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FINAL

Sampling and Analysis Plan

PCB Investigation

Site 1 – Former Drum Marshalling Area

Naval Weapons Industrial Reserve Plant

Bethpage, New York

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62470-08-D-1001
Contract Task Order WE44

May 2010

TETRA TECH

SAP Worksheet No. 1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
May 2010

NAVAL WEAPONS INDUSTRIAL RESERVE PLANT (NWIRP)
BETHPAGE, NEW YORK
SITE 1 – PCB INVESTIGATION

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 19406
610-491-9688

Prepared under:
Contract No. N62470-08-D-1001
Contract Task Order WE44

Review Signatures:

T. E. Johnston 5/20/10
Tom Johnston, PhD/CLEAN QAM/Date
Tetra Tech NUS, Inc.

5/20/10
Rob Sok/Project Manager/Date
Tetra Tech NUS, Inc.

Approval Signatures:

5/20/10
Lora Fly/RPM/Date
NAVFAC MidLant

*Government Chemist signature is attached

SAP Worksheet No. 1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

DRAFT FINAL

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
March 2010

NAVAL WEAPONS INDUSTRIAL RESERVE PLANT (NWIRP)
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Review Signatures:

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Lora Fly/RPM/Date
NAVFAC MidLant

Jonathan Tucker 3/29/10
Government Chemist/Date
NAVFAC QA Review

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EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for an additional Polychlorinated Biphenyl (PCB) Investigation at Naval Weapons Industrial Reserve Plant (NWIRP) Bethpage, Site 1 – Former Drum Marshalling Area (Site) in Bethpage, New York under Contract Task Order (CTO) No. WE44, Contract N62470-08-D-1001, Comprehensive Long-Term Environmental Action Navy (CLEAN). This PCB investigation will focus on delineating the vertical extent of PCB-contaminated soils in the source areas and determining if PCBs have impacted groundwater beyond the Site boundary.

Starting in 1969, hazardous waste management practices for Grumman facilities on Long Island included marshalling of drummed wastes on the Navy property at NWIRP Bethpage. This storage first took place on a cinder-covered surface over the cesspool field east of Plant 3 (Site 1). From the early 1950s through about 1978, drums containing waste were stored here. In 1978, the collection and marshalling point was moved a few yards south of the original unpaved site, to an area on a 100-by-100 foot concrete pad. This pad had no cover and no berms for containment of spills. Reportedly, all drums of waste marshaled at the Former Drum Marshalling Area were taken off-site by a private contractor for treatment or disposal. There are no reports of leaks or spills of drum contents. Materials stored at the Former Drum Marshalling Area included halogenated and non-halogenated waste solvents. Cadmium and cyanide wastes were also stored in this area. Reportedly, 200 to 300 drums were stored at each area at any one time. In 1982, drummed waste storage was transferred to a Drum Marshalling facility located in the Salvage Storage Area (Site 3). A cover was added to the pad in 1983.

A Record of Decision (ROD) for site soils was issued in May 1995. The 1995 ROD summarized what was at the time believed to be the nature and extent of contamination and identified where more data needed to be collected to further delineate the extent of contamination. The ROD also identified the Remedial Action Objectives (RAOs) and the selected remedy for Site 1. Implementation of the ROD began in 1995 with post-ROD remedial design studies intended to delineate the extent of PCB contamination at depth. The remedial design studies, completed in 1998, revealed that the depth of PCB contamination far exceeded the depth estimated in the ROD. An Air Sparging/Soil Vapor Extraction (AS/SVE) system went into operation in 1998 at Site 1 to address the Volatile Organic Compound (VOC)-contaminated soils and continued operating through 2002. In October 2002, the Navy reported that the objectives of the AS/SVE system had been met and recommended removal of the system. In December 2003, the New York State Department of Environmental Conservation (NYSDEC) concurred with this recommendation and the AS/SVE system was removed.

Prior investigations have shown PCB contamination in Site 1 soils at depths greater than 60 feet below ground surface (bgs) and at concentrations greater than 1,000 milligrams per kilogram (mg/kg). Groundwater sampling has indicated detections of PCBs in downgradient monitoring wells. This PCB investigation will focus on determining the vertical extent of PCB contaminated soils in the source areas and determining if PCBs have impacted groundwater beyond the Site boundary.

Soil and groundwater samples will be collected and analyzed for site-related VOCs and Target Compound List (TCL) PCBs. Soil samples will also be collected and analyzed for Total Petroleum Hydrocarbon (TPH) Diesel Range Organics (DRO) and Gasoline Range Organics (GRO), and Total Organic Carbon (TOC). Aqueous and solid investigation-derived wastes (IDW) will be analyzed for Hazardous Waste characteristics to ensure proper disposal. Samples will be analyzed by analytical methods with sufficient sensitivity to provide the detection limits required for comparison of results to applicable human health risk screening values, which are identified as the Project Screening Limits (PSLs).

The SAP contained herein was generated for, and complies with, applicable United States Navy, NYSDEC, and United States Environmental Protection Agency (USEPA) Region 2 requirements, regulations, guidance, and technical standards, as appropriate. This includes the Department of Defense (DoD), Department of Energy (DoE), and USEPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. To comply with IDQTF requirements, the SAP is presented in the format of standard worksheets as specified in the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) guidance document (IDQTF, 2005).

This SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the planned investigations at Site 1.

Protocols for sample collection, handling, and storage, chain-of-custody, laboratory and field analyses, data validation, and reporting are also addressed in this SAP. The investigation procedures used will comply with Tetra Tech Standard Operating Procedures (SOPs), which are included in Appendix A. The field work and sampling are scheduled to begin in April 2010. A complete project schedule is detailed in Worksheet No. 16.

Prior to conducting field activities under this SAP, a site-specific Health and Safety Plan will be completed to provide the safety and health requirements, practices, and procedures for Tetra Tech personnel participating in Site 1 investigative activities at NWIRP Bethpage.

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Appendices

** (See attached CD for Appendices) **

Appendix A – Tetra Tech SOPs

Appendix B – Laboratory SOPs

Appendix C – Dexsil L2000DX User's Manual and Soil Extraction Procedure

Acronyms

°C	Degrees Celsius
%	Percent
%D	Percent Difference or Percent Drift
%R	Percent Recovery
AS/SVE	Air Sparging/Soil Vapor Extraction
bgs	Below Ground Surface
BFB	Bromofluorobenzene
CCB	Continuing Calibration Blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CSM	Conceptual Site Model
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Absorption
DNAPL	Dense Non-Aqueous Phase Liquid
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
ECOR	ECOR Solutions, Inc.
ELAP	Environmental Laboratory Accreditation Program
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
g	Gram
GC/ECD	Gas Chromatograph/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
Grumman	Northrop Grumman Corporation
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HNUS	Halliburton NUS
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectroscopy
ICS	Interference Check Standard

ICV	Initial Calibration Verification
IDW	Investigation-Derived Waste
IS	Internal Standard
L	Liter
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
mg/kg	Milligrams per Kilogram
mL	Milliliter
mV	Millivolt
MPC	Measurement Performance Criterion
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAPL	Non-Aqueous Phase Liquid
NAVAIR	Naval Air Systems Command
NAVFAC	Naval Facilities Engineering Command
NTU	Nephelometric Turbidity Unit
NWIRP	Naval Weapons Industrial Reserve Plant
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OSHA	Occupational Safety and Health Administration
oz	Ounce
PAH	Polycyclic Aromatic Hydrocarbon
PSL	Project Screening Level
PCB	Polychlorinated Biphenyl
PDF	Portable Document Format
PID	Photoionization Detector
PM	Project Manager
POC	Point of Contact
PRAP	Proposed Remedial Action Plan
PT	Proficiency Testing
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QC	Quality Control
QL	Quantitation Limit
QSM	Quality Systems Manual

r	Linear Regression Correlation Coefficient
r^2	Coefficient of Determination
RF	Response Factor
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RT	Retention Time
SAP	Sampling and Analysis Plan
SCG	Soil Cleanup Goal
SCO	Soil Cleanup Objective
SDG	Sample Delivery Group
SMC	System Monitoring Compound
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TBD	To Be Determined
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
Tetra Tech	Tetra Tech NUS, Inc.
TPH	Total Petroleum Hydrocarbons
TPH-DRO	Total Petroleum Hydrocarbons-Diesel Range Organics
TPH-GRO	Total Petroleum Hydrocarbons-Gasoline Range Organics
TOC	Total Organic Carbon
TriMatrix	TriMatrix Laboratories, LLC
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
µg/L	Micrograms per Liter
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
UV	Ultraviolet
VOA	Volatile Organic Analyte
VOC	Volatile Organic Compound
ZHE	Zero Headspace Extractor

SAP Worksheet No. 2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Naval Weapons Industrial Reserve Plant (NWIRP) Bethpage Site 1
Operable Units:	Operable Unit #1
Contractor Name:	Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number:	N62470-08-D-1001
Contract Title:	Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number:	Contract Task Order (CTO) WE44

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

2. Identify regulatory program: New York State Department of Environmental Conservation (NYSDEC) Inactive Hazardous Waste Disposal Site Program (State Superfund).

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Naval Facilities Engineering Command (NAVFAC) and Tetra Tech Team Meeting	8/10/2009
Data Quality Objective (DQO) Meeting with NYSDEC, Navy, and Tetra Tech	11/10/2009

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

Title	Date
Not Applicable (NA)	NA

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

Naval Air Systems Command (NAVAIR) (property owner), NAVFAC Atlantic (lead organization), NYSDEC (regulatory oversight), and Tetra Tech (Navy contractor)

7. Lead organization: NAVFAC Atlantic

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

NA, as there are no exclusions.

UFP-QAPP Worksheet No.	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	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
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	Quality Control (QC) Samples Table Screening/Confirmatory Analysis Decision Tree	NA

UFP-QAPP Worksheet No.	Required Information	Crosswalk to Related Information
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 No. 3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address	Document Control Number
Lora Fly	Remedial Project Manager (RPM)/Manages Project Activities for the Navy	NAVFAC Mid-Atlantic	757-444-0781	lora.fly@navy.mil	NA
Bonnie Capito (copy of final cover letter only)	Librarian and Records Manager/Administrative Record	NAVFAC Mid-Atlantic	757-322-4785	bonnie.capito@navy.mil	NA
Glenn Wagner	NAVFAC Atlantic Administrative Record Project Manager (PM)/Administrative Record	Tetra Tech	412-921-8386	glenn.wagner@tetratech.com	NA
Al Taormina	NWIRP Bethpage Facility Point of Contact (POC)	ECOR Solutions, Inc. (ECOR)	516-346-0344	altaormina@worldnet.att.net	NA
Steve Scharf	RPM/Regulatory Oversight	NYSDEC	518-402-9620	sxscharf@gw.dec.state.ny.us	NA
John Trepanowski	Program Manager/Manages Program Activities	Tetra Tech	610-491-9688	john.trepanowski@tetratech.com	NA
Garth Glenn (copy of cover letter only)	Deputy Program Manager/Manages Program Activities	Tetra Tech	757-461-3926	garth.glenn@tetratech.com	NA
Dave Brayack	Project Engineer/Assists PM	Tetra Tech	757-461-3824	dave.brayack@tetratech.com	NA
Rob Sok	PM/Manages Project Activities	Tetra Tech	757-466-4904	rob.sok@tetratech.com	NA
Tom Johnston (electronic copy only)	Quality Assurance Manager (QAM)/Manages Corporate Quality Assurance (QA) Program and Implementation	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Matt Soltis [Health and Safety Plan (HASP) only]	Health and Safety Manager (HSM)/ Manages Corporate Health and Safety Program	Tetra Tech	412-921-8912	matt.soltis@tetratech.com	NA
Joe Samchuck (electronic copy only)	Data Validation Manager (DVM) /Manages Data Validation	Tetra Tech	412-921-8510	joseph.samchuck@tetratech.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-Mail Address or Mailing Address	Document Control Number
Lee Leck (electronic copy only)	Data Manager/Manages Databases	Tetra Tech	412-921-8856	lee.leck@tetratech.com	NA
TBD	Field Operations Leader (FOL) /Site Safety Officer (SSO) /Manages Field Operations	Tetra Tech	TBD	TBD	NA
Megan Ritchie (electronic copy only)	Project Chemist/Provides Technical Coordination with Laboratory(s)	Tetra Tech	610-382-1527	megan.ritchie@tetratech.com	NA
Phil Komar (electronic copy only)	Laboratory PM/Representative for Laboratory and Analytical Issues	TriMatrix Laboratories, LLC (TriMatrix)	616-846-9528	komarp@trimatrixlabs.com	NA
Driller (TBD) (electronic copy only)	Well Installation Subcontractor PM/ Provides Drilling Services	TBD	TBD	TBD	NA
Surveyor (TBD) (electronic copy only)	Surveyor/Provides Surveying Services	TBD	TBD	TBD	NA
Investigation-Derived Waste (IDW) Subcontractor (electronic copy only)	IDW Subcontractor/Provides IDW Services	TBD	TBD	TBD	NA

Each person in this table will be responsible for distributing copies of this SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of this SAP to all Tetra Tech personnel listed in Worksheet #4 (Project Personnel Sign-Off Sheet).

