104, which are used to determine when ion sets are changed.)

11.6.1.9 ICV – run the ICV immediately after the initial calibration. The result of the ICV must be within 70-130% of the true value. If it is not, determine the problem and recalibrate.

11.6.2 Continuing Calibration

11.6.2.1 With the following procedures, verify initial calibration at the beginning and end of each 12 hour period during which analyses are to be performed.

11.6.2.2 Calibrate and tune the MS with standards and procedures prescribed by the manufacturer.

11.6.2.3 Analyze a 2.0-uL aliquot of the DFTPP solution and ensure acceptable MS calibration and performance.

11.6.2.4 Inject a 2.0-uL aliquot of the Medium CAL and analyze with the same conditions used during Initial Calibration.

11.6.2.5 Determine that neither the area measured for m/z 240 nor that for chrysene-d_{12} has decreased by more than 30% from the area measured in the most recent previous analysis of a calibration solution or by more than 50% from the mean area measured during initial calibration.

11.6.2.6 Response Factor Reproducibility - For an acceptable Continuing Calibration Check, the measured RF for each analyte/surrogate compound must be within ±20% of the mean value calculated during

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Initial Calibration. If not, remedial action must be taken; recalibration may be necessary.

11.6.2.7 SIM Analyte Retention Time Reproducibility - Demonstrate and document acceptable reproducibility of absolute retention times of appropriate pesticide analytes and PCB retention times congeners.

11.6.2.8 Remedial actions must be taken if criteria are not met; possible remedies are:

- Check and adjust GC and/or MS operating conditions.
- Clean or replace injector liner.
- Break off a short portion of the column; check column performance by analysis of performance check solution.
- Replace GC column; performance of all initial calibration procedures then required.
- Adjust MS for greater or lesser resolution.
- Calibrate the MS mass scale.
- Prepare and analyze new concentration calibration/performance check solution.
- Prepare a new calibration curve.

11.7 GC/MS ANALYSIS

11.7.1 Add internal standard to extract.

11.7.2 Inject 2 uL of extract onto the GC operated under the same conditions used for calibration.

11.7.3 Examine data for saturated ions in mass spectra of target compounds, if saturation occurred, dilute and reanalyze the extract after the quantity of the internal standards is adjusted appropriately.

11.7.4 For each internal standard, determine that the area measured in the sample extract has not decreased by >30% from the area measured during the most recent previous analysis of the calibration solution or by >50% from the mean area measured during initial calibration. If either criterion is not met, remedial action must be taken to improve sensitivity, and the sample extract must be reanalyzed.

11.7.5 Analytical Sequence – When the Initial Calibration or Continuing Calibration requirements are met, analysis of client samples and batch QC samples (MB, LCS, MS/MSD) may begin. Samples may be analyzed until the close of the analytical window.

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11.7.6 Manual Integration – When the data system incorrectly quantitates or identifies analytes, manual integration is necessary. Data must be integrated consistently between standards, samples, and QC. See ADM-INT

11.8 INDENTIFICATION PROCEDURES

11.8.1 Using the ions shown in Tables 4 and 5 for PCBs, examine ion current profiles (ICPs) to locate internal standards, surrogate compounds, and PCBs for each isomer group.

11.8.2 SIM Data - Obtain appropriate selected ion current profiles (SIPS) for IS quantitation and confirmation ions for the quantitation and confirmation ions for each PCB isomer group.

11.8.3 For all PCB candidates, confirm the presence of an $(M\text{-}70)^+$ ion cluster by examining ICPs or spectra for at least one of the most intense ions in the appropriate ion cluster.

11.8.4 For Cl_3-Cl_7 isomer groups, examine ICPs or spectra for intense $(M+ 70)^+$ ions that would indicate a coeluting PCB containing two additional chlorines. (GC retention time data show that this is not a potential problem for other PCB isomer groups. If this interference occurs, a correction can be made. Obtain and record the area for the appropriate ion (Table 6) for the candidate PCB isomer group. Use the information in Table 7 to correct the measured abundance of M^+. For example, if a Cl_7-PCB and a Cl_5-PCB candidate coelute, the Cl_7-PCB will contribute to the ion measured for m/z 326 and m/z 324, the quantitation and confirmation ions, respectively, for a Cl_5-PCB. Obtain and record the area for m/z 322 (the lowest mass ion in the $(M^+\text{-}70)^+$ ion cluster of a Cl_5-PCB fragment produced by a Cl_7-PCB. To determine the m/z 326 and m/z 324 areas produced by the Cl_5-PCB, calculate the Cl_7-PCB contribution to each and subtract it from the measured area. In this example, 164% of the area measured for m/z 324, and 108% of the m/z 322 area should be subtracted from the area should be subtracted from the area measured for m/z 326 (Table 7).

11.8.5 For Cl_2-Cl_8-PCB candidates, examine ICPs or spectra for intense $(M+35)^+$ ions that would indicate a coeluting PCB containing one additional chlorine. This coelution causes interferences because of the natural abundance of ^{13}C. (This interference will be small and can be neglected except when measuring the area of a small amount of a PCB coeluting with a large amount of another PCB containing one more chlorine.) To correct for this interference, obtain and record the area for the appropriate ion (Table 8) from the $(M\text{-}1)^+$ ion cluster, and subtract 13.5% of the area measured for the $(M\text{-}1)^+$ ion from the measured area of the quantitation ion. For example, for Cl_5 - PCB candidates,

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obtain and record the area for m/z 325: subtract 13.5% of that area from the measured area of m/z 326.

11.8.6 All Analytes -- Use ICP data to calculate the ratio of the measured peak areas of the quantitation ion and confirmation ion(s), and compare to the acceptable ratio (Table 6). If acceptable ratios are not obtained, a coeluting or partially coeluting compound may be interfering. Examination of the data from several scans may provide information that will allow application of additional background corrections to improve the ion ratio.

11.8.7 Retention time must be within ± 10 seconds of that observed during the most recent acceptable calibration.

11.8.8 Absolute retention times of surrogate compounds must be within ±10 seconds of that measured during the last previous continuing calibration check.

11.8.9 Quantitation and confirmation ions for each PCB isomer group must maximize within ±1 scan of each other.

11.8.10 The integrated ion current for each quantitation and confirmation ion must be at least three time background noise and must not have saturated the detector.

11.8.11 For each PCB isomer group candidate, the ratio of the quantitation ion area to the confirmation ion area must be within limits shown in Table 6; at least one ion in the $(M-70)^+$ ion cluster must be present.

12 QA/QC REQUIREMENTS

12.1 The requirements for the ICV, CCV, internal standards and sample identification are listed in the procedure.

12.2 Method Blank – one MB must be extracted with each batch. The MB must demonstrate that interferences from the analytical and preparation steps are under control. No target analytes should be detected above the reporting limit. If the MB has contamination, determine and correct the cause and re-extract and re-analyze the samples, if possible. If it is not possible, flag the associated data.

12.3 Surrogates – The surrogate recovery must be within the limits in Appendix C of the Quality Assurance Manual. If surrogates(s) fail acceptance, the sample must be evaluated for matrix interferences and "historical results". If none are present, then correct any problems and re-extract and re-analyze the associated samples, if possible. If it is not possible due to limited sample volume, holding times, etc., flag the associated data.

12.4 LCS – The LCS recovery must be within the limits in Appendix C of the Quality Assurance Manual. One LCS must be extracted with each batch. If the lab control sample (LCS) fails

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acceptance limits for any of the spike compounds, the analyst must evaluate the system and calibration. Correct any problems and re-extract and re-analyze the associated samples, if possible. If it is not possible due to limited sample volume, holding times, etc., flag the associated data.

12.5 Matrix Spike/Matrix Spike Duplicate –

12.5.1 Frequency - One MS and one MSD should be extracted with each batch. If there is insufficient volume of sample to perform a MSD, an LCSD should be performed to demonstrate precision for the batch.

12.5.2 Recovery - The recovery of the MS/MSD should be within the limits in Appendix C of the Quality Assurance Manual. If the matrix spike fails acceptance, the sample must be evaluated for matrix interferences. If recovery of the MS or MSD fails on a sample, the batch is considered acceptable as long as the LCS meets acceptance criteria. It is recommended that the MS/MSD be reprepared and reanalyzed to confirm the outliers, however it is not required due to probable matrix interferences. If a duplicate LCS was run with the batch and one LCS was slightly out, but the other LCS was in, the data is accepted.

12.5.3 RPD – if the RPD value between samples or MS/MSD results exceed limits listed in Appendix C of the QAM, examine the chromatograms and benchsheets for potential matrix interferences. Examples may include product layers on aqueous samples that may result in non-homogenous subsampling, non-homogenous soil samples, chromatographic interferences resulting in poor peak resolution and inconsistent integrations, or poor purging efficiencies (indicated by surrogate recovery). Reanalyze the pair if deemed appropriate. The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately.

13 DATA REDUCTION AND REPORTING

13.1 Response Factor Calculation

Calculate response factors (RFs) for each PCB calibration congener, and surrogate compound relative to chrysene-d_{12}:

$$RF = A_x * Q_{is} / A_{is} * Q_x$$

where:

A_x = integrated ion abundance of quantitation ion for a PCB calibration congener or a surrogate compound,

A_{is} = integrated ion abundance of m/z 240, the quantitation ion when chrysene-d_{12} is used.

Q_{is} = injected quantity of chrysene-d_{12}

Q_x = injected quantity of PCB calibration congener or surrogate compound.

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RF is a unitless number, units used to express quantities must be equivalent.

13.2 From appropriate ICPs of quantitation ions, obtain and record the spectrum number of the chromatographic peak apex and the area of the entire chromatographic peak.

13.3 For PCBs, sum the areas for all isomers identified at each level of chlorination (i.e. sum all quantitation ion areas for Cl_4-PCBs).

13.4 Calculate the concentration of each surrogate compound and PCB isomer group using the formula:

$$C\ (ug/L) = (A_x * Q_{is})/(A_{is} * RF * V)$$

where:

C = Concentration

A_x = the sum of quantitation ion areas for all PCB isomers at a particular level of chlorination.

A_{is} = the area of internal standard quantitation ion, m/z 240 for chrysene-d_{12} or m/z 188 for phenanthrene-d_{10}.

Qis = quantity (ug) of internal standard added to extract before GC/MS analysis.

RF = calculated response factor for the surrogate compound or the PCB calibration compound for the isomer group (level of chlorination)

V = Volume in liters of water extracted.

13.5 Data is reviewed according to ADM-DREV.

14 METHOD PERFORMANCE

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

Demonstration of Capability is performed upon instrument set-up , whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office

Single laboratory accuracy and precision data is available in EPA Method 680.

15 WASTE MANAGEMENT AND POLLUTION PREVENTION

Standards and reagents are prepared on an as-needed basis in small quantities and minimum sample volumes are used during analysis. Samples are disposed according to SMO-SPLDIS.

16 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

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If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17 CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18 REFERENCES

Method 680 *Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography/Mass Spectrometry*, USEPA Environmental Monitoring and Support Laboratory Office of Research and Development, November 1985.

19 TRAINING OUTLINE

19.1 Review literature by reading EPA Method 680 . Most recent SOPs must be reviewed and documented on the training records. Follow policies in ADM-TRANDOC.

19.2 Observe the procedure as performed by an experienced analyst at least three times. Follow Training Plan Form. The analyst is to transition from observing to assisting to performing this SOP with supervision.

19.3 When the analyst is ready to perform the analysis independently, perform an IDC study by preparing and analyzing four replicate laboratory control samples. Calculate average percent recovery and relative standard deviation for the four replicate analyses on a spreadsheet. The acceptance criteria is the same as the LCS limits in Appendix C of the Quality Assurance Manual for each congener. Complete the summary spreadsheet, IDC certificate, and Training Plan Form and file with QA. Continuing demonstration of capability is performed annually using a PE, a blind, or a new four replicate study.

20 METHOD MODIFICATIONS

This SOP does not include a solvent exchange to hexane and the calibration standards are prepared in methylene chloride.

21 INSTRUMENT SPECIFIC ADDENDUM

None

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22 ATTACHMENTS

Table 1 – PCB Congeners Used as Calibration Standards
Table 2 – Composition and Approximate Concentrations of Calibration Solutions
Table 3 – DFTPP Tune Criteria
Table 4 – Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of <20 Ions Each.
Table 5 - Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of <20 Ions Each.
Table 6 – Quantitation, Confirmation, and Interference Check Ions for PCBs, Internal Standards, and Surrogate Standards
Table 7 – Correction for Interference of PCB Containing Two Additional Chlorines
Table 8 - Correction for Interference of PCB Containing One Additional Chlorine

23 CHANGES FROM PREVIOUS REVISION

- Section 1-Added Reference to MRLs being in QAM.
- Section 3 – Definitions – Added Analysis Window, Retention Time Window, Matrix, Sample, and MDL. Changed the ICV to being prepared by a second analyst instead of being from a second source standard. Grouped MS, MB, LCS under QA/QC samples
- Section 5 – Safety – reworded for consistency. Added reference to Safety Manual. Added section on gas cylinders
- Section 8 – Preventive Maintenance – Entirely new to reflect GC/MS methods
- Section 9 – Standards and Reagents – Changed concentrations of calibration standards. Added General info and Disclaimers. Changed the working tune standard from 50 to 25 ppm. Changed the injection from 1 uL to 2 uL.
- Added 11.1 – check for current MDL and IDC
- Added 11.6 about operating conditions being the same for standards, QC, and samples
- Changed all injections from 1 uL to 2 uL
- Added Analytical Sequence to 11
- Added reference to Manual Integration in 11.
- Expanded upon batch acceptance due to the MS in Section 12. Added section about RPD of MS/MSD. Mentioned that batch is accepted if one LCS out of LCS/LCSD is acceptable and the other is marginal.
- Training – took out use of single LCS for CDC.