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

(UFP-QAPP Manual Section 2.3.2)

Name [1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Lora Fly	Navy RPM/Manages Project Activities for the Navy	757-444-0781	See Worksheet 1 for signature	All	
Steve Scharf	NYSDEC RPM/Regulatory	518-402-9620		All	
Tetra Tech Project Team Personnel					
Rob Sok	Tetra Tech PM/Manages Project Activities	757-466-4904	See Worksheet 1 for signature	All	
TBD	Tetra Tech FOL/SSO/Manages Field Operations	TBD		All	
Tom Johnston	Tetra Tech QAM/Manages Corporate QA Program and Implementation	412-921-8615	See Worksheet 1 for signature	All	
Matt Soltis	Tetra Tech HSM/Manages Corporate Health and Safety Program	412-921-8912	See HASP for signature[2]	All	
Joe Samchuck	Tetra Tech DVM/Manages Data Validation	412-921-8510		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Lee Leck	Tetra Tech Data Manager/Manages Databases	412-921-8856		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Megan Ritchie	Tetra Tech Project Chemist/Provides Technical Coordination with Laboratory(s)	610-382-1527		All	
Subcontractor Personnel					
Phil Komar	TriMatrix Laboratory PM/ Representative for Laboratory and Analytical Issues	616-846-9528		Worksheet Nos. 6, 12, 14, 15, 19, 20, 23-28, 30, 34-36	

Name [1]	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Driller	Well Installation Subcontractor PM/ Provides Drilling Services	TBD		Worksheet Nos. 6, 14, 17, and Figures	
Surveyor	Surveyor/Provides Surveying Services	TBD		Worksheet No. 14 and Figures	
IDW Subcontractor	IDW Subcontractor/Provides IDW Services	TBD		Worksheet No. 14	

[1] – Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.
[2] – The HSM will be responsible for developing the site-specific HASP based on field activities presented in this SAP.

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

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

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

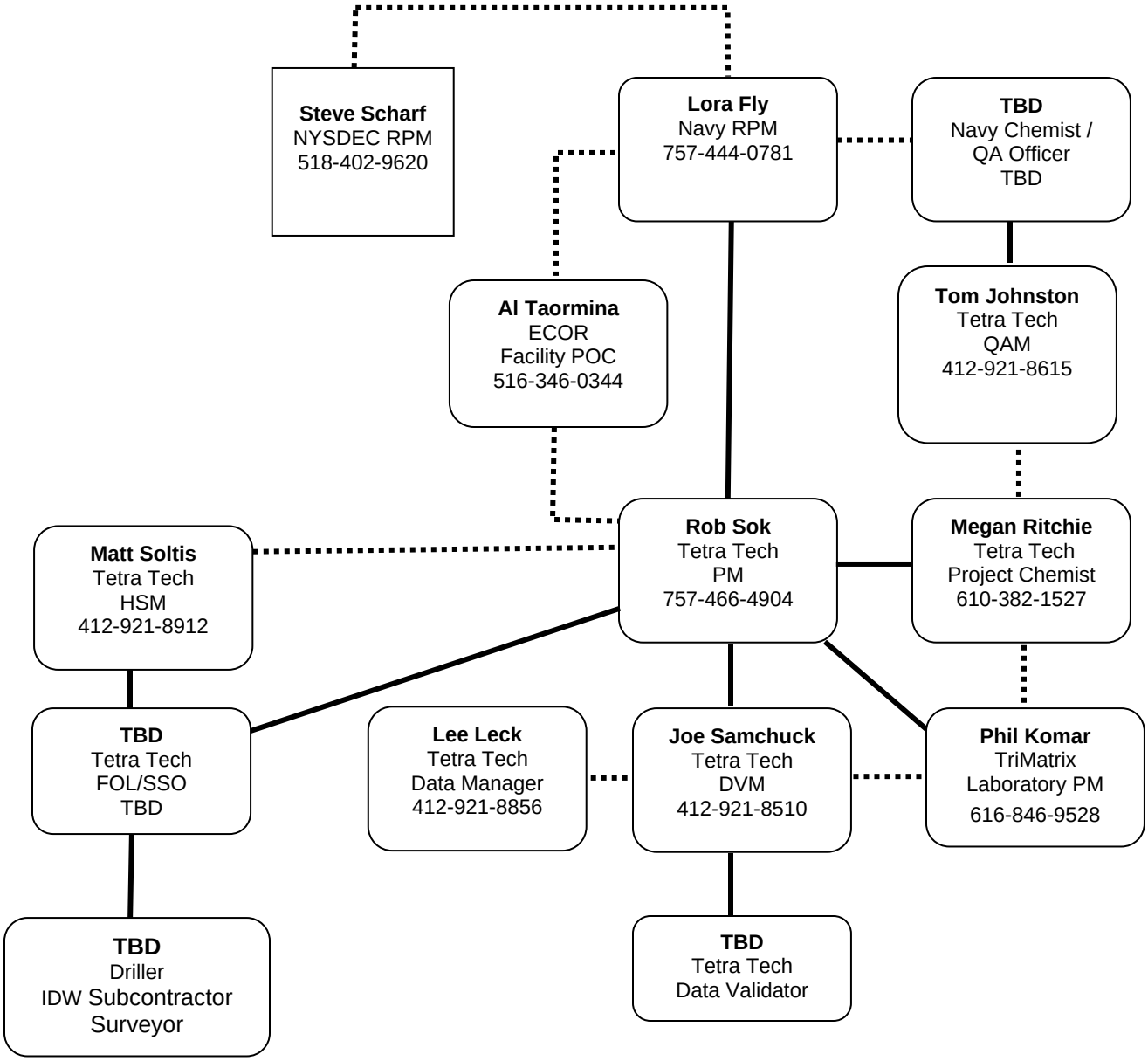
3. A copy of the signed Worksheet No. 4 will be retained in the project files and identified as a project document in Worksheet No. 29.

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

SAP Worksheet No. 5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

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



SAP Worksheet No. 6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Regulatory agency review	NYSDEC RPM	Steve Scharf	518-402-9620	Reviews technical documents prior to completion of the final SAP.
SAP amendments	Tetra Tech FOL Tetra Tech PM Navy RPM	TBD Rob Sok Lora Fly	TBD 757-466-4904 757-444-0781	Tetra Tech FOL will verbally inform Tetra Tech PM within 24 hours of realizing a need for an amendment. Tetra Tech PM will document the changes via a Field Task Modification Request (FTMR) form within five days and send the Navy RPM a concurrence letter within seven days of identifying the need for change. SAP Amendments submitted by Tetra Tech PM to NAVFAC Mid-Atlantic Program Management Office for review and approval. Tetra Tech PM will send scope changes to Project Team via e-mail within one business day.
Changes in schedule	Tetra Tech FOL Tetra Tech PM Navy RPM Facility POC	TBD Rob Sok Lora Fly Al Taormina	TBD 757-466-4904 757-444-0781 516-346-0344	Tetra Tech PM will verbally inform Navy RPM and Facility POC on the day that schedule change is known and document via schedule impact letter within one business day of when impact is realized.
Issues in the field that result in changes in scope of field work	Tetra Tech FOL Tetra Tech PM Navy RPM	TBD Rob Sok Lora Fly	TBD 757-466-4904 757-444-0781	Tetra Tech FOL will verbally inform Tetra Tech PM within one business day of when the issue is discovered. Tetra Tech PM will inform Navy RPM (verbally or via e-mail) by close of the next working day. Navy RPM will issue scope change approval (verbally or via e-mail) if warranted; scope change will be implemented before work is executed. The Tetra Tech FOL will document the changes on a FTMR form within two days of identifying the need for change and obtain required approvals within five days of initiating the form.

Recommendations to stop work and initiate work upon corrective action	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Tetra Tech HSM Navy RPM Facility POC	TBD Rob Sok Tom Johnston Matt Soltis Lora Fly Al Taormina	TBD 757-466-4904 412-921-7273 412-921-8912 757-444-0781 516-346-0344	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the Navy RPM, the Facility POC, and the identified Project Team members within one hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
Corrective action for field program	Tetra Tech QAM Tetra Tech PM	Tom Johnston Rob Sok	412-921-8615 757-466-4904	Tetra Tech QAM will notify Tetra Tech PM within one business day that the corrective action has been completed. Tetra Tech PM will then notify the Navy RPM within one business day.
Field data quality issues	Tetra Tech FOL/SSO Tetra Tech PM	TBD Rob Sok	TBD 757-466-4904	Tetra Tech FOL will inform Tetra Tech PM verbally or by e-mail on the same day that a field data quality issue is discovered.
Analytical data quality issues	TriMatrix Laboratory PM Tetra Tech Project Chemist	Phil Komar Megan Ritchie	616-846-9528 610-382-1527	Laboratory PM will notify (verbally or via e-mail) Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered. Tetra Tech Project Chemist will notify (verbally or via e-mail) data validation staff and Tetra Tech PM within one business day.

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

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
John Trepanowski	Program Manager	Tetra Tech	Oversees NAVFAC Program.
Rob Sok	PM	Tetra Tech	Oversees administrative, financial, schedule, and day-to-day technical management of the project, including the following: • Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations. • Functions as the primary Tetra Tech interface with the Navy RPM, facility personnel, Tetra Tech field and office personnel, and laboratory points of contact. • Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories. • Monitors and evaluates all Tetra Tech subcontractor performance. • Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data validation, data interpretation, and report preparation). • Coordinates and oversees maintenance of all Tetra Tech project records. • Coordinates and oversees review of Tetra Tech project deliverables. • Prepares and issues final Tetra Tech deliverables to the Navy.
TBD	FOL/SSO	Tetra Tech	Supervises, coordinates, and performs field sampling activities, including the following: • Ensures that health and safety requirements unique to the project are implemented. • Functions as the on-site communications link between field staff members, the facility POC, and the Tetra Tech PM. • Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • Oversees the mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to the work schedules provided by the Tetra Tech PM. • Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs when necessary. • Identifies and resolves problems in the field, in consultation with the Facility POC, implementing and documenting corrective action procedures, and providing communication between the field team and project management. As SSO, will be responsible for training project personnel and monitoring site conditions related to personnel safety. Details of the SSOs responsibilities are presented in the HASP.

Name	Title/Role	Organizational Affiliation	Responsibilities
Tom Johnston	QAM	Tetra Tech	Reviews the SAP, oversees preparation of the laboratory scope, and conducts data quality reviews. Ensures quality aspects of the CLEAN program are implemented, including the following: • Develops, maintains, and monitors QA policies and procedures. • Provides training to Tetra Tech staff in QA/QC policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, SAP requirements, and corporate policies and procedures. • Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of corrective action plans and ensures correction of non-conformances reported in internal or external audits. • Ensures that this SAP meets Tetra Tech, Navy, USEPA, and NYSDEC QA requirements. • Prepares QA reports for management.
Megan Ritchie	Project Chemist	Tetra Tech	Coordinates analyses with laboratory chemists, ensures the scope is followed and that QA has been performed for QA data packages, and communicates with Tetra Tech staff. • Assists in the preparation of the UFP-SAP. • Ensures that the project meets objectives from the standpoint of laboratory performance. • Provides technical advice to the Project Team on project chemistry matters. • Monitors and evaluates subcontracted laboratory performance. • Ensures timely resolution of laboratory-related technical, quality, or other issues affecting project goals. • Functions as the primary interface with the subcontracted laboratories and the Tetra Tech PM. • Coordinates and oversees work performed by the subcontracted laboratories. • Oversees the completion of Tetra Tech data validation. • Coordinates and oversees review of laboratory deliverables. • Recommends appropriate laboratory corrective actions.
Joseph Samchuck	DVM	Tetra Tech	Manages data validation activities within Tetra Tech, including the following: • Ensures QA of data validation deliverables. • Oversees data validation activities, including review schedule. • Serves as communication link between Tetra Tech and laboratory(s) on data validation and electronic data posting activities. • Provides technical advice on data usability. • Establishes Tetra Tech data validation protocols in support of projects.
Lee Leck	Data Manager	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.

Name	Title/Role	Organizational Affiliation	Responsibilities
Matt Soltis	HSM	Tetra Tech	Oversees CLEAN Program Health and Safety Program, including the following: • Provides technical advice to the Tetra Tech PM on matters of health and safety. • Oversees the development and review of the HASP. • Conducts health and safety audits. • Prepares health and safety reports for management.
Phil Komar	Laboratory PM	TriMatrix	Interfaces directly with the Tetra Tech Project Chemist, Tetra Tech PM, and Tetra Tech QAM. • Ensures that the laboratory scope is followed. • Coordinates analyses with laboratory chemists. • Performs QA on data packages for completeness, clarity, and compliance with project requirements. • Informs the Tetra Tech PM of project status and any sample receipt or analytical problems.
TBD	Driller	TBD	• Performs Rotosonic drilling techniques to advance soil borings and install monitoring wells according to scope of work.
TBD	IDW Subcontractor	TBD	• Responsible for transport and disposal of IDW according to scope of work.
TBD	Surveyor	TBD	• Determines location data for soil sample and well locations according to scope of work.

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

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

(UFP-QAPP Manual Section 2.4.4)

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 site-specific HASP which will be provided to the Navy and NYSDEC at their request prior to field activities.

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

(UFP-QAPP Manual Section 2.5.1)

Project Name: Polychlorinated Biphenyl (PCB) Investigation Projected Date(s) of Sampling: April 2010-November 2010 Project Manager: Robert Sok	Site Name: Site 1, Former Drum Marshalling Area Site Location: Bethpage, New York

Date of Session: August 10, 2009
Scoping Session Purpose: Scoping Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Lora Fly	RPM	NAVFAC	757-444-0781	lora.fly@navy.mil	RPM
Donna Caldwell	Senior Environmental Scientist	NAVFAC	757-322-4816	donna.caldwell@navy.mil	Technical Support
Robert Sok	PM	Tetra Tech	757-466-4904	rob.sok@tetratech.com	Management

Comments/Decisions: Discussed the scope of work and the need for additional soil borings and sampling to address preliminary DQOs for PCB Investigation at Site 1. A presentation needs to be prepared to introduce the UFP-SAP process and present draft DQOs to NYSDEC prior to moving forward with UFP-SAP production.

Action Items: Tetra Tech will prepare cost modification for CTO WE44 to address need for additional soil borings and sampling at Site 1. Tetra Tech will prepare a presentation to introduce UFP-SAP and DQOs for the PCB Investigation to NYSDEC.

Consensus Decisions: Preparation of UFP-SAP is on hold until UFP-SAP process and DQOs for the project are presented to NYSDEC.