Table 1

PCB Congeners Used as Calibration Standards

PCB Isomer Group	Congener Number[a]	Chlorine Substitution
Concentration Calibration Standard		
Monochlorobiphenyl	1	2
Dichlorobiphenyl	5	2, 3
Trichlorobiphenyl	29	2, 4, 5
Tetrachlorobiphenyl	50	2, 2', 4, 6
Pentachlorobiphenyl	87	2, 2', 3, 4, 5'
Hexachlorobiphenyl	154	2, 2', 4, 4', 5, 6'
Heptachlorobiphenyl	188	2, 2', 3, 4', 5, 6, 6'
Octachlorobiphenyl	200	2, 2', 3, 3', 4, 5', 6, 6'
Nonachlorobiphenyl[b]	-	---
Decachlorobiphenyl	209	2, 2', 3, 3', 4, 4', 5, 5', 6, 6'
Retention Time Calibration Standards		
Tetrachlorobiphenyl	77	3, 3', 4, 4'
Pentachlorobiphenyl	104	2, 2', 4, 6, 6'
Nonachlorobiphenyl	208	2, 2', 3, 3', 4, 5, 5', 6, 7'

[a] Numbered according to the system of Ballschmiter and Zell (2).
[b] Decachlorobiphenyl is used as the calibration congener for both nona- and decachlorobiphenyl isomer groups.

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Table 2 Composition and Approximate Concentrations of Calibration Standards

Concentrations in ppm

Compound	CAL 0	CAL 0.5	CAL 1	CAL 1.5	CAL 2	CAL 3	Stock
Cal Congener							
Cl_1 (#1)	0.02	0.05	0.1	0.25	0.5	1.0	50
Cl_2 (#5)	0.02	0.05	0.1	0.25	0.5	1.0	50
Cl_3 (#29)	0.02	0.05	0.1	0.25	0.5	1.0	50
Cl_4 (#50)	0.04	0.1	0.2	0.5	1.0	2.0	100
Cl_5 (#87)	0.04	0.1	0.2	0.5	1.0	2.0	100
Cl_6 (#154)	0.04	0.1	0.2	0.5	1.0	2.0	100
Cl_7 (#188)	0.06	0.15	0.3	0.75	1.5	3.0	150
Cl_8 (#200)	0.06	0.15	0.3	0.75	1.5	3.0	150
Cl_{10} (#209)	0.1	0.25	0.5	1.25	2.5	5.0	250
RT Congeners							
Cl_4 (#77)	0.04	0.1	0.2	0.5	4	10	100
Cl_5 (#104)	0.04	0.1	0.2	0.5	4	10	100
Cl_9 (#208)	0.08	0.2	0.4	1.0	8	20	200
Internal Standards							
Chrysene-d_{12}	0.75	0.75	0.75	0.75	0.75	0.75	
Phenanthrene-d_{10}	0.75	0.75	0.75	0.75	0.75	0.75	
Surrogate Compounds							
$^{13}C_6$-gamma BHC	0.04	0.1	0.2	0.5	4	10	
$^{13}c_{12}$-4,4'-DDT	0.04	0.1	0.2	0.5	4	10	

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Table 3

Criteria for DFTPP Spectrum

m/z	**Relative Abundance**
127	40-60%
197	<1%
198	100% (Base Peak)
199	5-9%
275	10-30%
365	>1%
441	Present and m/z 443
442	>40%
443	17-23% of m/z 442

Table 4

Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Sets of ≤20 Ions Each

Ion Set	Isomer Group/ IS/Surrogate	Quant. Ion	Confirm. Ions	M-70 Ions	M+70 Ions	M+35 Ions	Ion Measured[a] for Correction	
1	Cl_1	188	190	152,153[b]	256,258	222,224	-	-
	Cl_2	222	224	152,153,186,188[c]	290,292,294	256,258	-	221
	Cl_3	256	258	186,188	-	290,292,294	-	225
	Cl_4	292	290,294	220,222	-	-	-	-
	$^{13}C_6$-gamma-BHC	187	189	-	-	-	-	-
	Phenanthrene-d_{10}	188	189	-	-	-	-	-
2	Cl_3	256	258	188,188	324,326,328	290,292,294	254	255
	Cl_4	292	290,294	220,222	360,362	324,326,328	288	289
	Cl_5	326	324,328	254,256,258	-	360,362	-	323
	Cl_6	360	358,362	288,290,292	-	-	-	-
3	Cl_5	326	324,328	254,256	392,394,396,398	360,362	322	323
	Cl_6	360	358, 362	288,290	-	392,394,396,398	-	357
	Cl_7	394	392, 396	322,324,326	-	-	-	-
	$^{13}C_{12}$-4,4'-DDT	247	249	-	-	-	-	-
4	Cl_6	360	358,362	288,290	426,428,430,432	392,394,396	356	357
	Cl_7	394	392,396,398	322,324	-	428,430,432	-	391
	Cl_8	430	428,432	356,358,360	-	-	-	-
	Chrysene-d_{12}	240	241	-	-	-	-	-
5	Cl_8	430	426,428,432	356,358,360	494,496,498,500	462,464,466	-	425
	Cl_9	464	460,462,466	390,392,394	-	496,498,500	-	-
	Cl_{10}	498	494,496,500	424,426,428,430	-	-	-	-

[a]See Tables 12-13.
[b]Cl_1-PCBs lose HCl.
[c] Some Cl_2-PCBs lose Cl_2 and some lose Hcl.

Table 5

Ions for Selected Ion Monitoring to Determine PCBs by Acquiring Data for Five Ion Sets of ≤20 Ions

Ion Set No. 1[a]	Ion Set No. 2[b]	Ion Set No. 3[c]	Ion Set No. 4[d]	Ion Set No. 5[e]
152	186	247	240	356
153	188	249	241	358
186	220	254	288	360
187	222	256	290	390
188	254	288	322	392
189	255	290	324	394
190	256	322	326	424
220	258	323	356	425
221	288	324	357	426
222	289	326	358	428
224	290	328	360	430
255	292	357	362	432
256	294	358	391	462
258	323	360	392	464
290	324	362	394	466
292	326	392	396	496
294	328	394	398	498
	358	396	428	499
	360	398	430	500
	362		432	502
17 ions	20 ions	19 ions	20 ions	20 ions

[a] Ions to identify and measure Cl_1-Cl_4-PCBs, phenanthrene-d_{10}, and $^{13}C_6$-gamma-BHC.

[b] Ions to identify and measure Cl_3-Cl_6-PCBs.

[c] Ions to identify and measure Cl_5-Cl_7-PCBs and $^{13}C_{12}$-4, 4'-DDT.

[d] Ions to identify and measure Cl_6-Cl_8-PCBs and chrysene-d_{12}.

[e] Ions to identify and measure Cl_8-Cl_{10}-PCBs.

Table 6

Quantitation, Confirmation, and Interference Check Ions for PCBs, Internal Standards, and Surrogate Compounds

Analyte/ Internal Std.	Nom. MW	Quant. Ion	Confirm. Ion	Expected Ratio[a]	Accept. Ratio[a]	M-70 Confirm. Ion	Interference Check Ions M+70	Interference Check Ions M+35
PCB Isomer Group								
Cl_1	188	188	190	3.0	2.5-3.5	152[b]	256	222
Cl_2	222	222	224	1.5	1.3-1.7	152	292	256
Cl_3	256	256	258	1.0	0.8-1.2	186	326	290
Cl_4	290	292	290	1.3	1.1-1.5	220	360	326
Cl_5	324	326	324	1.6	1.4-1.8	254	394	360
Cl_6	358	360	362	1.2	1.0-1.4	288	430	394
Cl_7	392	394	396	1.0	0.9-1.2	322	464	430
Cl_8	426	430	428	1.1	0.9-1.3	356	498	464
Cl_9	460	464	466	1.3	1.1-1.5	390	-	498
Cl_{10}	494	498	500	1.1	0.9-1.3	424	-	-
Internal standards								
Chrysene-d_{12}	240	240	241	5.1	4.3-5.9	-	-	-
Phenanthrene-d_{10}	188	188	189	6.6	6.0-7.2	-	-	-
Surrogate compounds								
$^{13}C_6$-gamma-BHC	294	187	189	1.0	0.8-1.2	-	-	-
$^{13}C_{12}$-4,4'-DDT	364	247	249	1.5	1.3-1.7	-	-	-

[a] Ratio of quantitation ion to confirmation ion

[b] Monodichlorobipheyls lose HCl to produce an ion at m/z 152.

Table 7 Correction for Interference of PCB Containing Two Additional Chlorines

Candidate Isomer Group	Quant. Ion	Confirm. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subtracted from	
				Quant Ion Area	Confirm. Ion Area
Trichlorobiphenyls	256	258	254	99%	33%
Tetrachlorobiphenyls	292	290	288	65%	131%
Pentachlorobiphenyls	326	324	322	108%	164%
Hexachlorobiphenyls	360	362	356	161%	71%
Heptachlorobiphenyls	394	396	390	225%	123%

Table 8 Correction for Interference of PCB Containing One Additional Chlorine

Candidate Isomer Group	Quant. Ion	Ion Measured to Determine Interference	% of Meas. Ion Area to be Subtracted from Quant. Ion Area
Dichlorobiphenyls	222	221	13.5%
Trichlorobiphenyls	256	255	13.5%
Tetrachlorobiphenyls	292	289	17.4%
Pentachlorobiphenyls	326	323	22.0%
Hexachlorobiphenyls	360	357	26.5%
Heptachlorobiphenyls	394	391	30.9%
Octachlorobiphenyls	430	425	40.0%

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STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS AND LOW LEVEL ANALYSIS USING A LARGE VOLUME INJECTOR (LVI)

SOC-8270C

Revision 6

March 31, 2006

Approved By: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Manager Date

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1 Mustard Street, Suite 250
Rochester, New York 14609

Annual review of this SOP has been performed
and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

*See Appendix B for DOD Specific Criteria

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1. SCOPE AND APPLICATION

1.1. This SOP uses EPA SW-846 Method 8270C to determine the concentrations of Semi-Volatile Organic Compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media, and water. See Table 1 for a list of the analytes which may be analyzed by this SOP with the associated reporting limits for water and soil. The reported compound list and reporting limits may be adjusted based on the client's needs. Samples may also be analyzed by Method 8270C using a GC/MS equipped with a Large Volume Injector (LVI) to obtain a lower reporting limit. See Appendix A for 8270C LVI reporting limits, compound list, and standards preparation instructions. All other recommendations for low level analysis are noted herein.

1.2. Method 8270C can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. A 1 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass spectrometer. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

Note: A 2 µL aliquot of the extract is injected into the gas chromatograph (GC) equipped with an LVI for low level analyses.

*See Appendix B for DOD Specific Criteria

2.2. The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and the chromatography for this compound is poor.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph and is subject to a chemical reaction in acetone, and can undergo photochemical decomposition.
- N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described.
- N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. DEFINITIONS

3.1. **Analytical Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with injection of Decaflurortriphenylphsophine (DFTPP) followed by initial calibration standard(s). Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP injection time.

3.2. **Initial Calibration -** analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the detector to the target compounds.

3.3. **Laboratory Control Sample (LCS)-** a blank matrix with at least all of the target compounds needed for the batch. This sample is used to document laboratory performance including all of the extraction and analysis steps for each batch.

3.4. **Matrix -** the predominant material, component, or substrate (e.g., surface water, drinking water, etc.) of which the sample to be analyzed is composed.

3.5. **Matrix Spike/Matrix Spike Duplicate (MS/MSD)-** duplicate aliquots of sample are spiked with a known concentration of all target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix. The MSD is used as a measure of precision.

3.6. **Method Blank (MB)-** an analyte-free matrix to which all reagents are added in the sample volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

3.7. **Organic- Free Reagent Water** - ASTM Type II Deionized Water .

*See Appendix B for DOD Specific Criteria

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3.8. **Percent Drift or Percent Difference (%D) -** Used to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference).

3.9. **Sample -** a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

3.10. **Surrogates** (Surrogate Standards) - an organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process. For semivolatiles and pesticides/Aroclors, surrogate compounds are added to every blank, sample, matrix spike, matrix spike duplicate, LCS, matrix spike blank, and standard. These are used to evaluate analytical efficiency by measuring recovery. Surrogates are not expected to be detected in environmental media.

3.11. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.12. **Independent Calibration Verification Standard (ICV)** – a mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

3.13. **Relative Percent Difference (RPD)** – The absolute value of the difference of two values divided by the average of the same two values. Used to compare the precision of the analysis. The result is always a positive number.

3.14. **Analysis Window** – Samples are analyzed in a set referred to as a "window". The window begins with the injection of the tune verification standard. Standards, required QC samples, and samples may be run for 12 hours in this window. A new window must be opened to continue analysis.

3.15. **% Relative Standard Deviation (%RSD):** statistical measure of variation. Used in this method to measure the relative variation of initial calibration standards. Calculated by dividing the standard deviation of the individual calibration factors by the average calibration factor and multiplying by 100 to express as a percentage.

3.16. **Internal Standards –** Internal standards are organic compounds which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to calibrate the instrument's response.

3.17. **Preparation Batch –** A group of 20 or fewer samples of the same matrix prepared together on the same day. See ADM-BATCH for further discussion.

3.18. **Analytical Batch –** A group of 20 or fewer samples analyzed together. See ADM-BATCH for further discussion.

*See Appendix B for DOD Specific Criteria

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3.19. **Method Detection Limit (MDL):** a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero

3.20. **Method Reporting Limit (MRL):** The minimum amount of a target analyte that can be measured and reported quantitatively. The MRL is equivalent to Limit of Quantitation (LOQ), Practical Quantitation Level (PQL) and Estimated Quantitation Level (EQL). Typically, the MRL is calculated as five times the MDL (although this is a rule of thumb and not intended to be a strict policy of establishing the MRL for a compound).

3.21. **Target Analyte** – a compound of interest for which the method is capable of measuring.

4. INTERFERENCES

4.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples. Corrective action should be taken to eliminate the interferences.

4.2. Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination. Follow glassware cleaning procedures in EXT-GC.

4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

*See Appendix B for DOD Specific Criteria

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5. SAFETY

All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves are good protection to use while rinsing, pouring or transferring the solvent.

Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

Hydrochloric Acid is used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions

Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

Refer to the Safety Manual for further discussion of general safety procedures and information.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Purchased, precleaned, certified sample containers should be glass or Teflon, and have screw-caps with Teflon lined septa. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may <u>NOT</u> be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Solid samples should be collected (received) in 2 or 4 ounce glass jars with Teflon lined lids. Alternatively, soil samples may be received in brass sleeves prepared in the field.

6.3. Aqueous Samples should be collected (received) in purchased, precleaned, certified 1 Liter amber glass containers with Teflon lined caps or septa.

*See Appendix B for DOD Specific Criteria

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6.4. Waste samples such as oils can be collected (received) in a variety of sample containers.

6.5. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinsate as a field blank.

6.6. Samples are to be iced or refrigerated to 0-6°C from the time of collection until extraction.

6.7. Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. There is no holding time for non-aqueous liquid samples. These samples should be analyzed within a reasonable timeframe to meet the needs of the client.

6.8. Sample receipt, handling, and custody is discussed in SMO-GEN.

7. APPARATUS AND MATERIALS

7.1. Vials and caps - 20 mL scintillation vials with screw cap and aluminum foil liner, 2 ml capacity glass with crimp tops for GC auto sampler.

7.2. Pipets- glass, class A, volumetric 1 mL or 2 mL.

7.3. Gas Chromatograph/Mass Spectrometer System - See Appendix A of the Quality Assurance Manual for configuration of specific components, serial numbers and receipt of the major components of the instruments. See Appendix C of this SOP for Instrument Operating Conditions.

7.3.1. Large Volume Injector (LVI) – Injection system which allows for a greater quantity of analyte to be introduced to the GC/MS than standard injection systems, thus increasing measure sensitivity for that analyte.

7.3.2. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source (HP6890 GC).

7.3.3. Column: Phenomenex ZB-5 – 30m x 0.25 mm ID x 0.50 µm df fused-silica capillary column with a 10 meter guard column attached, or equivalent.

*See Appendix B for DOD Specific Criteria

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7.3.4. 8270C LVI Column: Restek RTX-5 SIL MS, 30 – m x 0.25 mm ID x 0.50 µm df fused-silica capillary column with a 10 meter guard column attached, or equivalent

7.3.5. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 2 when 1 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP) (HP5973).

7.3.6. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.3.7. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.3.8. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. PREVENTATIVE MAINTENANCE

8.1. Glass Insert/ Sleeve- Replace as needed. The insert should always be replaced before a new calibration and the o-ring should be replaced with the insert. Indicators include:

- A significant or sudden change in chromatography, or
- Difficulty in meeting % deviation criteria for a calibration check, or
- Reduced sensitivity to key compounds (Nitrophenols, Hexachlorocyclopentadiene) is the best symptom of a dirty injector.

8.2. Septum - Replaced approximately every 150 injections. A retention time shift or poor baseline chromatography is a common symptom.

8.3. Autosampler syringe - Cleaned daily and after samples with a particularly bad matrix. The syringe should be rinsed with methylene chloride; the plunger is rinsed, wiped with a Kimwipe, and rinsed again. The syringe should be replaced with a new one if

*See Appendix B for DOD Specific Criteria

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loose action is noted or inconsistent action is evident (uniform fluctuation in response, area counts) or obvious wear.

8.4. Injection Port - The port and the port/column interface should be cleaned as needed (when replacing the insert is ineffective and in conjunction with column pruning). Port should be reamed with a wire brush and swabbed with methylene chloride. Disc and washer can be sonicated in methylene chloride and then silanized, but should be replaced if wear is evident. The capillary inlet should also be cleaned in methylene chloride and the septum nut is sonicated in methylene chloride also.

8.5. Column Pruning - Cut back one revolution as needed. Indicators are same as insert replacement and is another remedy if insert replacement does not yield the desired results. After initial cut a graphite ferrule should first be installed and then another short length of column removed (cut should be carefully examined for smoothness - no jagged edges). The column end should extend 6 mm past the ferrule when installed (use the white out or septum marking method for reference).

8.6. Column Replacement - As needed, usually every 2-3 months for moderate sample volumes. Symptoms are ineffectiveness of insert replacement, injector cleaning, or column pruning to rectify a problem or when resolution becomes difficult (such as key isomeric pairs: anthracene/ phenanthrene, chrysene/ benzo(a)anthracene, and benzo (b)/benzo(k)fluoranthene). All ferrules replaced with column change.

8.7. Syringe Rinse Solvent - Supply is replenished as needed. Old solvent is discarded and vial rinsed before refilling. Rinse waste and sample waste vials are replaced when residue appears.

8.8. Carrier Gas - Tank is changed before pressure reaches approximately 200psi to avoid impurities that may be delivered by a nearly empty tank. Leak check with snoop after change.

8.9. System Baking - When the system is to be idle oven temperature should be set to 100°C to avoid water vapor condensation in column. Baking of injector, column, interface may be done for short periods of time to eliminate contaminants due to sample matrix residuals. Temperatures should not exceed recommended column temperature limits.

8.10. Source Cleaning - Performed on an as-needed basis. Source is cleaned/replaced when persistent difficulty in meeting and maintaining acceptable DFTPP tune checks is encountered. This is indicated by : 1.) reduced high-mass sensitivity (DFTPP m/z 365<1% of 198 or PFTBA m/z 502<0.5%) 2.) High EM setting resulting in excessive signal and inconsistent response in both tune file and instrument methods. A dirty source can sometimes be diagnosed by jagged lens-ramp plots in addition to tune difficulties. The procedure for removing and cleaning the ion source is followed as in the instrument Manual.

*See Appendix B for DOD Specific Criteria

8.11. Electron Multiplier - Preventive Maintenance or replacement as needed. Method of cleaning and conditioning a new EM can be found in accompanying literature from the manufacturer (Channeltron/Galileo). Cleaning is usually necessary when source replacement is not having a significant effect on instrument sensitivity. Replacement of the EM is necessary when cleaning does not yield the desired results, or when there is a catastrophic event (outlined in literature).

8.12. Leak Detection - A manual tune should be monitored at set intervals for peak characteristics of air leakage (abundance of ions corresponding to N_2, H_2O, O_2), or an Ion gauge reading taken regularly can show changes in vacuum. Proper Ion gauge readings should be in the 10^{-5} - 10^{-6} torr range.

8.13. Internal Standard Syringe - Rinsed with methylene chloride before and after use. The Teflon tip is checked for wear regularly and the tip, plunger or entire syringe is replaced when necessary.

8.14. Splitless Liner Preparation - Remove dirty glass wool plug. Solvent rinse splitless liner with methylene chloride. Air dry and pack with glass wool to an approximate 5mm thickness. The glass wool packing should not be very dense. Immerse prepared liners in Sylon solution for deactivation. Rinse with methanol and air dry.

8.15. Maintenance log - All Preventive maintenance, as well as instrument repair, should be documented in the appropriate instrument maintenance log. Most routine maintenance and troubleshooting are performed by CAS staff. Other maintenance or repairs may, or may not require factory service, depending upon the nature of the task. Any maintenance performed by outside services must also be documented – either through notes in the log or through documents provided by the service. The log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements. The datafile name of the first acceptable run after maintenance is to be documented in the maintenance log.

8.16. Troubleshooting – see Instrument manual or maintenance log for help with solving specific analytical or instrument problems.

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9. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1. Solvents: Hexane, acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences. Solvents are to be checked for contamination before use. See ADM-CTMN.

9.2. **Standards Preparation General Information and Disclaimers**

All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mLs or may be made by adding 4 mL of 50 mg/L to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

The initial calibration curves given are typical, but also subject to variation due to targets and detection levels needed. The curves will always be at least 5 points. The lowest concentration level shall be at the method reporting level. The remaining levels should define the working linear range of the analytical system.

Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

Stock standard solutions-are purchased as certified solutions and expire per manufacturer recommendations. Protect all standards from light. Samples, sample extracts and standards must be stored separately.

All standards should be stored at -10-20°C. They expire upon manufacturer's indication, six months unless otherwise stated, or if check standards indicate a problem, whichever occurs first.

Note: See Appendix A for 8270C LVI Standard Preparation Instructions.

9.3. Internal Standard Solutions Purchased at 4000ppm - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See method for corresponding compounds). Each 1 ml sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 mg/L of each internal standard.

*See Appendix B for DOD Specific Criteria

9.4. Surrogate Standards - Determine what concentration should be in the blank extracts after all extraction, cleanup, and concentration steps. Determine recovery of surrogate standards in all blanks, spikes, and sample extracts. Take into account all dilutions of sample extracts. If surrogates are diluted more than 10 times, report as "D", diluted below calibration.

9.5. Tune standard solution (50 ppm) – Add 500 uL of 1000 ppm purchased stock DFTPP to a 10 mL flask and dilute to volume with methylene chloride. For the tune, inject 1 uL of this solution for a 50 ng injection.

9.6. Prepare a 200 ppm calibration mid-stock in methylene chloride

Purchased stock	Conc. stock (ppm)	volume of stock used (mL)	Final Volume (mL)
8270 Calib Mix 1	1000	1.0	5
8270 Calib Mix 4	2000	0.5	5
Nitrosodiphenylamine	5000	0.2	5
8270 Benzidine Mix	2000	0.5	5
CLP SOW OLM 4 Mix	2000	0.50	5
1-Methylnaphthalene	5000	0.20	5
2-chloropyridine	2000	0.50	5
3-chloropyridine	2000	0.50	5
B/N Surrogate	5000	0.20	5
Acid Surrogate	10000	0.10	5

9.7. <u>CALIBRATION STANDARDS</u>

Dilute the following volumes of the 200 ppm Mid-Stock Standard Solution to 1.0 ml with Methylene Chloride.

CALIBRATION STD. CONCENTRATION (ppm)	VOLUME OF 200 ppm MID-STOCK (mL)	FINAL VOLUME (mL)
10	0.05	1.0
50	0.25	1.0
80	0.40	1.0
100	0.50	1.0
120	0.60	1.0
160	0.80	1.0

Add 10 uL of 4000 ppm Internal Standard to each calibration standard prior to analysis.

9.8. <u>CCV Standard</u>

Prepare 1 mL of 80 ppm calibration standard and store in 2-ml autosampler vial. Add 10µl of 4000 ppm internal standard.

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9.9. ICV Standard

Dilute 0.40 mL of a purchased 200 ppm stock mix to 1.0 mL in Methylene Chloride OR dilute 0.40 mL of a 200 ppm midstock made from stocks of a vendor different from the calibration standard stocks to 1 mL with methylene chloride.

9.10. Surrogate

9.10.1. B/N Surrogate Mix 100 ppm (Nitrobenzene-d5, 2-fluorobiphenyl, Terphenyl-d14)

9.10.2. AE Surrogate Mix 200 ppm (2-fluorophenol, phenol-d6, 2,4,5-Tribromophenol)

9.10.3. Spike each sample with 1.0 mL surrogate solution for an on-column concentration of 100 ppm for BNs and 200 ppm for AEs.

9.11. **LCS and MS Spiking Solution -** Purchased 100ppm in acetone:methylene chloride (90:10). All targets are routinely spiked in the LCS and MS/MSD.

9.12. **LCS and MB –** prepare according to the appropriate extraction SOP.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

11. PROCEDURE

11.1. Be sure the system has a current MDL and the analyst has a current Demonstration of Capability.

11.2. Samples may be prepared using any of the following SOPs, depending upon requirements of the sample matrix and/or client request:

EXT-3510C	EXT-3620B
EXT-3520B	EXT-3640A
EXT-3540C	EXT-3660B
EXT-3550B	EXT-3665A
EXT-3580	

11.3. Tuning - The GC/MS system must meet the DFTPP ion abundance criteria shown in Table 2. If hardware tuning criteria can not be met, the source may need cleaning,

*See Appendix B for DOD Specific Criteria

filaments replaced or other maintenance. The Analysis may not proceed until the tune meets these criteria. The mass spectrum of DFTPP is acquired as follows: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Alternatively, the apex scan, the scan preceding the apex or the scan following the apex can be used. When background subtraction is required, subtraction is accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. No part of the DFTPP peak may be used to background subtract.

The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%.

Calculate percent breakdown as follows:

$$\%\ Breakdown = \frac{Total\ DDT\ degradation\ peak\ area\ (DDE\ +\ DDD)}{Total\ DDT\ peak\ area\ (DDT\ +\ DDE\ +\ DDD)}\ x\ 100$$

Lastly, Benzidine and Pentachlorophenol should be present at their normal responses and the tailing factors must be less than 3 and less than 5, respectively, and no peak tailing should be visible.

If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 in. of the capillary column. The use of a guard column between the injection port and the analytical column may help prolong analytical column performance.

11.4. Initial Calibration – Follow ADM-ICAL.

11.4.1. Frequency - Each GC/MS system must be initially calibrated prior to conducting any sample analysis, whenever major instrument maintenance or modification is performed or if the calibration verification technical acceptance criteria have not been met.

11.4.2. At least a 5 point calibration must be run. More point are usually run to ensure proper response of poorly performing compounds. The lowest concentration level should be at the reporting level. The remaining levels define the working linear range of the analytical system. Starting with the lowest level, analyze each calibration standard and tabulate the area response of the characteristic quantitation ions vs. concentration for each compound, internal standard and surrogate.