Project Name: PCB Investigation Projected Date(s) of Sampling: April 2010-November 2010 Project Manager: Robert Sok	Site Name: Site 1, Former Drum Marshalling Area Site Location: Bethpage, New York

Date of Session: November 10, 2009
Scoping Session Purpose: DQO and SAP Meeting

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Lora Fly	RPM	NAVFAC	757-444-0781	lora.fly@navy.mil	RPM
Robert Sok	PM	Tetra Tech	757-466-4904	rob.sok@tetratech.com	Management
Dave Brayack	Engineer	Tetra Tech	610-382-1173	dave.brayack@tetratech.com	Engineer
Steve Scharf	Engineer	NYSDEC	518-402-9620	sxscharf@gw.dec.state.ny.us	Regulator
John Swartwout	Section Chief - Engineer	NYSDEC	518-402-9620	jbswarto@sw.dec.state.ny.us	Regulator

Comments/Decisions: Introduced the UFP-SAP process to NYSDEC and presented DQOs and general details for the proposed PCB investigation. NYSDEC introduced new draft soil cleanup guidance that was issued on November 4, 2009 and presented the soil cleanup levels of 1 ppm for surface soils and 10 ppm for PCBs in subsurface soils. Project Screening Levels (PSLs) will target 10 ppm for PCBs in subsurface soils at Site 1.

Action Items: Tetra Tech will prepare the UFP-SAP using the DQOs presented and submit to NYSDEC for review.

Consensus Decisions: NYSDEC was agreeable with overall scope of PCB Investigation and moving forward with UFP-SAP production.

SAP Worksheet No. 10 -- Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

The following Sections 10.1 – 10.3 present background information concerning Site 1 – Former Drum Marshalling Area, including site location and description, site history, and a brief summary of the site geology and hydrogeology. A summary of environmental investigations and remediation conducted at Site 1 is presented in Section 10.4 and Section 10.5, which discusses the limitations of data that were previously collected. Section 10.6 summarizes the Conceptual Site Model (CSM) and presents a description of the problem to be addressed by this investigation.

10.1 SITE LOCATION AND DESCRIPTION

NWIRP Bethpage is located in east-central Nassau County, Long Island, New York, approximately 30 miles east of New York City (**Figure 1**). Site 1 is situated along the eastern boundary of the former NWIRP Bethpage (**Figure 2**). Historically, NWIRP Bethpage was situated on 109 acres in Nassau County in the Hamlet of Bethpage, Town of Oyster Bay, New York. NWIRP Bethpage was located entirely within the Northrop Grumman Aerospace complex, which covered approximately 605 acres. The former NWIRP property was bordered on the north, west, and south by Northrop Grumman Corporation (Grumman) facilities, and on the east by a residential neighborhood. In 2002, 4 acres were transferred to Grumman including the area known as Plant 20. In 2008, 96 acres of the 109 former acres of NWIRP property were transferred from the Navy to the County of Nassau. The remaining 9 acres and access rights-of-way were retained by the Navy to continue the remedial efforts at Site 1 and Site 4. The 9-acre parcel of NWIRP is bordered on the east by the residential neighborhood and on the north, south, and west by county property. Access to the facility is from South Oyster Bay Road to the west.

10.2 SITE HISTORY

NWIRP Bethpage was established in 1942. Since its inception, the plant's primary mission has been the research prototyping, testing, design engineering, fabrication, and primary assembly of military aircraft. The facility included four plants used for assembly and prototype testing, a group of quality control laboratories, two warehouse complexes (north and south), a salvage storage area, groundwater recharge basins, the Industrial Wastewater Treatment Plant, and several smaller support buildings.

Starting in 1969, hazardous waste management practices for Grumman facilities on Long Island included marshalling of drummed wastes on the Navy property at NWIRP Bethpage. This storage first took place on a cinder-covered surface over the cesspool field east of Plant 3. From the early 1950s through about 1978, drums containing waste were stored here. In 1978, the collection and marshalling point was moved a few yards south of the original unpaved site, to an area on a 100-by-100 foot concrete pad. This pad had no cover and no berms for containment of spills. In 1982, drummed waste storage was transferred

to a Drum Marshalling facility located in the Salvage Storage Area (Site 3). A cover was added to Site 1 in 1983.

In 1985, an Initial Assessment Study (IAS) was conducted at NWIRP Bethpage that identified materials stored at Site 1, the Former Drum Marshaling Area, to include waste halogenated and non-halogenated solvents (Rogers, Golden & Halpern, 1986). Cadmium and cyanide wastes were also stored in this area from the early 1950s through 1974. Reportedly, 200 to 300 drums were stored at each area at any one time. Based on the data presented in the IAS, it was determined that there was a potential for volatile organic, semivolatile organic, and inorganic contamination at each of the three sites. Based on site observations in the early 1990s, there was the potential that transformers (possibly containing PCBs) and other high temperature devices (autoclaves) may have also been stored at these sites. However, there are no historic records to substantiate these observations and it is unknown whether or not the transformers were properly drained prior to storage.

Reportedly, there was no direct evidence of hazardous waste spills at Site 1 and all drums of waste marshaled at the Former Drum Marshalling Areas were reportedly taken off-site by a private contractor for treatment or disposal. There are no reports of leaks or spills of drum contents. An abandoned septic drainage system almost completely underlies Site 1.

In 1997, Grumman ceased its operations at NWIRP Bethpage. Since 1997, activities at the facility have included facility maintenance (security and mowing), storage of Nassau County impounded vehicles, and environmental investigations and/or remediation of soil, groundwater, and soil vapor.

10.3 GEOLOGY AND HYDROGEOLOGY

NWIRP Bethpage is underlain by approximately 1,100 feet of unconsolidated sediments that overlie crystalline bedrock (Isbister, 1966). The unconsolidated sediments consist of four distinct geologic units: (in descending order) Upper Glacial Formation, Magothy Formation, Raritan Clay, and Lloyd Sand Formation. The 30- to 45-foot-thick Upper Glacial Formation consists chiefly of coarse sands and gravels. The Upper Magothy Formation consists chiefly of coarse sands to a depth of approximately 100 feet, below which finer sands, silts, and clay predominate. The clay is common but laterally discontinuous; no individual clay horizon of regional extent underlies the facility. The 100- to 150-foot-thick Raritan Clay underlies the Magothy Formation at a depth of approximately 700 feet. The underlying Lloyd Sand Formation is approximately 300 feet thick.

Most of Long Island is bisected by an east-west-trending regional groundwater divide. NWIRP Bethpage occupies an area of recharge, lying to the south of the divide. Groundwater is in communication between the Upper Glacial and Upper Magothy Formations beneath the facility, and may be considered a common unconfined aquifer, which is encountered at approximately 50 feet below ground surface (bgs). The

glacial deposits are characterized by a high primary porosity (exceeding 30 percent) and high permeability. The high permeability of the glacial deposits allows for the rapid recharge of precipitation to the underlying Magothy (Isbister, 1966; McClymonds and Franke, 1972). The number and thickness of clay lenses increase with depth in the Magothy Formation; however, the horizontally discontinuous nature of these units prevents any one of them from functioning as an aquitard or semi-confining unit.

Groundwater beneath the site flows in a general southward direction toward the Atlantic Ocean. The horizontal hydraulic gradient and groundwater velocity in the unconfined common aquifer across the facility average 5.3 feet per mile and 0.3 foot per day, respectively [Halliburton NUS (HNUS), 1993]. Subtle vertical hydraulic gradients occur in a downward direction. Groundwater in the deeper portion of the Magothy is the primary source of potable water in Nassau County. Former NWIRP production wells (now abandoned) set in the Magothy yielded 1,200 gallons per minute. Grumman operates production wells (as well as a groundwater containment system) south of NWIRP Bethpage. The production wells and groundwater containment system operates with a combined flow rate of 3,800 gallons per minute.

10.4 SUMMARY OF ENVIRONMENTAL WORK CONDUCTED

A list of important Site 1 historical events and relevant dates in the site chronology through 2009 is shown below. The identified events are illustrative, not comprehensive. The paragraphs following the table below summarize some details of the historical environmental work conducted at Site 1.

DATE	EVENT
1942	NWIRP Bethpage established
1950s-1978	Cyanide and cadmium stored onsite in Area 2
1978	Drum collection was moved a few yards south to a concrete pad
1982	Drummed waste storage was transferred to the RCRA Compliant Drum Marshalling facility
1983	A cover was added to Site 1
1985	IAS began
1986	IAS completed
1991	Remedial Investigation (RI) began
1992	RI completed
1992	Phase 2 RI began
1993	Phase 2 RI completed
1994	Feasibility Study (FS) completed
1995 May	Record of Decision (ROD) signed
1995 May	Final Submission for Remedial Design Phase 1
1995 July	Draft Remedial Design Phase 1 Pre-Design completed
1995 September	Remedial Design Phase 2 Pre-Design Investigation completed
1995 December	Pre-Excavation soil sampling completed
1996	Pre-Excavation Sampling Results & an estimate on construction
1997 -2001	Installation and operation of onsite AS/SVE system
2002	AS/SVE shutdown
2003	Remediation of Former Drywells 20-08 & 34-07
2003 - 2007	Navy re-evaluates implementation for Site 1 PCBs/metal remedy
2005 - 2007	Soil Vapor Intrusion concerns identified

DATE	EVENT
2008	Soil Gas investigations performed
2009 -present	Soil Vapor Intrusion sampling and mitigation

In 1985, the IAS was conducted at Site 1, followed by a two-phase RI. The purpose of the RI was to identify the nature and extent of contamination associated with Site 1. The chemicals of concern at Site 1 included Volatile Organic Compounds (VOCs), PCBs, metals, and polycyclic aromatic hydrocarbons (PAHs). The Phase 1 RI conducted in May 1992 (HNUS, 1992) and Phase 2 RI conducted in October 1993 (HNUS, 1993) identified the nature of contamination, but was unable to establish the extent of PCB contamination. However, the RI process did adequately delineate the horizontal extent of PCB contaminated soils, and the vertical and horizontal extent of VOC, metal, and PAH contaminated soils. The identified contamination at Site 1 was significant enough to require an Interim Remedial Measure (IRM) that was conducted in July 1993. During the IRM, a soil cover was placed over the limits of Site 1 to eliminate risk associated with fugitive dust and dermal contact. With the IRM complete, a FS was conducted in March 1994 (HNUS, 1994). The FS led to the development of the Proposed Remedial Action Plan (PRAP), issued in October 1994, and the development of the Site 1 ROD, that was issued in May 1995 (NAVFAC, 1995).

The 1995 ROD summarized what was at the time believed to be the nature and extent of contamination and identified where more data was needed to further delineate the extent of contamination. The ROD also identified the Remedial Action Objective (RAOs) and the selected remedy for Site 1. The RAOs included:

- Comply with contaminant-specific, location-specific, and action-specific requirements to achieve the soil cleanup goal (SCG) of 10 milligrams per kilogram (mg/kg) of PCBs.
- Reduce, control, or eliminate the contamination within site soils.
- Prevent human exposure to contaminated soils at concentrations greater than the remedial action goals.
- Prevent leaching of contaminants in soil which could result in groundwater contamination in excess of groundwater remediation goals.
- Prevent offsite migration of contamination.

The selected remedy included:

- Collection of additional samples to verify and provide data necessary for a soil excavation and disposal to remediate the inorganic and PCB contamination and the installation of an AS/SVE system to remediate VOC contamination.

- Excavation of arsenic-contaminated soil (600 cubic yards) and PCB-contaminated soil (1,400 cubic yards) for treatment and disposal (volumes reported in the ROD were based on sampling that extended 5-feet bgs).

- Remediation of VOC-contaminated soils using the AS/SVE system.

- Remediation of VOC-contaminated groundwater using AS.

- Implementation of institutional controls. Institutional controls included a gravel or vegetative cover over residual contamination (permeable cover to encourage natural attenuation of residual VOCs) and deed restrictions to limit the use of and exposure to the Site 1 area.

- Provision for an IRM. Reimbursement of cost to the Bethpage Water District for providing water treatment to the public water supply wells.

Implementation of the ROD began in 1995 when the post-ROD remedial design studies began to delineate the extent of PCB contamination at depth. During the remedial design studies that were completed in 1998, it was determined that the depth of PCB contamination far exceeded the estimated depth reported in the ROD and two former dry well areas (34-07 and 20-08) were added to the Site 1 scope of work. In 1998, the AS/SVE system went into operation and removed approximately 4,500 pounds of VOCs through 2002. In October 2002, the Navy reported that the objectives of the AS/SVE system had been met and recommended removal of the system. In December 2003, the NYSDEC concurred with this recommendation and the AS/SVE system was removed.

Because the extent of PCB contamination was significantly greater than identified in the 1995 ROD, the Navy began looking into reopening the ROD for the purposes of re-evaluating remedial alternatives to address the PCB soil contamination. In the fall of 2006, the Navy began conducting internal meetings ("Tiger Team" meetings) to investigate remedial options and a direction forward for reopening the ROD. The Tiger Team concluded its work with a submission to the NYSDEC in March 2007 that outlined an approach to reopen the ROD. The submission included the current understanding regarding the nature and extent of Site 1 contamination and an evaluation (including cost and implementation) of remedial alternatives (including the original ROD remedy). In September 2008, a technical memorandum was submitted to the NYSDEC (Tetra Tech, 2008a) that evaluated alternatives for soil remediation developed by the Tiger Team in 2007. The report supported an anticipated in-situ pilot scale test to remediate PCB-

contaminated soil. **Figures 3 through 6** present the known extent of PCB-contaminated soils associated with Site 1 and the dry wells, as reported in the technical memorandum in 2008.