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11.4.3. The internal standards should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See method). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.4.4. Analyze 1 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound. Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF_x = \frac{(A_x)(C_{ISTD})}{(A_{ISTD})(C_x)}$$

Where: A_x =Area of the characteristic ion being measured.
A_{ISTD} =Area of the characteristic ion for the specified internal standard.
C_x =The concentration of the compound being measured (mg/L).
C_{ISTD} =The concentration of the specified internal standard (mg/L).

11.4.5. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compound (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dintro-phenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. The SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. If they are not acceptable, do maintenance; replace septa, linear, disks and/or cut 6-12" off the column.

11.4.6. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see Table 2) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{RF} \times 100$$

where:
RSD = relative standard deviation.
RF = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

*See Appendix B for DOD Specific Criteria

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}}$$

1

where:

RF_i = RF for each of the 5 calibration levels

N = Number of RF values (i.e., 5)

11.4.7. If the % RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.4.8. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If clients or state agencies do not allow average RSD, linear regression shall be used provided the correlation factor is ≥ 0.99.

11.4.8.1. If %RSD exceeds 15% the analyst may do one of the following:

- Calibration Curve, Linear Regression : This option may be used if the correlation coefficient is greater than or equal to 0.99*.

  This method of quantitation uses the equation of a line ($y=mx+b$). where:

  y = Instrument response (peak area or height)
  m = Slope of the line (also called the coefficient of x)
  x = Concentration of the calibration standard
  b = The intercept

  The curve <u>must not</u> be forced through zero. It will generally provide an acceptable fit when %RSD is between 0 - 30 percent and is therefore a useful option in evaluating the acceptability of the calibration curve for generating quantitative values, or

- If the compound with the RSD value greater than 15% is a non-target compound or non-detect in the associated samples, the analysis may continue with the present curve. The response for the compound must be evaluated to ensure it would be detected if present above the MDL.

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11.4.8.2. To determine the best approach, the analyst should assess the following:

11.4.8.2.1. Instrument's ability to attain MRL for this analyte. There needs to be sufficient sensitivity.

11.4.8.2.2. Low and high points of curve accurately quantitate. (Recalculate low and high points using curve. Value calculated should be within 50% on the low point and 30% on the high point). If this is not the case, the calibration needs to be rerun for that analyte. Evaluate performance of each standard. Reanalyze and replace a standard as needed. See CAS Calibration Policy for guidance.

11.4.8.2.3. Obtain a secondary opinion from a senior analyst or supervisor.

11.4.8.2.4. The data user should be made aware of those compounds exceeding 15% RSD.

11.4.9. ICV- inject and analyze the ICV to verify the initial calibration immediately after the curve. The percent recovery should meet 70-130%* for all target compounds. See ADM-ICAL. If an analyte's percent recovery is outside the limits, corrective action should be taken. However, some compounds may exceed this criteria and the initial calibration may still be valid. The analyst must use judgement when evaluating reactive compounds or those exhibiting poor chromatographic behavior. Analyses may continue if these analytes are not detected in the associated samples.

11.4.10. Instrument blank - Inject and analyze a blank. The results must be less than the reporting limit.

11.4.11. Only after the calibration has passed all of the above criteria may samples be analyzed.

11.5. Daily GC/MS Calibration Verification

11.5.1. A calibration standard at 80 ppm, containing all semivolatile analytes, including all required surrogates, must be analyzed every 12 hours during analysis. DFTPP must result in a mass spectrum for DFTPP which meets the criteria given above. These criteria must be demonstrated during each 12 hour shift.

11.5.2. System Performance Check Compounds (SPCCs): A system performance check must be made during every 12 hour shift. For each SPCC compound in the daily calibration a minimum response factor of 0.050 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation,

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injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

11.5.3. Calibration Check Compounds (CCCs): After the system performance check is met, the following CCCs are used to check the validity of the initial calibration:

Base/Neutral Fraction	**Acid Fraction**
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	Phenol
Diphenylamine	

- For linear regression calibration, calculate the percent drift using:

$$\% \, Drift = \frac{C_c - C_T}{C_T} x 100$$

where:
C_c = Calculated concentration of Calibration Check Compound standard.
C_T = Theoretical concentration of prepared standard.

- For calibrations based on RF, calculate the percent difference using:

$$\% \, Difference = \frac{RF_v - \overline{RF}}{\overline{RF}} x 100$$

where RF_v is the response factor from the analysis of the verification standard and $\overline{RF}$ is the mean response factor from the initial calibration.

If the percent difference or percent drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% difference or drift) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins. For non-CCC compounds, the % Drift or % Difference may exceed 20%, but should be less than 25%. Up to four analytes may exceed the 25%, but shall not exceed 40% if the analyte is being quantitated in the associated samples. Exceptions apply as follows:

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- If the CCV fails high, all associated non-detect samples may be reported, even if the MB or LCS fails high. Any samples with hits must be analyzed under a compliant CCV and the batch QC extracted with the samples must be re-run under a compliant CCV.

- If a CCV fails and another is run and passes, analysis may continue and data is accepted as long as the CCVs were within 12 hours of each other and no samples were run in between.

When any data is reported with a non-compliant CCV, the CCV is flagged in the report and associated sample results are to be considered less reliable.

11.5.4. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the last CCV corrections must be made, as appropriate. Once corrections have been made, reanalysis of associated samples is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.6. Daily Analytical Sequence

- Tune
- CCV
- Prepared samples (usually begins with MB, then LCS, then client samples)

11.7. GC/MS Analysis

11.7.1. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as was used for initial calibration.

11.7.2. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed. Dilutions are performed as per the company ADM-DIL SOP.

11.7.3. Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two injections after samples containing levels of target analyte near or over the high end of the calibration range. Reanalyze as needed to confirm.

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11.7.4. If the software integrates a peak incorrectly, follow the policies and procedures for manual integration in ADM-INT.

11.7.5. Store the extracts at 0-¯20°C, protected from light in screw-cap vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months after analysis.

11.8. Data Interpretation

11.8.1. The qualitative identification of compounds determined by this method is based on retention time, and comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

11.8.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion. See Table 3 for method-recommended ions for each analyte.

11.8.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

11.8.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

11.8.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.8.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative

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identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.8.2. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification.

12. QA/QC REQUIREMENTS

12.1. The ICV is discussed in the Procedure (Section 11)

12.2. Frequency of QC Samples - Method blanks, laboratory control samples, and matrix spikes are performed once per 20 samples. Client specific QC requirements shall always be considered for matrix-specific QC.

12.3. Acceptance Criteria and Corrective Action for QC Samples

12.3.1. Acceptance criteria for surrogates, lab control samples, matrix spikes, and duplicates are listed in Appendix C of the Quality Assurance Manual*under 8270C and 8270C LVI.

12.3.2. Evaluation of method blank acceptability is as follows. The method blank must demonstrate that interferences from the analytical and preparation steps are under control. No target analytes should be detected above the MRL* in the method blank, with the exception of common laboratory contaminants, such as phthalates. Phthalates are allowed in the method blanks up to a concentration of 5 times the reporting limit. If targets are detected greater than the MRL (or 5 times the MRL for phthalates), re-extract the batch or flag the data appropriately.

12.3.3. Surrogates

12.3.3.1. If the surrogates are outlying on the CCV, but the CCV is otherwise compliant, the associated data is accepted as long as the surrogate recoveries are acceptable in the samples.

12.3.3.2. If surrogates are diluted more than 10 times, report as "D", diluted below calibration. For package reports, include initial and confirmation analysis results. High outlying recoveries associated with non-detect sample results need not be re-extracted. They need only be noted in the case narrative as high bias with non-detect results.

12.3.3.3. If any of the surrogate compounds fail to meet the recovery acceptance criteria (Appendix C of the Quality Assurance Manual), check calculations, sample preparation logs, the surrogate compound spiking solutions, and the instrument operation. If the calculations were

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incorrect, correct the calculations and verify that the surrogate compound recoveries meet their acceptance criteria. If no surrogate spiking solution was added or if the surrogate spiking solution was improperly prepared, concentrated, or degraded, re-extract and reanalyze the samples. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. Sample results shall be reported from analysis in which both surrogates have acceptable recovery, unless matrix interferences are present

12.3.3.4. If the above actions do not correct the problem, then the problem may be due to a sample matrix effect. To determine if there was matrix effect, take the following corrective action steps.

12.3.3.4.1. Re-extract and reanalyze the sample. EXCEPTION: If surrogate compound recoveries in a sample used for a matrix spike and/or matrix spike duplicate were considered unacceptable, then it should be re-extracted/ re-analyzed only if surrogate compound recoveries met the surrogate acceptance criteria in both the matrix spike and matrix spike duplicate.

12.3.3.4.2. If the surrogate compound recoveries meet acceptance criteria in the re-extract/reanalyzed sample then the problem was within the Laboratory's control. Therefore, submit only data from the re-extraction/reanalysis.

12.3.3.4.3. If the surrogate recoveries fail to meet the acceptance criteria in the re-extracted/reanalyzed sample, then submit data from both analyses. Distinguish between the initial analysis and the re-extraction/ reanalysis on all.

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12.3.4. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix spike solution (which includes all target compounds of interest), and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Appendix C of the Quality Assurance Manual*. A subset of the entire list of analytes is used a control subset* of compounds which controls the acceptance of the batch. If the LCS recovery for any control analyte fails acceptance limits, corrective action is required. If instrument corrective action is not applicable or ineffective, re-extraction of the associated samples is required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated samples. Project-specific requirements may require all compounds to be treated as control analytes*, or dictate use of project acceptance criteria. In cases where the LCS is extracted in duplicate with the batch, the batch is considered acceptable as long as one LCS passes criteria. One LCS or the duplicate or both shall be reported with the batch. Do not report a combination of the two LCSs as a single LCS.

12.3.5. If the matrix spike recovery fails acceptance, the sample must be evaluated for matrix interferences requirements. Evaluate the recovery of the duplicate MS and/ or batch LCS. If the LCS is acceptable, continue with the analysis, assume matrix interferences, and note matrix interference in the case narrative. Re-extraction or reanalysis is not required solely on performance of MS or MSD.

12.3.6. RPD – If the RPD of the between MS/MSD is out of limits, repeat unless there is assignable matrix interference, historical failures, or lack of volume. If the pair is not repeated, note the reason on the data quality checklist. Examples may include product layers on aqueous samples that may result in non-homogenous subsampling, non-homogenous soil samples, chromatographic interferences resulting in poor peak resolution and inconsistent integrations, or poor purging efficiencies (indicated by surrogate recovery). The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately.

12.3.7. If a duplicate LCS was run with the batch and one LCS was slightly out, but the other LCS was in, the data is accepted.

*See Appendix B for DOD Specific Criteria

12.3.8. Internal standards must have RT ± 0.5 min from the ICAL for the CCV and ± 0.5 from the CCV for the samples. Any outliers for the ICAL or CCV requires a new ICAL. CCV internal standard area counts must be -50% to 100% of the initial calibration mid-point standard area counts, otherwise, a new curve is required.

12.3.9. Sample internal standard area counts must be -50% to 100% of the CCV area counts. Any internal standard outlier should be reanalyzed unless a matrix interference can be clearly demonstrated by the first analysis. Associated results from each outlying Internal Standard shall be flagged as estimated.

13. DATA REDUCTION AND REPORTING

13.1. Sample concentrations are reported in µg/L for waters and µg/kg for soils and other non-aqueous samples.

13.2. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions. The primary and secondary quantitation ions are given in Table 3. The internal standard with which the analytes are associated changes with each column and is documented in the Initial Calibration Summary Report.

13.3. Calculations: Once an analyte has been identified, the quantitation of that analyte is calculated as follows, depending upon matrix. See Table 4 for list of internal standards used for quantitation and their associated analytes.

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*See Appendix B for DOD Specific Criteria

13.4. Internal standard calibration

13.4.1. Aqueous samples

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

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 concentrated sample extract in mg/L.
D = Dilution factor, if the sample or extract 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. *V_i is not used in the equation. V_i is not needed when a mass/volume concentration (mg/L) is used for C_{is}. V_i is needed if only the mass (ng) is used for C_{is}.
$\overline{RF}$ = Mean response factor from the initial calibration. Unlike calibration factors for external standard calibration, the response factor is dimensionless.
V_s = Volume of the aqueous sample extracted or purged (mL). If units of liters are used for this term, multiply the results by 1000. Assume 1000 mL. Adjust D if sample volume is different from 1000 mL.
V_t = Volume of the concentrated extract (uL)

Using the units specified here for these terms will result in a concentration in units of mg/L ÷ 1000, which is equivalent to µg/L.

13.4.2. Soil and Nonaqueous samples

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

Where:
A_s, A_{is}, C_{is}, D, V_i, V_t, 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 specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The instrument results (mg/L) are multiplied by 33.3 which is 1000uL/30g. This results in units of ug/kg. Adjust D for changes in actual W_s or V_t.

*See Appendix B for DOD Specific Criteria

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13.5. Linear Regression

If a linear calibration that does not pass through the origin has been employed, then the regression equation is rearranged and the concentration of the analyte is calculated from the area response (y), the slope (a), and the intercept (b). When using this form of linear calibration, it is the laboratory's responsibility to ensure that the calculations take into account the volume or weight of the original sample, the dilution factor (if any), and dry weight (as applicable). One approach to this calculation is to perform the original linear regression using the concentration of the analyte in the final extract volume or the volume purged. The concentration of the analyte in the sample may then be calculated as follows:

13.5.1. Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{(C_{ex})(D)(V_t)}{(V_s)}$$

where:
C = Concentration in the final extract ex (mg/L)
D = Dilution factor
V_t = Volume of the concentrated extract (uL)
V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

13.5.2. Non-Aqueous samples

For solid samples, substitute the weight of the sample, W , for Vs.

13.6. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with a footnote.

13.7. Data reporting and review is handled by a computerized LIMS System. Specifics pertaining to reporting and review are discussed in ADM-DREV.