Sampling to evaluate the presence of PCBs in groundwater was first conducted in October 2006. A second round of groundwater sampling was conducted in January 2008. Four monitoring wells were sampled, including three water table wells (total depths of approximately 55 feet bgs) and one well at a total depth of approximately 120 feet bgs at the downgradient edge of the site. Low flow groundwater sampling techniques were used and PCBs (Aroclor-1242 and -1248) were detected in each of the wells, with concentrations ranging from 0.27 to 1.4 micrograms per liter (µg/L). **Figure 7** presents the analytical results from groundwater sampling conducted at Site 1 in 2006 and 2008. The New York State Department of Health (NYSDOH) maximum contaminant level (MCL) for PCBs is 0.5 µg/L.

In May 2009, three soil borings were advanced via mud rotary drilling at Site 1 (**Figure 8**). All three borings were advanced to approximately 310 feet bgs and gamma logging was conducted at each location to provide deeper lithological information. Limited subsurface soil sampling for PCBs (11 samples) and Hydro Punch™ groundwater sampling for VOCs (14 samples) were conducted at BPS1-3001 and BPS1-3003. No PCBs or site-related VOCs were detected in the samples. Cross sections A-A' (**Figure 9**), B-B' (**Figure 10**), and C-C' (**Figure 11**) were generated from the gamma log data obtained from the borings. The cross sections show potential clay units at approximately 100 feet and between 200 and 300 feet bgs that could underlay Site 1.

The vertical extent of PCB contamination is known to extend beyond the depth of the groundwater table. However, the actual vertical extent of PCB-contaminated soil in the source areas has not been delineated and the impact of PCBs to downgradient groundwater is uncertain. The known horizontal and vertical extent of PCB contamination defined in historical investigations will be used to position source area borings.

10.5 LIMITATIONS OF PREVIOUS DATA

Data collected during historical investigations at Site 1 have delineated the horizontal and vertical extent of PCB-contaminated soils from the ground surface to 25 feet bgs. These historical investigations did not define the deeper PCB contamination currently suspected in soil and groundwater at Site 1. The known vertical extent of PCB-contaminated soil is to depths of approximately 65 feet bgs, extending below the current top of the water table (approximately 52 feet bgs).

Existing monitoring wells at Site 1 were installed with hollow stem auger. Well installation procedures may have carried PCBs, via soil intrusion, to the depth of the well screens. Also, these monitoring wells were not installed at sufficient depths to monitor potential deeper contamination in site soils.

10.6 CONCEPTUAL SITE MODEL SUMMARY

A diagram of the CSM based on current conditions at the Site is shown on **Figure 12**. The basic components of the conceptual model representing an understanding of the site are presented below.

Contaminant Sources

Based on the historical investigations conducted at Site 1, PCBs and VOCs represent the primary contaminants of concern. It is assumed that the contamination has resulted from releases during surface storage and maintenance activities historically conducted at the site. Some limited inorganic contamination has also been observed in surface soil. Contamination has also been observed within several of the leach pits and is assumed to have resulted from the placement of contaminated soil or wastes into these pits.

Migration Pathways

Migration pathways potentially affecting current conditions at Site 1 include the following:

- Downward migration of PCBs via a carrier fluid (chlorinated and non-chlorinated organics)

PCBs are normally immobilbe. In the presence of organic solvents, PCBs can dissolve and migrate with a mobile solvent. Site history provides evidence that a carrier fluid may have caused PCBs to migrate downward from a surface release through 50 feet of soil, to the water table and/or continue to migrate below the water table unattenuated (i.e., surface soil concentrations at or similar to concentrations at the water table). Identification of this type of carrier fluid (LNAPL or DNAPL) would help define the horizontal and vertical extent of PCB contamination.

- Migration of PCBs via groundwater transport, in either a dissolved or colloidal form.

Once PCBs are in contact with groundwater, they can migrate in groundwater in dissolved or colloidal form. The theoretical solubility of PCBs (aroclor 1242 and 1248) can range from 43 to 100 µg/L, depending on the type of aroclor. Therefore, it is possible to find PCBs in the dissolved phase in groundwater. PCBs can also adsorb onto colloidal size particles and /or oils, which would allow PCBs to migrate without interacting with soil.

Exposure Pathways

Currently, the primary media at the site through which receptors can be exposed to PCBs include groundwater and subsurface soils. Exposure routes include dermal contact, ingestion, and inhalation. Potential exposure pathways at Site 1 include the following:

- Dermal contact with subsurface soils and groundwater;
- Inhalation of fugitive dust; and
- Ingestion of site soils and groundwater.

Potential Receptors

The potential receptors that may be exposed to contaminated media at Site 1 include site workers and area residents via ingestion, inhalation, or dermal contact with contaminated soils or groundwater.

Most of the VOCs in soil and shallow groundwater at Site 1 have been remediated via the historical AS/SVE system that operated from 1998 to 2002. Access restrictions at the site and the soil cover placed during the IRM prevent dermal exposure and ingestion of contaminated surface soils, and also prevent the potential inhalation of fugitive dusts. Currently, there is no exposure to site groundwater via ingestion or dermal contact, as there is no groundwater use at, or immediately downgradient of, Site 1. Mitigation of soil vapor is ongoing to address exposure to receptors with residential systems (including air purifying units and sub-slab depressurization systems) and the SVE containment system currently operating at Site 1.

Future site use is uncertain, but anticipated to be industrial or commercial after completion of remedial actions and transfer of the remaining nine acres to Nassau County.

Summary

The vertical and horizontal extent of PCB-contaminated soils from the ground surface to 25 feet bgs has been defined from historical investigations at Site 1. However, the extent of contamination below 25 feet bgs has not been delineated horizontally or vertically. Lithological data suggests clay units are present at approximately 110 feet and 220 feet bgs in the study area. It is unknown whether these units are continuous below the source areas or provide a boundary for vertical migration of site contaminants. Trace detections of PCBs have been observed in site groundwater. It is uncertain whether the contamination is due to the well installation technique or if the PCB detections are actually representative of downgradient groundwater conditions at Site 1. It is suspected that other known contamination at Site 1 (i.e., organic solvents) may have acted as a carrier fluid and affected the migration of PCBs.

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

(UFP-QAPP Manual Section 2.6.1)

11.1 PROBLEM DEFINITION

Past activities at NWIRP Bethpage Site 1 have impacted soils and may have impacted groundwater at the Site with concentrations of PCBs that exceed applicable screening criteria at depths below the previously defined extent of contamination. An additional PCB investigation is required to delineate the vertical extent of PCBs in subsurface soils (greater than 25 feet bgs) in the previously identified source areas, evaluate the potential impact of PCBs at multiple depths in downgradient groundwater, and determine if there is evidence of a carrier fluid (organic solvent)..

The PCB investigation will determine the following:

- Vertical extent of PCB-contaminated soils in the source areas;
- Whether PCBs have impacted downgradient groundwater beyond the Site boundary, and if so, determine the vertical and horizontal extent of these PCBs;
- Whether organics (fuel-related or chlorinated solvents) are present at sufficient concentrations to act as a carrier fluid that would promote PCB migration.

11.2 INFORMATION INPUTS

Soil data are needed for PCBs and will be collected via on-site PCB field screening with off-site laboratory confirmatory analysis. Soil data is also needed for VOCs, Total Organic Carbon (TOC), Total Petroleum Hydrocarbons (TPH) Diesel Range Organics (DRO), and TPH Gasoline Range Organics (GRO) to determine if there is evidence of a carrier fluid (organic solvent). Groundwater data are also needed for off-site laboratory analysis of PCBs and VOCs.

The following inputs are needed to attain project objectives:

1. Previously Collected Data: This data was used to delineate the study area that will be investigated for the deeper vertical extent of PCBs in subsurface soils at Site 1.

2. Field Screening Data:
    a. Field PCB screening data are needed to determine where PCBs have impacted soils at depths below 25 feet bgs. Laboratory confirmatory samples will be collected for PCBs and the Field PCB data will be correlated to laboratory data (see Worksheet No. 37 for correlation method).

    b. Visual observations and photoionization detector (PID) readings will be used at each soil boring and soil interval to help identify contamination, and to assist in the selection of

vertical sampling depths at soil locations. Visual and PID data inputs are considered qualitative because they do not provide a quantified value. This qualitative data is of sufficient quality to facilitate consideration of actual sample locations selected for laboratory analysis.

2. Chemical Data: Subsurface soil and groundwater data are needed for PCBs. These analytical fractions are included on the basis of the CSM to cover the known contaminants of concern that may have impacted soil and groundwater. Soil samples will also be collected at select locations and analyzed for VOCs, TOC, TPH-DRO, and TPH-GRO to determine if significant concentrations of petroleum hydrocarbons, and/or site-related VOCs are present that could potentially act as a carrier fluid and affect PCB migration. See Worksheet No. 15 for the full list of analytes. Sample collection procedures for various media are listed in Worksheet Nos. 14, 18, and 21.

   Hazardous waste analyses will be conducted on IDW soils in accordance with the Toxicity Characteristic Leaching Procedure (TCLP) requirements in 40 CFR 261. See Worksheet No. 15 for the full list of analytes. The data will be used to determine proper disposal procedures for IDW.

3. Physical Data: Groundwater stabilization parameters (collected in accordance with Standard Operating Procedure [SOP] SA-1.2) will include pH, specific conductance, turbidity, temperature, and dissolved oxygen and will be measured during groundwater sampling to ensure representative samples are collected. Groundwater levels will also be measured at site monitoring wells prior to the collection of samples to determine groundwater depths and flow direction. The physical data will help characterize groundwater at the Site.

4. PSLs: PSLs are needed to determine if the concentrations of PCBs exceed the regulatory criteria and if site-related VOCs and other organics are present at significant concentrations that could affect the migration of PCBs. Subsurface soil and groundwater data will be compared to the following criteria:

   a. A Field PCB (total organic chlorine) PSL of 10 mg/kg will be used to determine selection of subsurface soils samples for laboratory analysis. See worksheet No. 17 for details on laboratory sample selection.

   b. Subsurface soil data will be screened against the NYSDEC Determination of Soil Cleanup Objectives and Cleanup Levels (TAGM 4046) (January 1994). The NYSDEC Soil Cleanup Objectives are available online at: http://www.dec.ny.gov/regulations/2612.html.

Secondary Criteria - Subsurface soil data will also be compared against the NYSDEC 6 NYCRR Part 375 Remedial Soil Cleanup Objectives for Restricted Residential Use Table 375-6.8(b) (December 2006). These secondary criteria will be used for qualitative purposes only and will not be used in decision-making. The NYSDEC Soil Cleanup Objectives are available online at: http://www.dec.ny.gov/docs/remediation_hudson_pdf/part375.pdf.

c. Groundwater data for PCBs will be screened against the NYSDOH Part 5; Subpart 5-1 Public Water Systems MCLs (updated May 2009). The MCLs are available online at: http://www.health.state.ny.us/environmental/water/drinking/annual_water_quality_report/table.htm

Since there is a history of chlorinated and non-chlorinated organic use at Site 1 and there is evidence that PCBs may have migrated to and below groundwater and potentially impacted groundwater above MCLs, groundwater data for site-related organics will be compared to the one percent (1%) solubility of non-aqueous phase liquid/dense non-aqueous phase liquid (NAPL/DNAPL) values calculated for each site-related contaminant as presented in Worksheet No.15. Comparing the results to the 1% solubility values should to determine whether these organics are present in high enough concentrations to potentially to act as a carrier fluid and affect the migration of PCBs.

Secondary Criteria - Groundwater data for VOCs will also be compared against the NYSDOH Part 5, Subpart 5-1 Public Water Systems MCLs as presented above for PCBs. These secondary criteria will be used for qualitative purposes only and will not be used in decision-making.

Worksheet No. 15 lists the PSLs as well as TriMatrix's reported quantitation limits (QLs) and statistically-derived method detection limits (MDLs). TriMatrix is able to achieve QLs that are low enough to measure constituent concentrations that are less than the PSLs.

Some soil VOC analytes (three Freon compounds and cis- and trans-1,2-dichloroethene) do not have associated PSLs (the list of VOCs that will be analyzed is presented in Worksheet No. 15). However, these analytes are included in the VOC list because they are site-related contaminants that have been identified from historical data. If an analyte is detected in a matrix and has no associated PSL, then a qualitative discussion of the significance of that analyte will be included in the PCB Investigation Report.

11.3 STUDY BOUNDARIES

Soil

The population of interest is subsurface soil within the known source areas at Site 1. The horizontal boundary for this soil investigation is limited to the three source areas shown on **Figure 6** which represent the northern, western, and eastern boundaries. The horizontal boundary to the south is extended from the source areas to proposed soil boring locations near the warehouse buildings, as shown on **Figure 13**.

To establish the vertical boundaries, chemical data (PCB by field screening) and lithological data must be collected within the source area and below 25 feet bgs. Subsurface soil data represents the subsurface soil population contained within the boundaries. The subsurface soil population described above represents the subsurface soil decision unit.

Groundwater

Horizontal boundaries for groundwater include upgradient, side gradient, and downgradient groundwater from the source areas as described in the soil investigation boundaries above. The upgradient, or northern boundary for groundwater, would include the area north of the source areas and south of the former recharge basins shown on **Figure 8**. The eastern and southern boundaries are limited to the former NWIRP property boundaries as shown on **Figure 2**. The western boundary for groundwater is defined by the isolated source area observed south of Plant 3 that is approximately 400 feet west of the southeast corner of Plant 3 (**Figure 6**). The vertical boundary for groundwater is the top of the water table which is approximately 52 feet bgs, to the lowest depth of observed PCB-contaminated soils and groundwater. Data will be collected from several groundwater populations of interest, which include potentially contaminated groundwater downgradient of the identified source areas and groundwater that is both upgradient and side gradient of the source areas.

11.4 ANALYTIC APPROACH

Analytical results from the PCB field screening of subsurface soils will be compared to the PSL. Once laboratory analytical data is received, the PCB analytical results will also be compared to PSL and then the total PCB results will be correlated to the field screening data to determine usability of the field screening data for decision-making. Specific procedures for correlating the data are outlined in Worksheet No. 37. PSLs are detailed in Worksheet No. 15.

<u>Soil Boring Investigation</u>

Data from the field PCB screening analysis will be used to make project decisions as described below. The following decision rules apply to the soil decision unit:

Decision Rule 1a:

During the advancement of soil borings, subsurface soil samples will be screened and selected for laboratory analysis of VOCs, TOC, TPH-DRO, and TPH-GRO as follows:

If visual evidence of contamination (i.e., staining) or elevated PID readings are observed while screening the soil cores, then a soil sample will be collected from that soil interval. A maximum of 4 samples for

source area borings and 3 samples from downgradient borings will be selected for laboratory analysis of VOCs, TOC, TPH-DRO, and TPH-GRO.

Decision Rule 1b:

After each soil boring is complete and all subsurface soil samples have been field screened for PCBs, samples will be selected for laboratory confirmation sampling as follows:

If a soil sample was collected for VOC, TOC, and TPH-DRO, and TPH-GRO analysis based on Decision Rule 1a, then a confirmatory sample will be selected from the same soil interval for PCB laboratory analysis. The confirmatory soil samples will be selected with a goal of co-locating a minimum of two soil samples from each soil boring for laboratory analysis of PCBs, VOCs, TOC, and TPH-DRO, and TPH-GRO.

If the PCB field screening of a subsurface soil sample indicates a concentration greater than or equal to the PSL, then the subsurface soil sample will be selected for laboratory analysis of PCBs based on the following criteria:

- Up to four subsurface soil samples in each source area soil boring will be selected for laboratory confirmatory analysis from sample intervals indicating screening concentrations greater than the PSL.
- Up to three subsurface soil samples in each downgradient soil boring will be selected for laboratory confirmatory analysis from sample intervals indicating screening concentrations greater than the PSL.
- The confirmatory samples will be selected from intervals showing the highest PCB screening concentrations above the PSL; however,
- The vertical distribution of confirmatory samples, based on the range of concentrations observed from field screening above the PSL, will be considered during sample selection for laboratory analysis.

If the PCB field screening of a subsurface soil sample indicates detected concentrations that are less than the PSL, then the subsurface soil sample will be selected for laboratory analysis of PCBs based on the following criteria:

- A minimum of two subsurface soil samples in each source area soil boring will be selected for laboratory confirmatory analysis from sample intervals indicating screening concentrations less than the PSL.
- A minimum of one subsurface soil sample in each downgradient soil boring will be selected for laboratory confirmatory analysis from sample intervals indicating screening concentrations less than the PSL.

- These confirmatory soil samples will be biased towards the deeper intervals showing concentrations below the PSL in the soil boring to confirm the vertical extent of PCBs in subsurface soils.

For additional guidance, please refer to the decision tree in Worksheet No. 17.

Decision Rule 1c:
Based on soil PCB concentrations observed in field screening samples greater than or equal to the PSLs, and lithological information from the soil borings, the field team will determine target depths for groundwater grab sampling in downgradient soil boring locations. The target depths will be selected based on the following criteria:

- Minimum or maximum depth of PCB-contaminated soils determined from field screening in the source areas
- Soil intervals that are suspected to contain elevated levels of VOCs based on field screening data
- Depth of clay units observed below the source areas that could affect any vertical migration of contaminants

Groundwater Investigation

Analytical results from groundwater grab samples will be used to determine placement of permanent monitoring well clusters. Permanent monitoring well sample data will be used to determine whether groundwater described in Section 11.3 has been impacted by PCBs.

The following decision rules apply to the groundwater decision units:

Decision Rule 2a: If a PCB concentration in a groundwater grab sample is greater than or equal to the PSL, then a permanent monitoring well cluster will be placed further downgradient of that associated groundwater grab sample within the boundary of the former NWIRP property. If the PCB concentration in a groundwater grab sample is less than the PSL, then that location assumes capture of the PCB-contaminated groundwater and a permanent monitoring well cluster will be installed adjacent to the groundwater grab location.

Decision Rule 3: If a PCB concentration in a groundwater sample collected from a permanent monitoring well is greater than the respective PSL, then the Project Team will convene to determine the significance of each exceedance and the appropriate future action needed to address contaminated groundwater at Site 1 (e.g., another sampling round or additional downgradient wells). If target analyte

concentrations in permanent monitoring well samples are less than or equal to each respective PSL, then a proposed monitoring plan will be developed for groundwater at Site 1.

11.5 PERFORMANCE CRITERIA

Because this investigation depends on biased sampling, probability limits for false positive and false negative decision errors were not established for the laboratory analytical data**.** Direct comparisons of measured concentrations to PSLs are being used. Sample locations were selected to target the source areas delineated in previous investigations at the site. This biased selection of sample locations in contaminated areas does not support the use of quantitative statistics to evaluate decision performance as specified in the USEPA DQO guidance documents (EPA 2002a, 2002b, and 2005).

The following decision errors apply to the field PCB screening results, which may include matrix interferences:

If a false positive is detected at greater than or equal to the PSL, this may lead to a conclusion that the vertical extent of PCB contamination is deeper or more extensive than the actual extent of contamination. If a false negative is reported, this may not determine the actual vertical extent of PCBs at greater than the PSL. Laboratory confirmation samples including screening results greater than and less than the PSL will be used to verify field PCB screening results.

The following decision errors apply to the groundwater results:

If a false positive is detected for PCBs at greater than the PSL, this could lead to a decision to install a new monitoring well further downgradient than necessary. If a false negative for PCBs is determined less than or equal to the PSL, then the new well location may not capture the extent of the PCB-contaminated groundwater.

11.6 PLANS FOR OBTAINING DATA

See Worksheet No. 17 for a detailed sampling design.

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

(UFP-QAPP Manual Section 2.6.2)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPCs)	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Trip Blank	VOCs only	One per cooler of VOC samples shipped to laboratory	Accuracy/Bias/ Contamination	No analytes > ½ QL, except common lab contaminants, which must be < QL.	S & A
Field Blank	VOCs and PCBs	One per source[(2)] of rinse water or decontamination water	Accuracy/Bias/ Contamination	No analytes > ½ QL, except common lab contaminants, which must be < QL.	S & A
Equipment Rinsate Blank	All analytical groups	One per 20 samples per sampling equipment[1]	Accuracy/Bias/ Contamination	No analytes > ½ QL, except common lab contaminants, which must be < QL.	S & A
Field Duplicate	Field PCB screening	One per 10 field samples	Precision	Relative Percent Difference (RPD) ≤ 50.	S & A (field analysis only)
Field Duplicate (Groundwater)	All analytical groups	One per 10 field samples	Precision	Values > 5X QL: RPD ≤ 30. Values < 5X QL: Absolute difference of two values ≤ 2X QL.	S & A (laboratory analysis only)
Field Duplicate (Soil)	All analytical groups	One per 10 field samples	Precision	Values > 5X QL: RPD ≤ 50. Values < 5X QL: Absolute difference of two values ≤ 4X QL.	S & A (laboratory analysis only)
Cooler Temperature Indicator	All analytical groups	One per cooler	Representativeness	Temperature between 2 and 6 degrees Celsius (4 ± 2 °C).	S

1 – Equipment rinsate blanks will be collected if non-dedicated submersible pumps or non-disposable equipment are used.
2 – Source water and storage tanks will be provided by the drilling subcontractor, therefore, field blanks are necessary to ensure that the decontamination water is free of target analytes.

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

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Phase 1 RI - confirms the presence of solvents, metals, and PCBs	Halliburton NUS, 1992 (see references for more details)	Halliburton NUS, 1992 Soil Gas- VOCs Soil and Sediment- VOCs, PCBs, Pesticides, and metals Temporary and Permanent Monitoring Wells- VOCs	Will be used with data from this investigation to define extent of PCBs in subsurface soils.	Data is 18 years old and only provides shallow delineation of PCBs
Phase 2 RI – characterizes nature and extent of contamination	Halliburton NUS, 1993 (see references for more details)	Halliburton NUS, 1993 Soils- PCBs Groundwater- VOCs	Will be used with data from this investigation to define extent of PCBs in subsurface soils.	Data is 17 years old and only provides shallow delineation of PCBs
Pre-excavation soil sampling results	Foster Wheeler, 1995 (see references for more details)	Foster Wheeler, 1995 Soils- VOCs, PCBs, and arsenic	Will be used with data from this investigation to define extent of PCBs in subsurface soils.	Provides only limited data at depth for PCBs
Pre-excavation soil sampling results	Foster Wheeler, 1996 (see references for more details)	Foster Wheeler, 1996 Soils- VOCs, metals, and PCBs	Will be used with data from this investigation to define extent of PCBs in subsurface soils.	Provides only limited data at depth for PCBs
Remediation of Former Drywells 20-08 and 34-07	H2M Group, 2003 (see references for more details)	H2M Group, 2003 Soil- PCBs	Will be used with data from this investigation to define vertical extent of PCBs in subsurface soils.	Limited vertical delineation and remediation of PCBs
Groundwater Sampling	Tetra Tech, 2008b (see references for more details)	Tetra Tech, 2008b Groundwater- PCBs	Will be compared with data from this investigation to determine potential impact of PCBs in groundwater	Data from existing monitoring wells is suspect due to installation technique

SAP Worksheet No. 14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASK PLAN

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

- Mobilization/Demobilization
- Utility Clearance
- Monitoring Equipment Calibration
- Soil Borings and Subsurface Soil Sampling
- Groundwater Grab Sampling
- Monitoring Well Installation
- Monitoring Well Sampling
- Water Level Measurements
- IDW Management
- Surveying
- Field Decontamination Procedures
- Field Documentation Procedures
- Sample Custody and Shipment Tasks

Additional project-related tasks include:

- Analytical Tasks
- Data Management
- Assessment and Oversight
- Data Review
- Project Reports

FIELD TASKS

Mobilization/Demobilization

Mobilization shall consist of the delivery of all equipment, materials, and supplies to the Site, the complete assembly in satisfactory working order of all such equipment at the Site, and the satisfactory storage at the Site of all such materials and supplies. Tetra Tech will coordinate with the facility to identify locations for the storage of equipment and supplies. Site-specific Health and Safety Training will be provided to all Tetra Tech subcontractors as part of the Site mobilization.

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

Utility Clearance

Prior to the commencement of any intrusive activities, Tetra Tech will coordinate with the facility to identify and mark-out utilities that may be present within the proposed drilling areas. Subsurface utilities will also be cleared by the drilling subcontractor by notifying the Dig Safe One-Call utility clearing service. In the event that the Navy cannot reliably clear the facility for subsurface utilities, it may become necessary to procure and mobilize a subcontractor to clear the drilling locations for subsurface utilities before any intrusive work can be performed. See SOP HS-1.0 (Appendix A) for conducting subsurface soil investigations for further information.

Monitoring Equipment Calibration

These procedures are described in Worksheet No. 22.

Soil Borings and Subsurface Soil Sampling

Subsurface soil samples (>25 feet bgs) will be obtained from soil borings advanced using Rotosonic drilling methods. The proposed soil boring locations are presented on **Figure 13**. With a Rotosonic rig, continuous soil cores will be obtained to the target depth at each location by advancing a core barrel assembly (10-foot or 20-foot sections) into the subsurface to a depth of 250 feet. The core barrel assembly will be withdrawn and the soils will be screened, described, and sampled. This process will be repeated until the target depth of 250 feet is reached.

The soil will be described by the Tetra Tech Geologist and a boring log will be constructed. Soil cores will be screened with a PID for evidence of potential contamination. Any visual signs of potential contamination (such as soil staining) will be noted and samples will be collected for VOCs, TOC, TPH-GRO and TPH-DRO.

Soil samples will be collected as outlined in Worksheet No. 17. Subsurface soil samples will be analyzed on-site with the Dexsil L2000DX Analyzer. The Dexsil PCB test kit analysis will be performed as outlined in the Manufacturer's Instruction in Appendix C. Samples will be selected for laboratory confirmation based upon field test kit results as described in Worksheet No. 11 and as detailed in Worksheet No. 17.

Soil VOC, TOC, TPH-DRO and TPH-GRO samples will also be obtained from the soil cores along with field and laboratory QC samples.

Soil boring installation with Rotosonic drilling equipment is discussed in Tetra Tech SOP GH-1.3. Soil sampling procedures are discussed in Tetra Tech SOP SA-1.3, soil logging procedures are documented in Tetra Tech SOP GH-1.5, and the use of the PID is discussed in the manufacturer's instructions. The Tetra Tech SOPs are included in Appendix A.

Groundwater Grab Sampling

Groundwater grab samples will be collected from the three downgradient boring locations. Three groundwater grab samples from varying depths are planned at each boring location and actual depths will be determined from analytical and lithological data collected from the source area borings. Groundwater grab samples will be collected via a borehole pumping system using a stainless steel screen, K-packer, and submersible pump. A submersible pump or bladder pump will be employed to obtain groundwater samples using low-flow sampling techniques. Groundwater grab samples will be analyzed for PCBs and VOCs.

Monitoring Well Installation

Four monitoring well clusters will be installed to evaluate groundwater conditions in the study area. One monitoring well cluster will be installed in the hydraulically upgradient (north) portion of the study area to evaluate the background or ambient water quality conditions, and three monitoring well clusters will be installed hydraulically downgradient (south) of the Site to identify and evaluate any groundwater impacts caused by historical Site activities. The proposed monitoring well locations are illustrated on **Figure 13**.

The monitoring wells will be constructed to evaluate the water quality conditions downgradient of the known source areas at Site 1. It is anticipated that three monitoring wells will be installed at each downgradient monitoring well cluster. However, soil boring data will be evaluated and the actual number, placement, and depths of the monitoring wells will be determined by the Project Team prior to well installation. Depending on the depths and actual number of wells needed at each location, monitoring wells may be nested in the same borehole.