14. METHOD PERFORMANCE

Reporting limits are based upon an MDL study performed according to ADM-MDL and filed in the MDL binders in the QA office.

Demonstration of Capability is performed upon instrument set-up, whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office.

Accuracy and Precision Data is be available in the referenced method.

*See Appendix B for DOD Specific Criteria

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15. WASTE MANAGEMENT AND POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

15.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

15.2. Excess, unused sample and testing byproducts are disposed following the procedures in the *SOP for Waste Disposal.*

16. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18. REFERENCES

Test methods for Evaluating Solid Waste Physical/ Chemical Methods," SW-846 revision 3, Jan. 1995.

*See Appendix B for DOD Specific Criteria

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19. TRAINING OUTLINE

19.1. Read current SOP and applicable methodologies. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2. Observe Sample Preparation and Analysis. Follow SVOC GC/MS Training Plan Form.

19.3. Participate in the methodology, documentation, and data reduction with guidance.

19.4. Perform an IDC (Initial Demonstration of Competency) by independently analyzing four LCSs prior to analyzing client samples. If recovery is within acceptable limits, complete Training Plan Form, summary spreadsheet, and IDC Certificate. File with QA. Continuing proficiency (CDC) will be demonstrated annually using a PE, a single blind, or a new 4 replicate study.

20. METHOD MODIFICATIONS

None

21. INSTRUMENT SPECIFIC ADDENDUM

None, see instrument manuals located in the instrument lab.

22. ATTACHMENTS

Table 1 Typical Targets and Associated Reporting Limits
Table 2 DFTPP Key Ions and Abundance Criteria
Table 3 Characteristic Ions for SemiVolatile Compounds
Table 4 SemiVolatile Internal Standards with Corresponding Analytes Assigned for Quantitation
Appendix A – 8270C LVI Instructions and Standard Preparation
Appendix B - DOD Summary and DOD Limits Tables
Appendix C – Instrument Operating Conditions

*See Appendix B for DOD Specific Criteria

23. CHANGES FROM LATEST REVISION

- Added Low Level analysis discussion using LVI technology throughout (title page, scope, method summary (injection volume), apparatus (equipment and column information), standards (recipes referenced), and Appendix A (list analyte list, MRLs, and standards preparation information).
- Rearranged attached tables to place analyte list as Table 1.
- Bullet listed problem performing compounds discussed in Section 2.2.
- Added definitions of Analysis Window, %RSD, Internal Standards, MDL, MRL, and Target Analyte.
- Modified Safety and Waste Management for consistency with other SOPs.
- Expanded Section 6 to include minimum volume/mass requirements and non-aqueous liquid requirements.
- Added reference to Appendix A of QAM for instrument details.
- Expanded upon Maintenance Log – also added Troubleshooting.
- Updated column reference in Section 7 to Phenomenex.
- Changed the column pruning section in 8 so that the column end should extent 6 mm past the ferrule instead of 27 mm.
- Reformated Section 8.1 to list out the indicators vs. paragraph format.
- Corrected the number of calibration standards from 3 to 5 in Section 9 Section 11 was correct at 5 standards.
- Added Standards Prep General Info and Disclaimers to 9
- Added need to check for DOC and MDL in 11
- Expanded Tuning Criteria in Section 11 to include apex scan as an acceptable scan to use for tuning, DDT breakdown evaluation and calculation, and monitoring benzidine and pentachlorophenol.
- Expanded upon the discussion of linear regression in the Initial Calibration, Section 11.
- Added reference to ADM-INT to section 11.
- Allowed flexibility with the ICV criteria in Section 11 to allow some occasional outliers.
- Added diphenylamine and phenol to CCCs, Section 11.
- Differentiated between %Drift and %Difference and revised criteria to 20%; 25%, and upto 4 outliers not to exceed 40% in Daily Calibration Section 11.
- Added new table reference in Section 11 for Method recommended characteristic ions for identification.
- Added CCV data acceptance exceptions in 12
- Expanded 12 to reference Appendix C of the QAM for surrogates, LCS, MS, and duplicate QC limits.
- Added corrective action details for outlying recoveries of surrogates Matrix Spikes and LCSs in Section 12.
- Added the allowance of phthalate contamination in MB up to 5 times the MRL.
- Added discussion regarding software in 13
- Expanded sample calculations to include calculations using internal standards and/or linear regression, Section 13.
- Added paragraph to Method Performance to reference the Demonstration of Capability in Section 19 and ADM-TRANDOC.
- Added DOD requirements

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*See Appendix B for DOD Specific Criteria

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Table 1
8270C Typical Target Compounds and Reporting Limits

Appendix IX Compounds	Water MRL (ug/L)	Soil MRL (ug/Kg)
1,2,4,5-TETRACHLOROBENZENE	10	330
1,2,4-TRICHLOROBENZENE	10	330
1,2-DICHLOROBENZENE	10	330
1,3,5-TRINITROBENZENE	10	330
1,3-DICHLOROBENZENE	10	330
1,3-DINITROBENZENE	10	330
1,4-DICHLOROBENZENE	10	330
1,4-NAPHTHOQUINONE	50	1700
1-NAPHTHYLAMINE	10	330
2,2'-OXYBIS(1-CHLOROPROPANE)	10	330
2,3,4,6-TETRACHLOROPHENOL	10	330
2,4,5-TRICHLOROPHENOL	10	330
2,4,6-TRICHLOROPHENOL	10	330
2,4-DICHLOROPHENOL	10	330
2,4-DIMETHYLPHENOL	10	330
2,4-DINITROPHENOL	50	1700
2,4-DINITROTOLUENE	10	330
2,6-DICHLOROPHENOL	10	330
2,6-DINITROTOLUENE	10	330
2-ACETYLAMINOFLUORENE	10	330
2-CHLORONAPHTHALENE	10	330
2-CHLOROPHENOL	10	330
2-METHYLNAPHTHALENE	10	330
2-METHYLPHENOL	10	330
2-NAPHTHYLAMINE	50	1700
2-NITROANILINE	50	1700
2-NITROPHENOL	10	330
2-PICOLINE (2-METHYLPRYIDINE)	10	330
3,3'-DIMETHYLBENZIDINE	50	1700
3,3'-DICHLOROBENZIDINE	10	330
3+4-METHYLPHENOL (m+p-CRESOL)	10	330
3-METHANCHOLANTHRENE	10	330
3-NITROANILINE	50	1700
4,6-DINITRO-2-METHYLPHENOL	50	1700
4-AMINOBIPHENYL	50	1700
4-BROMOPHENYL-PHENYLETHER	10	330
4-CHLORO-3-METHYLPHENOL	10	330
4-CHLOROANILINE	10	330
4-CHLOROPHENYL-PHENYLETHER	10	330
4-DIMETHYLAMINOAZOBENZENE	10	330
4-METHYLPHENOL	10	330
4-NITROANILINE	50	1700

*See Appendix B for DOD Specific Criteria

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Table 1 (Continued)		
Appendix IX Compounds	**Water MRL (ug/L)**	**Soil MRL (ug/Kg)**
4-NITROPHENOL	50	1700
5-NITRO-O-TOLUIDINE	10	330
7,12-DIMETHYLBENZ(a)ANTHRACENE	10	330
a,a-DIMETHYLPHENETHYLAMINE	50	1700
ACENAPHTHENE	10	330
ACENAPHTHYLENE	10	330
ACETOPHENONE	10	330
ANTHRACENE	10	330
ARAMITE	50	1700
BENZO(A)ANTHRACENE	10	330
BENZO(A)PYRENE	10	330
BENZO(B)FLUORANTHENE	10	330
BENZO(G,H,I)PERYLENE	10	330
BENZO(K)FLUORANTHENE	10	330
BIS(-2-CHLOROETHOXY)METHANE	10	330
BIS(2-CHLOROETHYL)ETHER	10	330
BIS(2-ETHYLHEXYL)PHTHALATE	10	330
BUTYL BENZYL PHTHALATE	10	330
CARBAZOLE	10	330
CHLOROBENZILATE	10	330
CHRYSENE	10	330
DIALLATE	10	330
DIBENZO(A,H)ANTHRACENE	10	330
DIBENZOFURAN	10	330
DIETHYLPHTHALATE	10	330
DIMETHOATE	50	1700
DIMETHYL PHTHALATE	10	330
DI-N-BUTYLPHTHALATE	10	330
DI-N-OCTYL PHTHALATE	10	330
DINOSEB	50	1700
DIPHENYLAMINE	10	330
DISULFOTON	10	330
ETHYL METHANESULFONATE	10	330
ETHYL PARATHION	10	330
FLUORANTHENE	10	330
FLUORENE	10	330
HEXACHLOROBENZENE	10	330
HEXACHLOROBUTADIENE	10	330
HEXACHLOROCYCLOPENTADIENE	10	330
HEXACHLOROETHANE	10	330
HEXACHLOROPHENE	-	-
HEXACHLOROPROPENE	10	330
INDENO(1,2,3-CD)PYRENE	10	330
ISODRIN	10	330
ISOPHORONE	10	330
ISOSAFROLE	10	330

*See Appendix B for DOD Specific Criteria

Table 1 (Continued)		
Appendix IX Compounds	**Water MRL (ug/L)**	**Soil MRL (ug/Kg)**
METHAPYRILENE	50	1700
METHYL METHANESULFONATE	10	330
METHYL PARATHION	10	330
NAPHTHALENE	10	330
NITROBENZENE	10	330
NITROQUINOLINE-1-OXIDE	50	1700
n-NITROSODIETHYLAMINE	10	330
n-NITROSO-DI-N-BUTYLAMINE	10	330
N-NITROSO-DI-N-PROPYLAMINE	10	330
N-NITROSODIPHENYLAMINE	10	330
n-NITROSOMETHALETHYLAMINE	10	330
n-NITROSOMORPHOLINE	10	330
n-NITROSOPIPERIDINE	10	330
n-NITROSOPYRROLIDINE	10	330
ooo-TRIETHYL PHOSPHOROTHIOATE	10	330
o-TOLUIDINE	10	330
PARATHION	10	330
PENTACHLOROBENZENE	10	330
PENTACHLOROETHANE	10	330
PENTACHLORONITROBENZENE	10	330
PENTACHLOROPHENOL	50	1700
PHENACETIN	10	330
PHENANTHRENE	10	330
PHENOL	10	330
PHORATE	10	330
p-PHENYLENEDIAMINE	50	1700
PRONAMIDE (KERB)	10	330
PYRENE	10	330
SAFROLE	10	330
SULFOTEPP	10	330
THIONAZIN (ZINOPHOS)	10	330

Other Compounds by Request		
1,1'-BIPHENYL	10	330
1,2-DIPHENYLHYDRAZINE	10	330
ANILINE	10	330
ATRAZINE	10	330
BENZYL ALCOHOL	10	330
BENZIDINE	100	3300
BENZOIC ACID	50	1700
CAPROLACTAM	50	1700
N-NITROSODIMETHYLAMINE	10	330
PYRIDINE	50	1700

*See Appendix B for DOD Specific Criteria

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TABLE 2
DFTPP KEY IONS AND ION ABUNDANCE 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 abundance
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

*See Appendix B for DOD Specific Criteria

TABLE 3
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
2-Picoline	3.75[a]	93	66,92
Aniline	5.68	93	66,65
Phenol	5.77	94	65,66
Bis(2-chloroethyl) ether	5.82	93	63,95
2-Chlorophenol	5.97	128	64,130
1,3-Dichlorobenzene	6.27	146	148,111
1,4-Dichlorobenzene-d_4 (IS)	6.35	152	150,115
1,4-Dichlorobenzene	6.40	146	148,111
Benzyl alcohol	6.78	108	79,77
1,2-Dichlorobenzene	6.85	146	148,111
N-Nitrosomethylethylamine	6.97	88	42,43,56
Bis(2-chloroisopropyl) ether	7.22	45	77,121
Ethyl carbamate	7.27	62	44,45,74
Thiophenol (Benzenethiol)	7.42	110	66,109,84
Methyl methanesulfonate	7.48	80	79,65,95
N-Nitrosodi-n-propylamine	7.55	70	42,101,130
Hexachloroethane	7.65	117	201,199
Maleic anhydride	7.65	54	98,53,44
Nitrobenzene	7.87	77	123,65
Isophorone	8.53	82	95,138
N-Nitrosodiethylamine	8.70	102	42,57,44,56
2-Nitrophenol	8.75	139	109,65
2,4-Dimethylphenol	9.03	122	107,121
p-Benzoquinone	9.13	108	54,82,80
Bis(2-chloroethoxy)methane	9.23	93	95,123
Benzoic acid	9.38	122	105,77
2,4-Dichlorophenol	9.48	162	164,98
Trimethyl phosphate	9.53	110	79,95,109,140
Ethyl methanesulfonate	9.62	79	109,97,45,65
1,2,4-Trichlorobenzene	9.67	180	182,145
Naphthalene-d_8 (IS)	9.75	136	68
Naphthalene	9.82	128	129,127
Hexachlorobutadiene	10.43	225	223,227
Tetraethyl pyrophosphate	11.07	99	155,127,81,109
Diethyl sulfate	11.37	139	45,59,99,111,125
4-Chloro-3-methylphenol	11.68	107	144,142
2-Methylnaphthalene	11.87	142	141
2-Methylphenol	12.40	107	108,77,79,90
Hexachloropropene	12.45	213	211,215,117,106,141
Hexachlorocyclopentadiene	12.60	237	235,272