Soil borings will be drilled at the proposed monitoring well locations to confirm the subsurface lithology and depths to ensure proper depths for well installation.. The soil cores will be screened visually and with a PID in accordance with the manufacturer's instructions. The boring information will be recorded in accordance with Tetra Tech SOP SA-6.3. The monitoring wells will be installed using Rotosonic drilling

methods.

Monitoring well installation procedures are discussed in Tetra Tech SOP GH-2.8 and each of the new monitoring wells will be developed in accordance with this SOP. All applicable Tetra Tech SOPs are included in Appendix A.

Groundwater Sampling

Groundwater sampling will be conducted at each monitoring well using Low Flow Sampling Procedures, in accordance with Tetra Tech SOP SA-1.1 (Appendix A).

A 2-inch diameter, variable speed submersible pump and controller (e.g., Grundfos) or bladder pump will be used for groundwater sample purging and collection activities, in combination with a continuous flow-through cell suitable for taking water quality measurements. Turbidity measurements will be made using a separate field turbidity meter that is specifically designed to measure turbidity only. The turbidity sample will be collected downstream of the flow-through cell.

Water Level Measurements

Synoptic groundwater elevations will be measured in each monitoring well during each of the two rounds of sampling to generate groundwater elevation contour maps and provide information on groundwater flow patterns and gradients. Water-level measurements will be completed within the shortest time possible on the same day, and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or north side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement and between each monitoring well.

Investigation-Derived Waste Management

Soils will be generated during the installation of the soil borings and monitoring wells. The soil IDW consists of the excess soil cuttings from the soil borings that were not collected for laboratory analyses, and the soils produced during the drilling of the boreholes for monitoring well installations. The soils will be collected and placed in roll-off boxes or 55-gallon drums for waste characterization sampling and analysis. The soil cuttings from the source area borings will be separated from downgradient soil borings, given the potential for PCB-contaminated soils in the source area borings. Based upon the results of the waste characterization, the soils will be transported and appropriately disposed off-site at an approved disposal facility by the IDW subcontractor.

Water will be generated during groundwater grab sampling, well development, sampling of the wells, and during decontamination procedures. All aqueous IDW will be containerized pending waste characterization analyses. Based on the results of the waste characterization, the water will be either transported and appropriately disposed offsite at an approved disposal facility by the IDW subcontractor or, if permission is obtained from Nassau County, discharged into the sanitary sewer.

Surveying

A surveyor licensed in the state of New York will be subcontracted by Tetra Tech to survey the horizontal location and vertical elevation of each of the soil locations and new monitoring wells. The horizontal measurements shall be accurate to 0.1 foot. The vertical elevation measurements shall be accurate to 0.01 foot at each location, and for the top-of-riser at each monitoring well. Each of the locations will be surveyed relative to the coordinates of established Site benchmarks or the nearest United States Geological Survey (USGS) benchmark.

Field Decontamination Procedure

Sample containers will be provided from TriMatrix that are certified-clean (I-Chem 300 or equivalent). Decontamination of reusable sampling equipment (e.g., submersible pumps) will be conducted prior to and between sampling at each location. Drilling equipment, specifically drilling rods, augers, core barrels, and other downhole tools will require decontamination between borings and monitoring well locations. All soil sampling and groundwater sampling equipment or devices used during drilling will be decontaminated between sample locations. Decontamination of equipment will be conducted in accordance with Tetra Tech SOP SA-7.1 (Appendix A).

Field Documentation Procedures

Field documentation will be performed in accordance with SOP SA-6.3 (Appendix A).

A summary of all field activities will be properly recorded in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

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

- Name of the person to whom the logbook is assigned.

- Project name.
- Project start date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of Site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information, including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.

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

Boring logs, sampling forms, and other field forms will be used to document field activities according to the SOPs provided in Appendix A.

Sample Custody and Shipment Tasks

Data management and sample tracking tasks are described in Worksheet No. 27 and in Tetra Tech SOP CT-05 (Appendix A).

ADDITIONAL PROJECT-RELATED TASKS

Analytical Tasks

Chemical analysis for VOCs, PCBs, TOC, TPH-DRO, TPH-GRO, and hazardous waste characteristics will be performed by TriMatrix. TriMatrix is currently a Department of Defense (DoD) Environmental Laboratory Approval Program (ELAP) and NYSDOH ELAP approved laboratory. The DoD and NYSDOH ELAP accreditation documentation is included in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 30. TriMatrix will meet the PALs for the target analytes specified in Worksheet No. 15, with the exceptions noted, and will perform the chemical analyses following laboratory-specific SOPs (Worksheet Nos. 19 and 23) that were developed based on the methods listed in Worksheet Nos. 19 and 30. Copies of TriMatrix SOPs that will be used on this project are included in Appendix B.

Data Management

Project documentation and records:

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

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 these sites 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 on the basis of date and activity.

The data-handling procedures to be followed by TriMatrix will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

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

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

- Data Storage, Archiving, and Retrieval – The data packages received from TriMatrix are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs are submitted by the Tetra Tech 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 are stored by Tetra Tech and ultimately sent to NAVFAC.

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

Assessment and Oversight

Refer to Worksheet No. 32 for assessment findings and Corrective Actions and to Worksheet No. 33 for QA management reports.

Data Review

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

Project Report

A Draft PCB Investigation Report will be prepared and submitted to the Navy and NYSDEC for review. The report will include a summary of the work performed in the approved UFP-SAP, field modifications as documented by the Tetra Tech FOL, a summary of the analytical results, and conclusions and/or recommendations for further corrective action at Site 1.

Tetra Tech will respond to comments received on the draft report. The final version of the report will be submitted in hardcopy and electronic format to the project stakeholders.

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

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil
Analytical Group: VOCs

Analyte	CAS Number	Project Screening Level (mg/kg)	Project Screening Level Reference [1]	Project Quantitation Limit Goal [2] (mg/kg)	TriMatrix	
					QLs (mg/kg)	MDLs (mg/kg)
1,1,1-Trichloroethane	71-55-6	100	NYSDEC SCO RR	33	0.05	0.016
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	NA	NA	NA	0.25	0.0083
1,1-Dichloroethane	75-34-3	19	NYSDEC SCO RR	6.3	0.05	0.012
1,1-Dichloroethene	75-35-4	100	NYSDEC SCO RR	33	0.05	0.014
1,2-Dichloroethane	107-06-2	2.3	NYSDEC SCO RR	0.77	0.05	0.0086
2-Butanone	78-93-3	100	NYSDEC SCO RR	33	2.5	0.025
Acetone	67-64-1	100	NYSDEC SCO RR	33	0.75	0.081
Benzene	71-43-2	2.9	NYSDEC SCO RR	1	0.05	0.014
cis-1,2-Dichloroethene	156-59-2	NA	NA	NA	0.05	0.011
Dichlorodifluoromethane	75-71-8	NA	NA	NA	0.05	0.0094
Ethylbenzene	100-41-4	30	NYSDEC SCO RR	10	0.05	0.015
m,p-Xylene	179601-23-1	100	NYSDEC SCO RR	33	0.1	0.029
Methylene chloride	75-09-2	51	NYSDEC SCO RR	17	0.25	0.01
o-Xylene	95-47-6	100	NYSDEC SCO RR	33	0.05	0.014
Tetrachloroethene	127-18-4	5.5	NYSDEC SCO RR	1.8	0.05	0.019
Toluene	108-88-3	100	NYSDEC SCO RR	33	0.05	0.015
trans-1,2-Dichloroethene	156-60-5	NA	NA	NA	0.05	0.017
Trichloroethene	79-01-6	10	NYSDEC SCO RR	3.3	0.05	0.018
Trichlorofluoromethane	75-69-4	NA	NA	NA	0.05	0.016
Vinyl chloride	75-01-4	0.21	NYSDEC SCO RR	0.07	0.05	0.012

[1] Soil screening references: NYSDEC Soil Cleanup Objective (SCO) for Restricted Residential (RR) Use, December 2006.
[2] Project Quantitation Limit Goals were set 3X lower than the PSL.

Matrix: Soil
Analytical Group: Field Total PCB Screening

Analyte	CAS Number	Project Screening Level (mg/kg)	Project Screening Level Reference[1]	Project Quantitation Limit Goal [2] (mg/kg)	Dexsil L2000DX
					Detection Range (mg/kg)
Total Organic Chlorine	NA	10	NYSDEC SCO SB	2	2-2000

[1] Soil screening references: NYSDEC Soil Cleanup Objective (SCO) for Subsurface Soil (SB), TAGM 4046 Table 3 http://www.dec.ny.gov/regulations/30582.html.

Matrix: Soil
Analytical Group: PCBs

Analyte	CAS Number	Project Screening Level (mg/kg)	Project Screening Level Reference[1]	Project Quantitation Limit Goal [2] (mg/kg)	TriMatrix	
					QLs (mg/kg)	MDLs (mg/kg)
Aroclor-1016	12674-11-2	10	NYSDEC SCO RR	3.3	0.33	0.0042
Aroclor-1221	11104-28-2	10	NYSDEC SCO RR	3.3	0.33	0.0078
Aroclor-1232	11141-16-5	10	NYSDEC SCO RR	3.3	0.33	0.0045
Aroclor-1242	53469-21-9	10	NYSDEC SCO RR	3.3	0.33	0.0046
Aroclor-1248	12672-29-6	10	NYSDEC SCO RR	3.3	0.33	0.0065
Aroclor-1254	11097-69-1	10	NYSDEC SCO RR	3.3	0.33	0.0059
Aroclor-1260	11096-82-5	10	NYSDEC SCO RR	3.3	0.33	0.005
Aroclor-1262	37324-23-5	10	NYSDEC SCO RR	3.3	0.33	0.0052
Aroclor-1268	11100-14-4	10	NYSDEC SCO RR	3.3	0.33	0.0065

[1] Soil screening references: NYSDEC Soil Cleanup Objective (SCO) for Restricted Residential (RR) Use, December 2006.
[2] Project Quantitation Limit Goals were set 3X lower than the PSL.

Matrix: Soil
Analytical Group: TPH-DRO, TPH-GRO, and TOC

Analyte	CAS Number	Project Screening Level	Project Screening Level Reference[1]	Project Quantitation Limit Goal	TriMatrix	
					QLs	MDLs
TPH-DRO (mg/kg)	NA	NA	NA	NA	6.7	1.3
TPH-GRO (mg/kg)	NA	NA	NA	NA	5	2.8
TOC (%)	NA	NA	NA	NA	0.0062	0.20

[1] No soil screening value is available for these parameters. These parameters will not be evaluated for exceedance of PSLs, but used to aid evaluation of soils for remedy selection.

Matrix: Groundwater
Analytical Group: VOCs

Analyte	CAS Number	Project Screening Level (µg/L)	Project Screening Level Reference [1]	Project Quantitation Limit Goal [2] (µg/L)	TriMatrix	
					QLs (µg/L)	MDLs (µg/L)
1,1,1-Trichloroethane	71-55-6	12,900	1% Solubility	4,300	1	0.12
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1,700	1% Solubility	567	1	0.16
1,1-Dichloroethane	75-34-3	50,400	1% Solubility	16,800	1	0.092
1,1-Dichloroethene	75-35-4	24,200	1% Solubility	8,070	1	0.17
1,2-Dichloroethane	107-06-2	86,000	1% Solubility	28,700	1	0.096
2-Butanone	78-93-3	2,230,000	1% Solubility	743,000	5	0.27
Acetone	67-64-1	10,000,000	1% Solubility	3,330,000	10	2.5
Benzene	71-43-2	17,900	1% Solubility	5,970	1	0.088
cis-1,2-Dichloroethene	156-59-2	64,100	1% Solubility	21,400	1	0.12
Dichlorodifluoromethane	75-71-8	2,800	1% Solubility	933	1	0.13
Ethylbenzene	100-41-4	1,690	1% Solubility	563	1	0.1
m,p-Xylene	179601-23-1	1,610	1% Solubility	537	3	0.2
Methylene chloride	75-09-2	130,000	1% Solubility	43,300	5	0.081
o-Xylene	95-47-6	1,780	1% Solubility	593	1.7	0.2
Tetrachloroethene	127-18-4	2,060	1% Solubility	687	1	0.1
Toluene	108-88-3	5,260	1% Solubility	1,750	1	0.22
trans-1,2-Dichloroethene	156-60-5	45,200	1% Solubility	15,100	1	0.086
Trichloroethene	79-01-6	12,800	1% Solubility	4,270	1	0.13
Trichlorofluoromethane	75-69-4	11,000	1% Solubility	3,670	1	0.11
Vinyl chloride	75-01-4	88,000	1% Solubility	29,300	1	0.062

[1] Groundwater screening references: 1% Solubility of NAPL/DNAPL derived from aqueous solubility (see Appendix D for calculation worksheet).
[2] Project Quantitation Limit Goals were set 3X lower than the PSL.

Matrix: Groundwater
Analytical Group: PCBs

Analyte	CAS Number	Project Screening Level (µg/L)	Project Screening Level Reference[1]	Project Quantitation Limit Goal [2] (µg/L)	TriMatrix	
					QLs (µg/L)	MDLs (µg/L)
Aroclor-1016	12674-11-2	0.5	NYSDOH MCL	0.17	0.2	0.025
Aroclor-1221	11104-28-2	0.5	NYSDOH MCL	0.17	0.2	0.029
Aroclor-1232	11141-16-5	0.5	NYSDOH MCL	0.17	0.2	0.032
Aroclor-1242	53469-21-9	0.5	NYSDOH MCL	0.17	0.2	0.04
Aroclor-1248	12672-29-6	0.5	NYSDOH MCL	0.17	0.2	0.03
Aroclor-1254	11097-69-1	0.5	NYSDOH MCL	0.17	0.2	0.033
Aroclor-1260	11096-82-5	0.5	NYSDOH MCL	0.17	0.2	0.026
Aroclor-1262	37324-23-5	0.5	NYSDOH MCL	0.17	0.2	0.033
Aroclor-1268	11100-14-4	0.5	NYSDOH MCL	0.17	0.2	0.026

[1] Groundwater screening references: NYSDOH Maximum Contaminant Level (MCL), May 2009.
[2] Project Quantitation Limit Goals were set 3X lower than the PSL.

SAP Worksheet No. 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		
Project Planning (UFP-SAP)	Tetra Tech	11/10/2009	3/31/2010	UFP-SAP for PCB Investigation	3/31/2010 (draft) 5/21/2010 (final)
Soil Borings and Soil Sampling	Tetra Tech	7/12/2010	8/20/2010	NA	NA
Monitoring Well Installation and Groundwater Sampling Round 1	Tetra Tech	10/4/2010	11/30/2010	NA	NA
Groundwater Sampling Round 2	Tetra Tech	2/28/11	3/11/11	NA	NA
Report	Tetra Tech	12/1/2010	4/22/2011	PCB Investigation Report	3/1/2011 (draft) 4/22/2011 (final)

SAP Worksheet No. 17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The PCB Investigation will include three phases of fieldwork. The first phase will include advancement of six soil borings in the source areas and three downgradient soil borings. The second phase will include monitoring well installation and groundwater sampling. The third phase will include a second round of groundwater sampling at Site 1 monitoring wells.

Soil Boring Investigation

Subsurface soil samples will be collected at Site 1 to evaluate and determine the vertical extent of PCB-contaminated soils in the source areas. Soil boring locations will be placed within the known source areas as presented on **Figure 13**. This biased sampling approach was used to target the known areas where historical sampling had not determined the vertical extent of PCBs. On-site field PCB screening will be used to delineate PCB-impacted subsurface soil and to aid in selection of soil samples for laboratory confirmation analyses. The sampling design and rationale are described below and were selected to fulfill the DQOs presented in Worksheet No. 10.

A table representing the information contained in this worksheet including depths, analytes, and sample nomenclature are presented in Worksheet No. 18. Sampling procedures are described in Worksheet No. 14 and listed in Worksheet No. 21.

Subsurface Soil Sampling and Field PCB Screening

A total of six soil borings will be placed in the known source areas and three soil borings will be located downgradient of Site 1 as presented on **Figure 13**. Each of these nine soil borings will be advanced to 250 feet bgs. For the source area borings, field PCB test kit analysis will be performed on subsurface soil samples collected from each five-foot interval, starting at 25 feet bgs down to the potential clay unit observed at approximately 100 feet bgs, or to 120 feet if the clay is not observed in the soil cores. From 120 feet bgs to 250 feet bgs, composite soil samples will be collected for each 10-foot interval. Composite soil samples will be collected by homogenizing the soil from each 10-foot interval. Therefore, approximately 19 subsurface soil samples and 13 10-foot composite samples will be collected at each source area boring location and analyzed on-site for PCBs.

For the three downgradient borings, since it is suspected that PCB-impacted soils in these borings will be minimal given the distance from known source areas, field PCB test kit analysis will be performed on composite samples collected from 10-foot intervals starting from the ground surface down to the potential clay unit at approximately 100 feet bgs, or to 120 feet if the clay is not observed in the soil cores. If PCBs are not detected in the subsurface soil samples collected from 80-100 feet bgs or just above the clay unit, then deeper soil samples will not be screened for PCBs unless visible evidence of contamination is

observed by the on-site Tetra Tech Field Geologist. Therefore, approximately 10-12 10-foot composite samples will be collected at each downgradient boring location and analyzed on-site for PCBs.

Visual observations (e.g., staining) and PID readings will be used to select locations for PCB soil samples by identifying potential contamination and will be used to determine the location of discrete VOC, TOC, TPH-DRO, and TPH-GRO samples during the advancement of soil borings. Visual and PID data inputs are considered qualitative because they do not provide a quantified value. This qualitative data is of sufficient quality to facilitate selection of sample locations and/or depths relative to other data in the same area. Calibration and operation of the PID will be conducted in accordance with the manufacturer's instructions.

If visual evidence of contamination or elevated PID readings are observed while screening the soil cores, then a soil sample will be collected from that soil interval for laboratory analysis. A maximum of 4 samples from source area borings and 3 samples from downgradient borings will be selected for laboratory analysis of VOCs, TOC, TPH-DRO, and TPH-GRO. If no visual evidence of contamination is observed or if PID readings do not indicate elevated levels of VOCs, then discrete samples will be selected based on observed changes in lithology (just above potential confining units) and biased towards selecting samples below the top of the groundwater table.

Soil Sample Selection for Laboratory Confirmation

Six samples from each source area soil boring will be selected for off-site laboratory confirmation PCB analysis and four samples from each source area boring will also be selected for VOCs, TOC, TPH-DRO, and TPH-GRO analyses. After each soil boring is complete and all subsurface soil samples have been field screened for PCBs, samples will be selected for laboratory confirmation sampling based on the decision rules outlined in Worksheet No. 10. If a soil sample was collected for VOC, TOC, TPH-DRO, and TPH-GRO analysis, then a confirmatory sample will be selected from the same soil interval for PCB laboratory analysis. The confirmatory soil samples will be selected with a goal of co-locating a minimum of two soil samples from each soil boring for laboratory analysis of PCB, VOC, TOC, TPH-DRO, and TPH-GRO. Up to four of the six subsurface soil samples proposed for laboratory confirmatory analysis of PCBs in each soil boring will be selected from sample intervals indicating screening concentrations greater than or equal to the PSL. If the PCB field screening indicates less than four soil intervals are greater than or equal to the PSL, confirmatory samples will then be selected to provide good vertical spatial distribution and biased toward the highest PCB field screening results. A minimum of two of the six subsurface soil samples proposed for laboratory confirmatory analysis in each soil boring will be selected from sample intervals indicating PCB field screening concentrations less than the PSL. These confirmatory samples will be biased towards the deeper intervals in the soil boring to confirm the vertical extent of PCBs in subsurface soils. For additional guidance, please refer to the decision tree presented below.

Six samples from each downgradient boring will be selected for laboratory confirmation analysis of PCBs and three samples from each downgradient boring will be selected for laboratory analysis of VOCs, TOC, TPH-DRO and TPH-GRO. Samples will be selected for off-site laboratory confirmation analysis for PCBs based upon the results of the field PCB test kit screening and the visual observations made during the logging of soil cores. VOC, TOC, TPH-DRO and TPH-GRO sample locations do not need to be co-located with PCB samples, since only composite samples will be collected for PCBs in downgradient borings. Sample locations for VOC, TOC, TPH-DRO, and TPH-GRO will be selected based upon PID and visual observations. TOC sample locations will also be selected by the Tetra Tech Field Geologist based upon observed lithological changes at depth.

Groundwater Sampling

Based on PCB concentrations observed during the PCB field screening and lithological information from the source area soil borings, the field team will target depths for groundwater grab sampling in downgradient soil boring locations. The target depths will be based on the following criteria:

- Actual depths of PCB contamination observed in screening of source area borings
- Actual depths of potentially elevated VOCs as determined from field screening data
- Actual depths of clay units encountered in source area borings and the downgradient projection of these lithological units

Three groundwater grab samples will be collected at the targeted depths in each of the three (3) downgradient soil borings. The groundwater samples will be analyzed for VOCs and PCBs. Analytical results from groundwater grab samples will be used to determine placement of permanent monitoring well clusters. The following rationale will be used for selecting final placement and depths of the monitoring well clusters:

- If PCB concentrations in a groundwater grab sample are less than or equal to the PSL, then the permanent monitoring well cluster will be installed adjacent to the associated groundwater grab sample.
- If PCB concentrations in a groundwater grab sample are greater than the PSL, then a permanent monitoring well cluster will be placed further downgradient of that associated groundwater grab sample within the boundary of the former NWIRP property.

Four monitoring well clusters (one upgradient and three downgradient) will be installed to monitor potential migration of PCBs from the source areas. It is anticipated that each monitoring well cluster will include three wells at varying depths. The actual well depths will be determined by the Project Team from lithology and analytical data collected from subsurface soil sampling and groundwater grab samples

collected during the first phase of the investigation. The proposed monitoring well locations are illustrated in **Figure 13**.

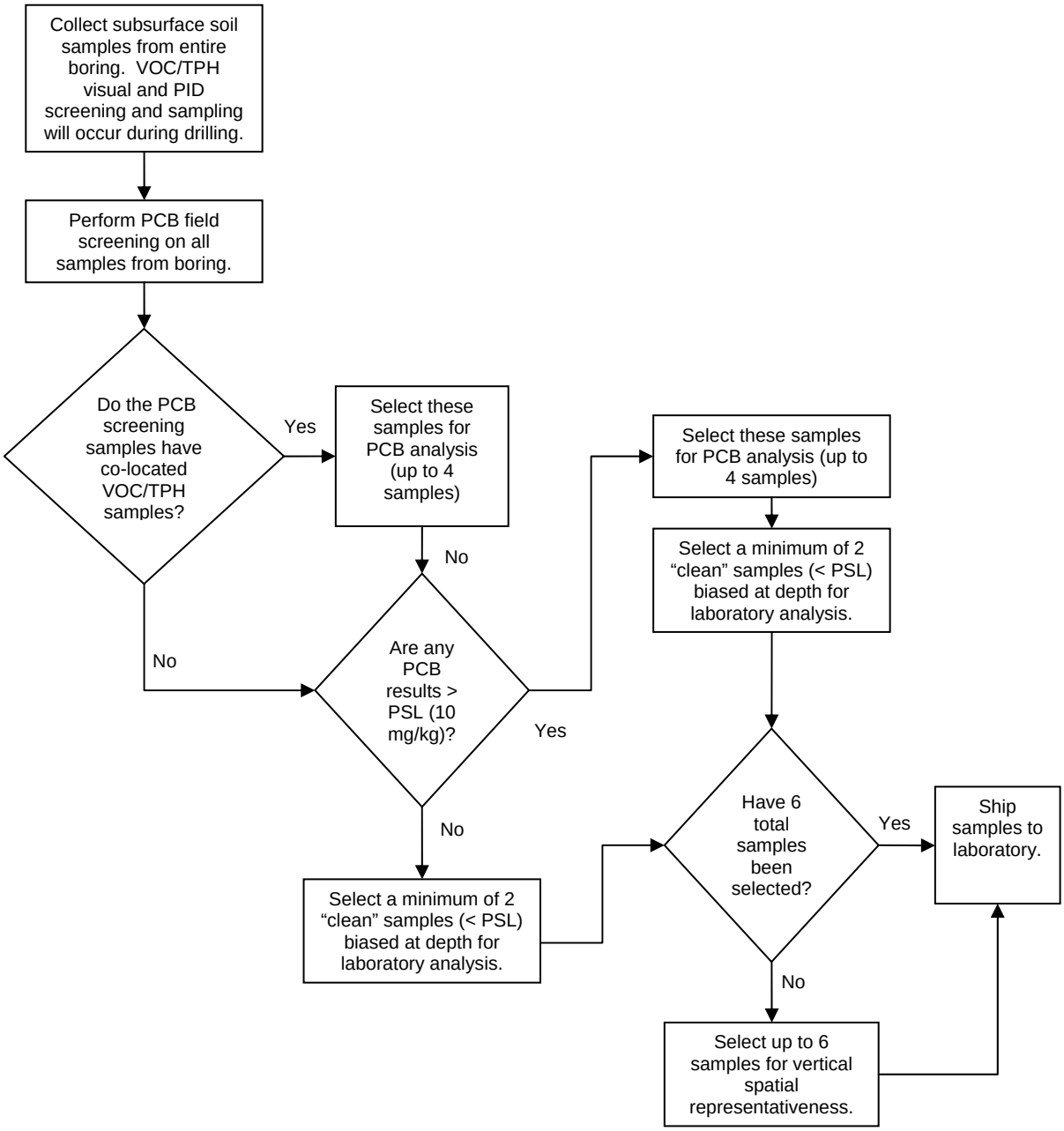
Two rounds of groundwater samples will be collected to assess the potential contribution of PCB contamination to groundwater from the Site 1 source areas. Groundwater samples will be collected in each round of sampling from the four existing monitoring wells (FW-MW01, FW-MW02, FW-MW03, and HN-MW29I). It is anticipated that a total of 12 groundwater samples will be collected from the four new monitoring well clusters (BPS1-TT-MW30, BPS1-TT-MW31, BPS1-TT-MW32, and BPS1-TT-MW33) in each round of sampling. The first round of sampling will be conducted approximately one week after monitoring well installation. The second round will be conducted approximately 3 months after the first round of sampling.

Groundwater elevation measurements will be obtained from each well to create a groundwater contour map in order to determine groundwater flow direction and to confirm each well's location (upgradient, downgradient, or sidegradient) relative to the Site source areas.