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
N-Nitrosopyrrolidine	12.65	100	41,42,68,69
Acetophenone	12.67	105	71,51,120
4-Methylphenol	12.82	107	108,77,79,90
2,4,6-Trichlorophenol	12.85	196	198,200
o-Toluidine	12.87	106	107,77,51,79
3-Methylphenol	12.93	107	108,77,79,90
2-Chloronaphthalene	13.30	162	127,164
N-Nitrosopiperidine	13.55	114	42,55,56,41
1,4-Phenylenediamine	13.62	108	80,53,54,52
1-Chloronaphthalene	13.65[a]	162	127,164
2-Nitroaniline	13.75	65	92,138
5-Chloro-2-methylaniline	14.28	106	141,140,77,89
Dimethyl phthalate	14.48	163	194,164
Acenaphthylene	14.57	152	151,153
2,6-Dinitrotoluene	14.62	165	63,89
Phthalic anhydride	14.62	104	76,50,148
o-Anisidine	15.00	108	80,123,52
3-Nitroaniline	15.02	138	108,92
Acenaphthene-d_{10} (IS)	15.05	164	162,160
Acenaphthene	15.13	154	153,152
2,4-Dinitrophenol	15.35	184	63,154
2,6-Dinitrophenol	15.47	162	164,126,98,63
4-Chloroaniline	15.50	127	129,65,92
Isosafrole	15.60	162	131,104,77,51
Dibenzofuran	15.63	168	139
2,4-Diaminotoluene	15.78	121	122,94,77,104
2,4-Dinitrotoluene	15.80	165	63,89
4-Nitrophenol	15.80	139	109,65
2-Naphthylamine	16.00[a]	143	115,116
1,4-Naphthoquinone	16.23	158	104,102,76,50,130
p-Cresidine	16.45	122	94,137,77,93
Dichlorovos	16.48	109	185,79,145
Diethyl phthalate	16.70	149	177,150
Fluorene	16.70	166	165,167
2,4,5-Trimethylaniline	16.70	120	135,134,91,77
N-Nitrosodi-n-butylamine	16.73	84	57,41,116,158
4-Chlorophenyl phenyl ether	16.78	204	206,141
Hydroquinone	16.93	110	81,53,55
4,6-Dinitro-2-methylphenol	17.05	198	51,105
Resorcinol	17.13	110	81,82,53,69
N-Nitrosodiphenylamine	17.17	169	168,167
Safrole	17.23	162	104,77,103,135

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Hexamethyl phosphoramide	17.33	135	44,179,92,42
3-(Chloromethyl)pyridine hydrochloride	17.50	92	127,129,65,39
Diphenylamine	17.54[a]	169	168,167
1,2,4,5-Tetrachlorobenzene	17.97	216	214,179,108,143,218
1-Naphthylamine	18.20	143	115,89,63
1-Acetyl-2-thiourea	18.22	118	43,42,76
4-Bromophenyl phenyl ether	18.27	248	250,141
Toluene diisocyanate	18.42	174	145,173,146,132,91
2,4,5-Trichlorophenol	18.47	196	198,97,132,99
Hexachlorobenzene	18.65	284	142,249
Nicotine	18.70	84	133,161,162
Pentachlorophenol	19.25	266	264,268
5-Nitro-o-toluidine	19.27	152	77,79,106,94
Thionazine	19.35	107	96,97,143,79,68
4-Nitroaniline	19.37	138	65,108,92,80,39
Phenanthrene-d_{10} (IS)	19.55	188	94,80
Phenanthrene	19.62	178	179,176
Anthracene	19.77	178	176,179
1,4-Dinitrobenzene	19.83	168	75,50,76,92,122
Mevinphos	19.90	127	192,109,67,164
Naled	20.03	109	145,147,301,79,189
1,3-Dinitrobenzene	20.18	168	76,50,75,92,122
Diallate (cis or trans)	20.57	86	234,43,70
1,2-Dinitrobenzene	20.58	168	50,63,74
Diallate (trans or cis)	20.78	86	234,43,70
Pentachlorobenzene	21.35	250	252,108,248,215,254
5-Nitro-o-anisidine	21.50	168	79,52,138,153,77
Pentachloronitrobenzene	21.72	237	142,214,249,295,265
4-Nitroquinoline-1-oxide	21.73	174	101,128,75,116
Di-n-butyl phthalate	21.78	149	150,104
2,3,4,6-Tetrachlorophenol	21.88	232	131,230,166,234,168
Dihydrosaffrole	22.42	135	64,77
Demeton-O	22.72	88	89,60,61,115,171
Fluoranthene	23.33	202	101,203
1,3,5-Trinitrobenzene	23.68	75	74,213,120,91,63
Dicrotophos	23.82	127	67,72,109,193,237
Benzidine	23.87	184	92,185
Trifluralin	23.88	306	43,264,41,290
Bromoxynil	23.90	277	279,88,275,168
Pyrene	24.02	202	200,203
Monocrotophos	24.08	127	192,67,97,109
Phorate	24.10	75	121,97,93,260

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Sulfallate	24.23	188	88,72,60,44
Demeton-S	24.30	88	60,81,89,114,115
Phenacetin	24.33	108	180,179,109,137,80
Dimethoate	24.70	87	93,125,143,229
Phenobarbital	24.70	204	117,232,146,161
Carbofuran	24.90	164	149,131,122
Octamethyl pyrophosphoramide	24.95	135	44,199,286,153,243
4-Aminobiphenyl	25.08	169	168,170,115
Dioxathion	25.25	97	125,270,153
Terbufos	25.35	231	57,97,153,103
α,α-Dimethylphenylamine	25.43	58	91,65,134,42
Pronamide	25.48	173	175,145,109,147
Aminoazobenzene	25.72	197	92,120,65,77
Dichlone	25.77	191	163,226,228,135,193
Dinoseb	25.83	211	163,147,117,240
Disulfoton	25.83	88	97,89,142,186
Fluchloralin	25.88	306	63,326,328,264,65
Mexacarbate	26.02	165	150,134,164,222
4,4'-Oxydianiline	26.08	200	108,171,80,65
Butyl benzyl phthalate	26.43	149	91,206
4-Nitrobiphenyl	26.55	199	152,141,169,151
Phosphamidon	26.85	127	264,72,109,138
2-Cyclohexyl-4,6-Dinitrophenol	26.87	231	185,41,193,266
Methyl parathion	27.03	109	125,263,79,93
Carbaryl	27.17	144	115,116,201
Dimethylaminoazobenzene	27.50	225	120,77,105,148,42
Propylthiouracil	27.68	170	142,114,83
Benz(a)anthracene	27.83	228	229,226
Chrysene-d_{12} (IS)	27.88	240	120,236
3,3'-Dichlorobenzidine	27.88	252	254,126
Chrysene	27.97	228	226,229
Malathion	28.08	173	125,127,93,158
Kepone	28.18	272	274,237,178,143,270
Fenthion	28.37	278	125,109,169,153
Parathion	28.40	109	97,291,139,155
Anilazine	28.47	239	241,143,178,89
Bis(2-ethylhexyl) phthalate	28.47	149	167,279
3,3'-Dimethylbenzidine	28.55	212	106,196,180
Carbophenothion	28.58	157	97,121,342,159,199
5-Nitroacenaphthene	28.73	199	152,169,141,115
Methapyrilene	28.77	97	50,191,71
Isodrin	28.95	193	66,195,263,265,147

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Captan	29.47	79	149,77,119,117
Chlorfenvinphos	29.53	267	269,323,325,295
Crotoxyphos	29.73	127	105,193,166
Phosmet	30.03	160	77,93,317,76
EPN	30.11	157	169,185,141,323
Tetrachlorvinphos	30.27	329	109,331,79,333
Di-n-octyl phthalate	30.48	149	167,43
2-Aminoanthraquinone	30.63	223	167,195
Barban	30.83	222	51,87,224,257,153
Aramite	30.92	185	191,319,334,197,321
Benzo(b)fluoranthene	31.45	252	253,125
Nitrofen	31.48	283	285,202,139,253
Benzo(k)fluoranthene	31.55	252	253,125
Chlorobenzilate	31.77	251	139,253,111,141
Fensulfothion	31.87	293	97,308,125,292
Ethion	32.08	231	97,153,125,121
Diethylstilbestrol	32.15	268	145,107,239,121,159
Famphur	32.67	218	125,93,109,217
Tri-p-tolyl phosphate[b]	32.75	368	367,107,165,198
Benzo(a)pyrene	32.80	252	253,125
Perylene-d_{12} (IS)	33.05	264	260,265
7,12-Dimethylbenz(a)anthracene	33.25	256	241,239,120
5,5-Diphenylhydantoin	33.40	180	104,252,223,209
Captafol	33.47	79	77,80,107
Dinocap	33.47	69	41,39
Methoxychlor	33.55	227	228,152,114,274,212
2-Acetylaminofluorene	33.58	181	180,223,152
4,4'-Methylenebis(2-chloroaniline)	34.38	231	266,268,140,195
3,3'-Dimethoxybenzidine	34.47	244	201,229
3-Methylcholanthrene	35.07	268	252,253,126,134,113
Phosalone	35.23	182	184,367,121,379
Azinphos-methyl	35.25	160	132,93,104,105
Leptophos	35.28	171	377,375,77,155,379
Mirex	35.43	272	237,274,270,239,235
Tris(2,3-dibromopropyl) phosphate	35.68	201	137,119,217,219,199
Dibenz(a,j)acridine	36.40	279	280,277,250
Mestranol	36.48	277	310,174,147,242
Coumaphos	37.08	362	226,210,364,97,109
Indeno(1,2,3-cd)pyrene	39.52	276	138,227
Dibenz(a,h)anthracene	39.82	278	139,279
Benzo(g,h,i)perylene	41.43	276	138,277
1,2:4,5-Dibenzopyrene	41.60	302	151,150,300

*See Appendix B for DOD Specific Criteria

TABLE 3 (cont.)
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Retention Time (min)	Primary Ion	Secondary Ion(s)
Strychnine	45.15	334	334,335,333
Piperonyl sulfoxide	46.43	162	135,105,77
Hexachlorophene	47.98	196	198,209,211,406,408
Aldrin	--	66	263,220
Aroclor 1016	--	222	260,292
Aroclor 1221	--	190	224,260
Aroclor 1232	--	190	224,260
Aroclor 1242	--	222	256,292
Aroclor 1248	--	292	362,326
Aroclor 1254	--	292	362,326
Aroclor 1260	--	360	362,394
α-BHC	--	183	181,109
β-BHC	--	181	183,109
δ-BHC	--	183	181,109
γ-BHC (Lindane)	--	183	181,109
4,4'-DDD	--	235	237,165
4,4'-DDE	--	246	248,176
4,4'-DDT	--	235	237,165
Dieldrin	--	79	263,279
1,2-Diphenylhydrazine	--	77	105,182
Endosulfan I	--	195	339,341
Endosulfan II	--	337	339,341
Endosulfan sulfate	--	272	387,422
Endrin	--	263	82,81
Endrin aldehyde	--	67	345,250
Endrin ketone	--	317	67,319
2-Fluorobiphenyl (surr)	--	172	171
2-Fluorophenol (surr)	--	112	64
Heptachlor	--	100	272,274
Heptachlor epoxide	--	353	355,351
Nitrobenzene-d_5 (surr)	--	82	128,54
N-Nitrosodimethylamine	--	42	74,44
Phenol-d_6 (surr)	--	99	42,71
Terphenyl-d_{14} (surr)	--	244	122,212
2,4,6-Tribromophenol (surr)	--	330	332,141
Toxaphene	--	159	231,233

IS = internal standard
surr = surrogate
[a]Estimated retention times
[b]Substitute for the non-specific mixture, tricresyl phosphate

*See Appendix B for DOD Specific Criteria

Table 4
SemiVolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d_4	Naphthalene-d_8	Acenaphthene-d_{10}
Aniline	Acetophenone	Acenaphthene
Benzyl alcohol	Benzoic acid	Acenaphthylene
Bis(2-chloroethyl) ether	Bis(2-chloroethoxy)methane	1-Chloronaphthalene
Bis(2-chloroisopropyl) ether	4-Chloroaniline	2-Chloronaphthalene
2-Chlorophenol	4-Chloro-3-methylphenol	4-Chlorophenyl phenyl ether
1,3-Dichlorobenzene	2,4-Dichlorophenol	Dibenzofuran
1,4-Dichlorobenzene	2,6-Dichlorophenol	Diethyl phthalate
1,2-Dichlorobenzene	α,α-Dimethyl-phenethylamine	Dimethyl phthalate
Ethyl methanesulfonate	2,4-Dimethylphenol	2,4-Dinitrophenol
2-Fluorophenol (surr)	Hexachlorobutadiene	2,4-Dinitrotoluene
Hexachloroethane	Isophorone	2,6-Dinitrotoluene
Methyl methanesulfonate	2-Methylnaphthalene	Fluorene
2-Methylphenol	Naphthalene	2-Fluorobiphenyl (surr)
4-Methylphenol	Nitrobenzene	Hexachlorocyclo-pentadiene
N-Nitrosodimethylamine	Nitrobenzene-d_8 (surr)	1-Naphthylamine
N-Nitroso-di-n-propyl-amine	2-Nitrophenol	2-Naphthylamine
Phenol	N-Nitrosodi-n-butylamine	2-Nitroaniline
Phenol-d_6 (surr)	N-Nitrosopiperidine	3-Nitroaniline
2-Picoline	1,2,4-Trichlorobenzene	4-Nitroaniline
		4-Nitrophenol
		Pentachlorobenzene
		1,2,4,5-Tetra-chlorobenzene
		2,3,4,6-Tetra-chlorophenol
		2,4,6-Tribromo-phenol (surr)
		2,4,6-Trichloro-phenol
		2,4,5-Trichloro-phenol

(surr) = surrogate

*See Appendix B for DOD Specific Criteria

Table 4 (cont.)
SemiVolatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Phenanthrene-d_{10}	Chrysene-d_{12}	Perylene-d_{12}
4-Aminobiphenyl	Benzidine	Benzo(b)fluor-anthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluor-anthene
4-Bromophenyl phenyl ether	Bis(2-ethylhexyl) phthalate	Benzo(g,h,i)-perylene
Di-n-butyl phthalate	Butyl benzyl phthalate	Benzo(a)pyrene
4,6-Dinitro-2-methyl-phenol	Chrysene	Dibenz(a,j)acridine
Diphenylamine	3,3'-Dichlorobenzidine	Dibenz(a,h)-anthracene
Fluoranthene	p-Dimethylaminoazobenzene	
Hexachlorobenzene	Pyrene	
N-Nitrosodiphenylamine	Terphenyl-d_{14} (surr)	
Pentachlorophenol	7,12-Dimethylbenz-(a)anthracene	
Pentachloronitrobenzene	Di-n-octyl phthalate	
Phenacetin	Indeno(1,2,3-cd) pyrene	
Phenanthrene	3-Methylchol-anthrene	
Pronamide		

(surr) = surrogate

*See Appendix B for DOD Specific Criteria

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APPENDIX A

8270C LARGE VOLUME INJECTOR (LVI) INSTRUCTIONS AND STANDARDS PREPARATION

*See Appendix B for DOD Specific Criteria

8270C LVI Instructions

See SOC-8270C SOP for analytical requirements, instrument conditions, and QC requirements. Lower reporting limits may be achieved with a GC/MS equipped with a Large Volume Injector (LVI) which allows a larger volume of extracted to be injected onto the LVI thus loading the column with more sample. The linear range is 0.1-6.0 ppm for Polynuclear Aromatic Compounds (PAHs) and 0.2-12.0 ppm for 1,4-Dioxane. Reporting limits and Standard Preparation Instructions are noted below:

METHOD		ANALYTE	MATRIX	MRL ug/L	MRL ug/Kg
8270C LVI		ACENAPHTHENE	WATER	0.20	6.6
8270C LVI		ACENAPHTHYLENE	WATER	0.20	6.6
8270C LVI		ANTHRACENE	WATER	0.20	6.6
8270C LVI		BENZO(A)ANTHRACENE	WATER	0.10	3.3
8270C LVI		BENZO(A)PYRENE	WATER	0.20	6.6
8270C LVI		BENZO(B)FLUORANTHENE	WATER	0.20	6.6
8270C LVI		BENZO(G,H,I)PERYLENE	WATER	0.20	6.6
8270C LVI		BENZO(K)FLUORANTHENE	WATER	0.20	6.6
8270C LVI		CHRYSENE	WATER	0.20	6.6
8270C LVI		DIBENZO(A,H)ANTHRACENE	WATER	0.20	6.6
8270C LVI		FLUORANTHENE	WATER	0.20	6.6
8270C LVI		FLUORENE	WATER	0.20	6.6
8270C LVI		INDENO(1,2,3-CD)PYRENE	WATER	0.20	6.6
8270C LVI		NAPHTHALENE	WATER	0.20	6.6
8270C LVI		PHENANTHRENE	WATER	0.20	6.6
8270C LVI		PYRENE	WATER	0.20	6.6
8270C LVI		2-FLUOROBIPHENYL -SURR	WATER	NA	NA
8270C LVI		NITROBENZENE-d5 -SURR	WATER	NA	NA
8270C LVI		TERPHENYL-d14 -SURR	WATER	NA	NA
8270C LVI ADDITIONAL COMPOUNDS BY REQUEST					
8270C LVI		1,4-DIOXANE	WATER	2.0	66.6
8270C LVI		1-METHYLNAPHTHALENE	WATER	0.20	6.6
8270C LVI		2-METHYLNAPHTHALENE	WATER	0.10	3.3
8270C LVI		BIS(2-ETHYLHEXYL)PHTHALATE	WATER	2.0	66.6
8270C LVI		CARBAZOLE	WATER	1.0	33.3
8270C LVI		DIBENZOFURAN	WATER	0.20	6.6
8270C LVI		HEXACHLOROBENZENE	WATER	0.20	6.6
8270C LVI		NITROBENZENE	WATER	0.20	6.6

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*See Appendix B for DOD Specific Criteria

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8270 LVI Standards Preparation Summary

These receipes may change to accommodate the availability of standards and compound lists/reporting limits requested.

50/100 ppm Stock Solution (50 ppm for PAH, and 100 ppm for Dioxane, BEHP)

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
PAH NSI Ready Stock	1.25	200	5.0	50
1-Methylnaphthalene	0.05	5000	5.0	50
1,4-Dioxane	0.10	5000	5.0	100
Bis (2-ethylhexyl)phthalate	0.25	1000	5.0	100*

*since BEHP is already in NSI Ready Stock (50 ppm)

10/20 ppm Mid Stock Solution (10 ppm for PAH and 20 ppm for Dioxane, BEHP)

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
50/100 Stock Soln.	1.00	50/100	5.0	10 PAH/ 20 Dioxane, BEHP

8270 LVI Calibration Standards

Compound(s) Mix	Volume µL	Concentration ppm	Final Volume diluted with MeCl (µL)	Final Conc. ppm
10/20 Mid Stock Soln.	10	10/20	1000	0.1 PAH/ 0.2 Dioxane, BEHP
10/20 Mid Stock Soln.	20	10/20	1000	0.2 PAH/ 0.4 Dioxane, BEHP
10/20 Mid Stock Soln.	50	10/20	1000	0.5 PAH/ 1.0 Dioxane, BEHP
10/20 Mid Stock Soln.	100	10/20	1000	1.0 PAH/ 2.0 Dioxane, BEHP
10/20 Mid Stock Soln.	200	10/20	1000	2.0 PAH/ 4.0 Dioxane, BEHP
10/20 Mid Stock Soln.	400	10/20	1000	4.0 PAH/ 8.0 Dioxane, BEHP
10/20 Mid Stock Soln.	600	10/20	1000	6.0 PAH/ 12.0 Dioxane, BEHP

All standards require a 10 µL spike of 100 ppm Internal Standard Spiking Solution (see below)

8270 LVI LCS/MS Spike Solution

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
Puchased 8270 LCS Mix	0.50	100	100.0	0.5
1,4-Dioxane, purchased	0.10	5000	100.0	5.0
Bis (2-ethylhexyl)phthalate	0.45	1000	100.0	4.5

** Since BEHP is present in the 8270 LCS Mix (0.5), the actual final concentration for BEHP in this solution is 5.0 ppm

8270 LVI 100 ppm Internal Standard Spiking Solution

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
ISTD Mix	0.025	4000	1.0	100

*See Appendix B for DOD Specific Criteria

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8270 LVI Standards Preparation Summary

These receipes may change to accommodate the availability of standards and compound lists/reporting limits requested.

8270 LVI 10 ppm Initial Calibration Verification Mid Stock Solution

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
8270 Calibration Mid Stock	0.05	200	1.0	10

8270 LVI Initial Calibration Verification Standard

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
ICV Mid Stock	0.10	10	1.0	1

8270 LVI Surrogate Mix

Compound(s) Mix	Volume mls	Concentration ppm	Final Volume diluted with MeCl (mL)	Final Conc. ppm
8270 BN Surrogate Mix	0.02	100	1.0	2

*See Appendix B for DOD Specific Criteria

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APPENDIX B

DOD SPECIFIC CRITERIA

*See Appendix B for DOD Specific Criteria

*DOD SUMMARY

For work for the Department of Defense – the DOD Quality Systems Manual must be followed. The DOD Manual is based on the NELAC Standards with some additional requirements. The following are the requirements which are different or additional to routine analysis and must be followed for DOD work:

- Calibration Curve, Linear Regression: The correlation coefficient must be greater then or equal to 0.995.
- The Second Source Calibration Verification (ICV) must have a recovery of 75-125% of the true value.
- The Method Blank must not have any hits above ½ the reporting limit.
- ~~If using the Grand Mean, no individual analyte (except CCCs) may be above 25%D in the CCV. Apply J flag to all results associated with the analytical batch for all analytes between 20 and 25 %D. Identify in case narrative analytes with %D >20% and document the grand mean.~~ Grand Mean may not be used for DoD.
- Apply J flag to all hits between MDL and MRL.
- Reporting Limits – "the lowest standard of the calibration establishes the [reporting limit], but it must be greater than 3 times the [MDL]." DoD QSM, V3, Appendix B- Glossary.
- The limits for surrogates, LCS, and MS are different from Appendix C of the QAM. Follow the DOD limits given in the following tables. All DOD targets are "control analytes" for the LCS for DOD projects. All DOD targets are spiked and evaluated.

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*See Appendix B for DOD Specific Criteria

The lower control limit generated for alternative or modified methods must be greater than 10% to be considered acceptable.

D.6 Surrogates

The surrogate compounds for each method are added to all samples, standards, and blanks to assess the ability of the method to recover specific non-target analytes from a given matrix and to monitor sample-specific recovery. Control limits for these compounds were calculated in the same study as the other analytes on the target analyte lists. Below are the limits for some of the surrogates of Methods 8260, 8270, 8081, and 8082, based on 3 standard deviations around the mean (Table D-3). Control limits are not available for some surrogates that appear on the target analyte lists in Appendix DoD-C. Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. No ME limits are presented as marginal exceedances are not acceptable for surrogate spikes. Note: DoD prefers the use of those surrogates not identified as poor performing analytes in Table D-2 above.

TABLE D-3. SURROGATES

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
8260 Water:				
1,2-Dichloroethane-d4	95	8	70	120
4-Bromofluorobenzene	98	7	75	120
Dibromofluoromethane	100	5	85	115
Toluene-d8	102	6	85	120
8260 Solid:				
4-Bromofluorobenzene	101	6	86	120
Toluene-d8	100	5	85	115
8270 Water:				
2-Fluorobiphenyl	79	10	50	110
Terphenyl-d14	92	14	50	135
2,4,6-Tribromophenol	82	13	40	125
2-Fluorophenol	63	14	20	110
Nitrobenzene-d5	76	11	40	110
8270 Solid:				
2-Fluorobiphenyl	72	10	45	105
Terphenyl-d14	78	15	30	125
2,4,6-Tribromophenol	80	15	35	125
2-Fluorophenol	70	11	35	105
Phenol-d5/d6	71	10	40	100
Nitrobenzene-d5	69	10	35	100
8081 Water:				
Decachlorobiphenyl	83	17	30	135
TCMX	81	19	25	140
8081 Solid:				
Decachlorobiphenyl	94	13	55	130
TCMX	97	9	70	125
8082 Water:				
Decachlorobiphenyl	88	15	40	135
8082 Solid:				
Decachlorobiphenyl	91	11	60	125

*See Appendix B for DOD Specific Criteria

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz(a)anthracene	82.7	8.9	55	110	45	120
Benzo(a)pyrene	81.3	9.5	55	110	45	120
Benzo(b)fluoranthene	81.8	12.1	45	120	35	130
Benzo(g,h,i)perylene	80.5	14.1	40	125	25	135
Benzo(k)fluoranthene	84.6	13.2	45	125	30	135
Chrysene	82.1	8.9	55	110	45	120
Dibenz(a,h)anthracene	84.7	14.1	40	125	30	140
Fluoranthene	85.2	10.4	55	115	45	125
Fluorene	80.6	10.3	50	110	40	120
Indeno(1,2,3-cd)pyrene	84.3	13.6	45	125	30	140
Naphthalene	70.8	10.5	40	100	30	115
Phenanthrene	84.0	11.0	50	115	40	130
Pyrene	88.6	13.2	50	130	35	140
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	50	110	40	120
2,4,6-Trichlorophenol	80.7	10.7	50	115	40	125
2,4-Dichlorophenol	76.3	9.6	50	105	40	115
2,4-Dimethylphenol	68.8	13.5	30	110	15	125
2,4-Dinitrophenol	75.8	20.6	15	140	10	160
2-Chlorophenol	71.3	11.4	35	105	25	115
2-Methylphenol	73.3	11.7	40	110	25	120
2-Nitrophenol	75.8	12.4	40	115	25	125
3-Methylphenol/4-Methylphenol	71.3	13.0	30	110	20	125
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145
4-Chloro-3-methylphenol	78.6	10.7	45	110	35	120
Pentachlorophenol	77.6	13.3	40	115	25	130
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	20	110	10	125
4-Chloroaniline	62.2	15.6	15	110	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	40	125	30	140
Butyl benzyl phthalate	81.1	11.7	45	115	35	130

[14] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

*See Appendix B for DOD Specific Criteria

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14] (CONTINUED)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Di-n-butyl phthalate	84.8	10.3	55	115	45	125
Di-n-octyl phthalate	87.4	16.6	35	135	20	155
Diethyl phthalate	79.2	12.9	40	120	30	130
Dimethyl phthalate	75.9	16.9	25	125	10	145
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	35	130	20	145
N-Nitrosodimethylamine	67.9	14.1	25	110	10	125
N-Nitrosodiphenylamine	79.6	10.6	50	110	35	120
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	76.2	10.2	45	105	35	115
Bis(2-chloroethyl) ether	73.3	12.3	35	110	25	120
Bis(2-chloroisopropyl) ether	78.2	17.5	25	130	10	150
Hexachlorobutadiene	65.2	12.6	25	105	15	115
Hexachloroethane	60.9	11.1	30	95	15	105
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	35	105	25	120
1,2-Dichlorobenzene	67.3	11.4	35	100	20	115
1,3-Dichlorobenzene	64.8	10.9	30	100	20	110
1,4-Dichlorobenzene	64.8	10.9	30	100	20	110
2-Chloronaphthalene	76.5	9.3	50	105	40	115
4-Bromophenyl phenyl ether	82.9	10.2	50	115	40	125
4-Chlorophenyl phenyl ether	80.6	10.3	50	110	40	120
Hexachlorobenzene	82.3	10.0	50	110	40	120
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	50	120	40	130
2,6-Dinitrotoluene	82.7	11.3	50	115	35	130
2-Nitroaniline	81.8	11.2	50	115	35	125
3-Nitroaniline	72.6	17.7	20	125	10	145
4-Nitroaniline	77.2	13.7	35	120	20	130
Nitrobenzene	76.8	10.8	45	110	35	120
Neutral Aromatis						
Carbazole	82.5	11.4	50	115	35	130
Dibenzofuran	80.3	8.8	55	105	45	115
Others						
1,2-Diphenylhydrazine	84.8	9.4	55	115	45	120
Benzyl alcohol	71.0	13.8	30	110	15	125
Isophorone	81.0	10.5	50	110	40	125

*See Appendix B for DOD Specific Criteria

TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	45	105	35	115
Acenaphthene	77.3	10.3	45	110	35	120
Acenaphthylene	75.7	10.4	45	105	35	115
Anthracene	79.9	9.0	55	105	45	115
Benz(a)anthracene	81.6	9.8	50	110	40	120
Benzo(a)pyrene	80.7	10.3	50	110	40	120
Benzo(b)fluoranthene	79.7	11.4	45	115	35	125
Benzo(g,h,i)perylene	81.8	14.7	40	125	25	140
Benzo(k)fluoranthene	83.8	12.9	45	125	30	135
Chrysene	82.6	9.9	55	110	45	120
Dibenz(a,h)anthracene	82.9	13.9	40	125	25	140
Fluoranthene	83.9	10.1	55	115	45	125
Fluorene	78.3	9.8	50	110	40	115
Indeno(1,2,3-cd)pyrene	79.7	13.8	40	120	25	135
Naphthalene	73.4	11.1	40	105	30	120
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	45	125	35	135
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	50	110	40	120
2,4,6-Trichlorophenol	76.3	11.0	45	110	30	120
2,4-Dichlorophenol	77.2	10.9	45	110	35	120
2,4-Dimethylphenol	67.3	11.9	30	105	20	115
2,4-Dinitrophenol	72.6	20.0	15	130	10	150
2-Chlorophenol	74.7	10.3	45	105	35	115
2-Methylphenol	71.7	10.6	40	105	30	115
2-Nitrophenol	76.2	11.5	40	110	30	120
3-Methylphenol/4-Methylphenol	73.9	10.9	40	105	30	120
4,6-Dinitro-2-methylphenol	83.1	18.0	30	135	10	155
4-Chloro-3-methylphenol	79.5	11.1	45	115	35	125
4-Nitrophenol	77.0	20.2	15	140	10	160
Pentachlorophenol	71.9	15.6	25	120	10	135
Phenol	69.7	10.2	40	100	30	110
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	87.4	13.3	45	125	35	140
Butyl benzyl phthalate	86.4	12.3	50	125	35	135
Di-n-butyl phthalate	83.2	9.1	55	110	45	120

[15] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spike in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

*See Appendix B for DOD Specific Criteria

TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15] (CONTINUED)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Di-n-octyl phthalate	86.4	15.2	40	130	25	145
Diethyl phthalate	82.2	10.6	50	115	40	125
Dimethyl phthalate	79.6	10.2	50	110	40	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	115	30	125
N-Nitrosodimethylamine	66.1	15.9	20	115	10	130
N-Nitrosodiphenylamine	82.4	11.1	50	115	40	125
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	75.5	10.9	45	110	30	120
Bis(2-chloroethyl) ether	71.1	11.2	40	105	25	115
Bis(2-chloroisopropyl) ether	68.4	15.7	20	115	10	130
Hexachlorobutadiene	78.2	12.9	40	115	25	130
Hexachloroethane	71.9	12.6	35	110	20	120
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	45	110	30	120
1,2-Dichlorobenzene	70.9	8.7	45	95	35	105
1,3-Dichlorobenzene	69.7	10.3	40	100	30	110
1,4-Dichlorobenzene	69.0	11.4	35	105	25	115
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	45	115	35	130
4-Chlorophenyl phenyl ether	79.6	10.7	45	110	35	120
Hexachlorobenzene	82.5	11.7	45	120	35	130
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	50	115	35	130
2,6-Dinitrotoluene	80.2	10.7	50	110	35	125
2-Nitroaniline	81.0	12.2	45	120	30	130
3-Nitroaniline	68.8	13.8	25	110	15	125
4-Nitroaniline	73.6	13.1	35	115	20	125
Nitrobenzene	77.2	11.9	40	115	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	45	115	30	130
Dibenzofuran	77.1	8.8	50	105	40	110
Others						
Benzyl alcohol	70.9	17.4	20	125	10	140
Isophorone	77.0	11.4	45	110	30	125

*See Appendix B for DOD Specific Criteria

UNCONTROLLED COPY

APPENDIX C

INSTRUMENT OPERATING CONDITIONS

*See Appendix B for DOD Specific Criteria

TOPLEVEL PARAMETERS

Method Information For: J:\ACQUDATA\5973A\METHODS\82700317.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

8270

Method Comments:
This is the method used for 8270 TCL analysis on MS 5973-A.

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

==========
HP6890 GC METHOD
==========

OVEN
Initial temp: 50 'C (On)
Initial time: 2.00 min
Maximum temp: 350 'C
Equilibration time: 0.50 min
Ramps:

#	Rate	Final temp	Final time
1	25.00	150	0.00
2	12.00	290	6.00
3	6.00	320	4.23
4	0.0(Off)		

Tem. programm.

Post temp: 0 'C
Post time: 0.00 min
Run time: 32.90 min

FRONT INLET (UNKNOWN)
Mode: Splitless
Initial temp: 80 'C (On)
Pressure: 20.78 psi (On)
Purge flow: 60.0 mL/min
Purge time: 0.20 min
Total flow: 65.3 mL/min
Gas saver: On
Saver flow: 20.0 mL/min

BACK INLET ()

Method: 82700317.M Wed Mar 29 09:10:14 2006 Page: 1

*See Appendix B for DOD Specific Criteria

```
   Saver time:  5.00 min
   Gas type:  Helium

COLUMN 1                                  COLUMN 2
   Capillary Column                          (not installed)
   Model Number:  ZEBRON ZB-5
   5% Phenyl Polysiloxane
   Max temperature:  360 'C
   Nominal length:  30.0 m
   Nominal diameter:  250.00 um
   Nominal film thickness:  0.50 um
   Mode:  ramped pressure
   Initial pressure:  20.78 psi
   Initial time:  0.00 min
      #  Rate  Final pres  Final time
      1 99.00    70.00         0.30
      2 99.00    20.78         1.88
      3   0.0(Off)
   Post pressure:  7.60 psi
   Nominal initial flow:  2.5 mL/min
   Average velocity:  58 cm/sec
   Inlet:  Front Inlet
   Outlet:  MSD
   Outlet pressure:  vacuum

FRONT DETECTOR (NO DET)                   BACK DETECTOR (NO DET)

SIGNAL 1                                  SIGNAL 2
   Data rate:  20 Hz                         Data rate:  20 Hz
   Type:  test plot                          Type:  test plot
   Save Data:  Off                           Save Data:  Off
   Zero:  0.0 (Off)                          Zero:  0.0 (Off)
   Range:  0                                 Range:  0
   Fast Peaks:  Off                          Fast Peaks:  Off
   Attenuation:  0                           Attenuation:  0

COLUMN COMP 1                             COLUMN COMP 2
   (No Detectors Installed)                  (No Detectors Installed)

THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  300 'C (On)
   Initial time:  0.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

                                          POST RUN
                                             Post Time: 0.00 min

TIME TABLE
   Time       Specifier                      Parameter & Setpoint


                              7673 Injector


     Front Injector:
        Sample Washes                 3
        Sample Pumps                  5

Method: 82700317.M            Wed Mar 29 09:10:14 2006            Page: 2
```

*See Appendix B for DOD Specific Criteria

```
        Injection Volume            1.0 microliters
        Syringe Size               10.0 microliters
        PostInj Solvent A Washes      5
        PostInj Solvent B Washes      0
        Viscosity Delay               1 seconds
        Plunger Speed              Fast
        PreInjection Dwell         0.00 minutes
        PostInjection Dwell        0.00 minutes

     Back Injector:
No parameters specified
                              MS ACQUISITION PARAMETERS


General Information
------- -----------

Tune File                 : MTMSA.U
Acquistion Mode           : Scan


MS Information
-- -----------

Solvent Delay             : 2.50 min

EM Absolute               : False
EM Offset                 : 153
Resulting EM Voltage      : 1223.5

[Scan Parameters]

Low Mass                  : 35
High Mass                 : 500
Threshold                 : 500
Sample #                  : 2       A/D Samples    4

[MSZones]

MS Quad                   : 150 C   maximum 200 C
MS Source                 : 230 C   maximum 250 C


                            END OF MS ACQUISITION PARAMETERS


                          END OF INSTRUMENT CONTROL PARAMETERS
                          ------------------------------------




                          DATA ANALYSIS PARAMETERS
                          ------------------------

Method Name: J:\ACQUDATA\5973A\METHODS\82700317.M


Method: 82700317.M            Wed Mar 29 09:10:14 2006            Page: 3
```

*See Appendix B for DOD Specific Criteria

TOPLEVEL PARAMETERS

Method Information For: J:\ACQUDATA\5973C\METHODS\LVI1214.M

Method Sections To Run:

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
(X) Data Analysis
( ) Post-Run Cmd/Macro =

8270 LVI.

Method Comments:
This is the method used for 8270 TCL analysis on MS 5973-B.

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

6890 GC METHOD

OVEN
Initial temp: 40 'C (On)
Initial time: 4.00 min
Ramps:

#	Rate	Final temp	Final time
1	10.00	80	0.00
2	30.00	270	0.00
3	10.00	300	5.00
4	30.00	320	5.00
5	0.0(Off)		

Post temp: 0 'C
Post time: 0.00 min
Run time: 28.00 min

Maximum temp: 335 'C
Equilibration time: 0.50 min

Temperature

FRONT INLET (HP PTV)
Mode: Pulsed Splitless
Initial temp: 200 'C (On)
Initial time: 0.00 min
Ramps:

#	Rate	Final temp	Final time
1	360.00	300	0.00
2	0.0(Off)		

BACK INLET (SPLIT/SPLITLESS)
Mode: Split
Initial temp: 50 'C (Off)
Pressure: 0.00 psi (Off)
Total flow: 45.0 mL/min
Gas saver: Off
Gas type: Helium

Method: LVI1214.M Wed Mar 29 09:10:43 2006 Page: 1

*See Appendix B for DOD Specific Criteria

```
   Cryo:  Off
   Cryo use temp:  25 'C
   Cryo Timeout:  10.00 min (On)
   Cryo Fault:  On
   Pressure:  4.39 psi (On)
   Pulse pressure:  30.0 psi
   Pulse time:  1.00 min
   Purge flow:  10.0 mL/min
   Purge time:  2.00 min
   Total flow:  13.5 mL/min
   Gas saver:  Off
   Gas type:  Helium

COLUMN 1                                   COLUMN 2
   Capillary Column                           (not installed)
   Model Number:  RESTEK 12738-127
   RTX-5MS
   Max temperature:  330 'C
   Nominal length:  30.0 m
   Nominal diameter:  250.00 um
   Nominal film thickness:  0.25 um
   Mode:  ramped flow
   Initial flow:  0.8 mL/min
   Initial time:  2.00 min
      #  Rate  Final flow  Final time
      1  1.00      1.0         3.00
      2  1.00      1.2         8.00
      3  1.00      1.5         0.00
   Post flow:  0.8 mL/min
   Nominal init pressure:  4.39 psi
   Average velocity:  32 cm/sec
   Inlet:  Front Inlet
   Outlet:  MSD
   Outlet pressure:  vacuum

FRONT DETECTOR (NO DET)                    BACK DETECTOR (NO DET)

SIGNAL 1                                   SIGNAL 2
   Data rate:  20 Hz                          Data rate:  20 Hz
   Type:  test plot                           Type:  test plot
   Save Data:  Off                            Save Data:  Off
   Zero:  0.0 (Off)                           Zero:  0.0 (Off)
   Range:  0                                  Range:  0
   Fast Peaks:  Off                           Fast Peaks:  Off
   Attenuation:  0                            Attenuation:  0

COLUMN COMP 1                              COLUMN COMP 2
   (No Detectors Installed)                   (No Detectors Installed)

THERMAL AUX 2
   Use:  MSD Transfer Line Heater
   Description:
   Initial temp:  300 'C (On)
   Initial time:  0.00 min
      #  Rate  Final temp  Final time
      1   0.0(Off)

                                           POST RUN
                                              Post Time: 0.00 min

Method: LVI1214.M          Wed Mar 29 09:10:43 2006          Page: 2
```

*See Appendix B for DOD Specific Criteria

```
TIME TABLE
  Time       Specifier                      Parameter & Setpoint

                              7673 Injector

    Front Injector:
       Sample Washes                  3
       Sample Pumps                   5
       Injection Volume             2.0 microliters
       Syringe Size                10.0 microliters
       PostInj Solvent A Washes       5
       PostInj Solvent B Washes       0
       Viscosity Delay                0 seconds
       Plunger Speed               Fast
       PreInjection Dwell          0.00 minutes
       PostInjection Dwell         0.00 minutes

    Back Injector:
No parameters specified
                                MS ACQUISITION PARAMETERS

General Information
------- -----------

Tune File                 : LVIMSC.U
Acquistion Mode           : Scan

MS Information
-- -----------

Solvent Delay             : 6.00 min

EM Absolute               : False
EM Offset                 : 200
Resulting EM Voltage      : 1564.7

[Scan Parameters]

Low Mass                  : 35.0
High Mass                 : 500.0
Threshold                 : 500
Sample #                  : 2       A/D Samples    4

[MSZones]

MS Quad                   : 150 C   maximum 200 C
MS Source                 : 230 C   maximum 250 C

                              END OF MS ACQUISITION PARAMETERS

                            END OF INSTRUMENT CONTROL PARAMETERS
                            ------------------------------------

Method: LVI1214.M             Wed Mar 29 09:10:43 2006             Page: 3
```

*See Appendix B for DOD Specific Criteria