Field Quality Control Samples

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

FIGURE 14
DECISION TREE FOR SELECTION OF SOIL SAMPLES FROM SOURCE AREA BORINGS FOR LABORATORY ANALYSES



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

(UFP-QAPP Manual Section 3.1.1)

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
Source Area Soil Borings						
BPS1-SB3004	BPS1-SB3004-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	SA-1.3 – Rotosonic drilling method, disposable hand trowels, Terracore samplers for VOCs
			TBD	PCBs	6	
				VOCs, TOC, TPH-DRO, TPH-GRO	4	
BPS1-SB3005	BPS1-SB3005-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	
			TBD	PCBs	6 (plus 1 field duplicate[3])	
				VOCs, TOC, TPH-DRO, TPH-GRO	4 (plus 1 field duplicate[3])	
BPS1-SB3006	BPS1-SB3006-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	
			TBD	PCBs	6	
				VOCs, TOC, TPH-DRO, TPH-GRO	4	

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
BPS1-SB3007	BPS1-SB3007-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	SA-1.3 – Rotosonic drilling method, disposable hand trowels, Terracore samplers for VOCs
			TBD	PCBs	6 (plus 1 field duplicate[3])	
				VOCs, TOC, TPH-DRO, TPH-GRO	4	
BPS1-SB3008	BPS1-SB3008-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	
			TBD	PCBs	6 (plus 1 field duplicate[3])	
				VOCs, TOC, TPH-DRO, TPH-GRO	4 (plus 1 field duplicate[3])	
BPS1-SB3009	BPS1-SB3009-XXXX[2]	Soil	Discrete sample depth from each 5-ft interval TBD; Composite of 10-ft intervals from 120-250 ft bgs.	PCB Field Screening	25-120 ft: 19 (plus 2 field duplicates[3]) 120-250 ft: 13 (plus 2 field duplicates[3])	
			TBD	PCBs	6	
				VOCs, TOC, TPH-DRO, TPH-GRO	4	

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
Downgradient Soil Borings						
BPS1-SB3010	BPS1-SB3010-XXXX[2]	Soil	Composite of 10-ft intervals from ground surface to 120 ft bgs.	PCB Field Screening	0-120 ft: 12 (plus 2 field duplicates[3])	SA-1.3 – Rotosonic drilling method, disposable hand trowels, Terracore samplers for VOCs
			TBD	PCBs	6 (plus 1 field duplicate[3])	
				VOCs, TOC, TPH-DRO, TPH-GRO	3 (plus 1 field duplicate[3])	
BPS1-SB3011	BPS1-SB3011-XXXX[2]	Soil	Composite of 10-ft intervals from ground surface to 120 ft bgs.	PCB Field Screening	0-120 ft: 12 (plus 2 field duplicates[3])	
			TBD	PCBs	6	
				VOCs, TOC, TPH-DRO, TPH-GRO	3	
BPS1-SB3012	BPS1-SB3012-XXXX[2]	Soil	Composite of 10-ft intervals from ground surface to 120 ft bgs.	PCB Field Screening	0-120 ft: 12 (plus 2 field duplicates[3])	
			TBD	PCBs	6 (plus 1 field duplicate[3])	
				VOCs, TOC, TPH-DRO, TPH-GRO	3	
Groundwater Grab Samples						
BPS1-SB3010	BPS1-GW3010-XXXX[2]	Groundwater	TBD	PCBs, VOCs	3	SA-1.1 – Grab samples from soil borings
BPS1-SB3011	BPS1-GW3011-XXXX[2]	Groundwater	TBD	PCBs, VOCs	3	
BPS1-SB3012	BPS1-GW3012-XXXX[2]	Groundwater	TBD	PCBs, VOCs	3 (plus 1 field duplicate[3])	

Sampling Location	ID Number	Matrix	Depth (feet)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
Groundwater Sampling Rounds 1 and 2						
BPS1-FW-MW01	FW-MW01-YYYYMMDD	Groundwater	55	PCBs, VOCs	1	SA-1.1 – Low Flow using Submersible Grundfos pump
BPS1-FW-MW02	FW-MW02-YYYYMMDD	Groundwater	55	PCBs, VOCs	1	
BPS1-FW-MW03	FW-MW03-YYYYMMDD	Groundwater	68	PCBs, VOCs	1	
BPS1-HN-MW29I	HN-MW29I-YYYYMMDD	Groundwater	127	PCBs, VOCs	1	
BPS1-TT- MW30S	BPS1-MW301S-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW30I	BPS1-MW301I-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW30D	BPS1-MW301D-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW31S	BPS1-MW302S-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW31I	BPS1-MW302I-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW31D	BPS1-MW302D-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1 (plus 1 field duplicate)	
BPS1-TT-MW32S	BPS1-MW303S-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW32I	BPS1-MW303I-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW32D	BPS1-MW303D-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW33S	BPS1-MW304S-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW33I	BPS1-MW304I-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1	
BPS1-TT-MW33D	BPS1-MW304D-YYYYMMDD	Groundwater	TBD	PCBs, VOCs	1 (plus 1 field duplicate)	

[1] SOP or worksheet that describes the sample collection procedures (Worksheet No. 21).
[2] XXXX represents depth of the sample. For example, for a sample from 2 feet, use “-0002”. For a composite sample from 110 to 120 feet use “-110120”.
[3] Field duplicate locations may change in the field based on visual and olfactory observations and PID readings.

SAP Worksheet No. 19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Soil	PCB Field Screening	Dexsil L2000DX User's Manual	12-inch x 12-inch 4-mil baggie	Minimum of 500g	Cool to (4 ± 2) °C until field screening analysis, then, samples will then be transferred to jars and stored at (4 ± 2) °C until selection of laboratory samples (one to two days).	See holding times below for analyses.
	VOCs	SW-846 5035/8260B TriMatrix SOPs GR-04-105/GR-04-104	1 – 5 gram (g) Terracore into pre-preserved or blank tare weighted vial	5g	Cool to (4 ± 2) °C; no headspace	Pre-preserved in the field, 14 days to analysis.
	PCBs	SW-846 3546/8082 TriMatrix SOPs GR-09-108/GR-03-128	1 – 8 ounce (oz) wide-mouth glass jar	250g	Cool to (4 ± 2) °C	14 days to extraction; 40 days analysis
	TPH-DRO	SW-846 3550C/8015B TriMatrix SOPs GR-09-123/GR-03-122	1 – 8 oz wide-mouth glass jar	250g	Cool to (4 ± 2) °C	14 days to extraction; 40 days analysis
	TPH-GRO	SW-846 5035A/8015B TriMatrix SOP GR-03-121	1 – 5 g Terracore into pre-preserved or blank tare weighted vial	5g	Cool to (4 ± 2) °C; no headspace	Pre-preserved in the field, 14 days to analysis.
	TOC	Walkley Black Method TriMatrix SOP GR-06-105	1 – 8 oz wide-mouth glass jar	250g	Cool to (4 ± 2) °C	28 days to analysis
Groundwater and aqueous QC blanks	VOCs	SW-846 8260B TriMatrix SOP GR-04-104	3 – 40 milliliter (mL) clear glass vials	120mL	Cool to (4 ± 2) °C Hydrochloric acid (HCl) to pH < 2; no headspace	14 days to analysis
	PCBs	SW-846 3510C/8082 TriMatrix SOPs GR-09-107/GR-03-128	2 – 1 liter (L) amber glass bottle	2L	Cool to (4 ± 2) °C	7 days to extraction; 40 days analysis
IDW	TCLP VOCs	SW-846 5035/8260B TriMatrix SOPs GR-01-119/GR-04-104	3 – 5g Terracores into pre-preserved and weighed vials	15g	Cool to (4 ± 2) °C; no headspace	48 hours for preparation; 14 days to analysis

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference**[1]	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time (preparation / analysis)**
	TCLP Semivolatile Organic Compounds (SVOCs)	SW-846 1311/8270C TriMatrix SOPs GR-01-119/GR-04-103	1 – 8 oz wide-mouth glass jar	250g	Cool to (4 ± 2) °C	14 days to extraction; 40 days analysis
	TCLP Pesticides/ Herbicides	SW-846 1311/8270C TriMatrix SOPs GR-01-119/ GR-04-103				
	Total PCBs	SW-846 8082 TriMatrix SOP GR-03-128				
	TCLP Metals (except mercury)	SW-846 1311/6010B TriMatrix SOPs GR-01-119/GR-01-100/ GR-01-147	1 – 8 oz wide-mouth glass jar	250g	Cool to (4 ± 2) °C	180 days to analysis
	TCLP Mercury	SW-846 1311/7470A TriMatrix SOPs GR-01-119/GR-01-123/ GR-01-140				28 days to analysis
	Ignitability	SW-846 1030 TriMatrix SOP GR-18-124	1 – 8 oz wide-mouth glass jar	250g	Cool to (4 ± 2) °C	7 days to analysis for Sulfide, 14 days to analysis for Cyanide, and 28 days to analysis for Corrosivity
	Reactivity (Cyanide/Sulfide)	SW-846 9010C/9014/SW-846 Chapter 7.7.3.4 TriMatrix SOPs GR-18-106,109/GR-05-122/ GR-18-117/GR-06-108				
	Corrosivity	SW-846 1110/9045D TriMatrix SOP GR-07-113				

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

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

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs[1]	No. of Source Blanks	No. of Equip. Blanks[2]	No. of VOA[3] Trip Blanks	No. of PT[4] Samples	Total No. of Samples to Lab
Soil	PCBs Field Screening	222	23	NA	NA	NA	NA	NA	NA
Soil	VOCs	33	4	2/2	1	2	6	NA	46
	PCBs	54	6	3/3	1	3	NA	NA	64
	TPH-DRO	33	4	2/2	NA	2	NA	NA	39
	TPH-GRO	33	4	2/2	NA	2	6	NA	45
	TOC	33	4	2/2	NA	2	NA	NA	39
Groundwater (Grabs)	VOCs	9	1	1/1	NA	1	1	NA	12
	PCBs	9	1	1/1	NA	1	NA	NA	11
Groundwater (Round 1)	VOCs	16	2	1/1	NA	1	10	NA	29
	PCBs	16	2	1/1	NA	1	NA	NA	19
Groundwater (Round 2)	VOCs	16	2	1/1	NA	1	10	NA	29
	PCBs	16	2	1/1	NA	1	NA	NA	19

[1]Although the MS/MSD is not typically considered a field QC; it is included here because location determination is often established in the field. The MS and MSD are **not** included in the "Total No. of Samples to Lab".

[2]Primarily disposable equipment will be used for soil sampling. Equipment blanks will only be collected if non-disposable or non-dedicated equipment is used.

[3]VOA – Volatile Organic Analyte

[4]PT – Proficiency Testing

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

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
CT-05	Database Records and Quality Assurance Rev. 2, January 2001	Tetra Tech	NA	N	None.
GH-1.3	Soil and Rock Drilling Methods Rev. 1, June 1999	Tetra Tech	Drill Rig and associated equipment	N	None.
GH-1.5	Borehole and Sample Logging Rev. 1, June 1999	Tetra Tech	Boring Log sheets	N	None.
GH-2.8	Groundwater Monitoring Well Installation Rev. 3, September 2003	Tetra Tech	PVC, grout, filter sand	N	None.
HS-1.0	Utility Locating and Excavation Clearance Rev. 2, December 2003	Tetra Tech	NA	N	None.
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing Rev. 7, April 2008	Tetra Tech	Submersible Pump, Tubing, Water Quality Meter, Turbidity Meter, Water Level Indicator	N	None.
SA-1.3	Soil Sampling Rev. 9, April 2008	Tetra Tech	Sampling Procedures, Methods	N	None.
SA-6.1	Non-radiological Sample Handling Rev. 3, February 2004	Tetra Tech	Sample Bottle ware, Packaging Material, Shipping Materials	N	None.
SA-6.3	Field Documentation Rev. 3, March 2009	Tetra Tech	Field Logbook, Field Sample Forms, Boring Logs	N	None.
SA-7.1	Decontamination of Field Equipment Rev. 6, January 2009	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	Y	Nitric acid is removed from the decontamination procedure. Isopropyl Alcohol to be used, if field conditions warrant.

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

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[2]	Comments
Dexsil L2000DX	Calibration Verification (see Worksheet No. 25)	Daily	Manufacturer's Guidance	Replace the instrument	Tetra Tech Project Chemist or designee	Manufacturer's Guidance	None.
PID	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	Manufacturer's Guidance	None.
Drill Rig	Inspection	Daily	Equipment inspection sheet criteria	Replace	Tetra Tech FOL or designee	GH-1.5, SA-2.5	None.
Disposable Hand Trowel	Inspection	Per use	NA	Replace	Tetra Tech FOL or designee	SA-1.3	None.
Depth to Water Meter	Visual Inspection	Daily	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	Manufacturer's Guidance	None.
Multi-Parameter Water Quality Meter	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	Manufacturer's Guidance	None.
Turbidity Meter	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance; Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <10 Nephelometric Turbidity Units (NTUs).	Replace	Tetra Tech FOL or designee	Manufacturer's Guidance	None.

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

SAP Worksheet No. 23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L2000DX User's Manual	L2000DX User's Manual 13 July 2006	Screening; Definitive if sufficient correlation to laboratory data	PCBs - Soil	Dexsil L2000DX	Tetra Tech	Y –Soil will be analyzed wet. PCB screening data will be used to select soil sampling locations for off-site laboratory analysis.
GR-01-100	Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), 02/05/09 – rev. 5.7 6010C	Definitive	Metals – Aqueous and Soil Analysis	Perkin Elmer ICP 3000 3300DV 5300DV (ICP-AES)	TriMatrix	N
GR-01-119	Toxicity Characteristic Leaching Procedure (TCLP), 09/25/09 – rev. 2.2, 1311	Definitive	VOCs/ SVOCs/Pesticides/ PCB/Herbicides – Zero Headspace Extractor (ZHE) and Regular TCLP	NA	TriMatrix	N
GR-01-123	Mercury by Semi-Automated Cold Vapor Atomic Absorption 02/01/09 – rev. 5.6, 7470A/7471A	Definitive	Mercury – Aqueous Analysis	PSA Millennium Cold Vapor Mercury Analyzer (CVAA)	TriMatrix	N
GR-01-137	Block Digestion of Solids for ICP and ICPMS, 02/01/09 – rev. 1.5 3050B	Definitive	Metals – Soil Digestion	None	TriMatrix	N
GR-01-140	Digestion of Mercury in Water, Wastewater, and Aqueous Waste, 02/20/09 – rev. 0.4 7470A	Definitive	Mercury – Aqueous Digestion	None	TriMatrix	N
GR-01-147	Block Digestion for Total Metals in Water for ICP, 01/20/09 – rev. 0.4 3010A	Definitive	Metals – Aqueous Digestion	None	TriMatrix	N
GR-03-121	GRO by Gas Chromatography, 08/20/09 – rev. 3.4 8015B	Definitive	TPH-GRO – Soil Analysis	Hewlett Packard 5890 II (GC/Flame Ionization Detector [FID])	TriMatrix	N
GR-03-122	DRO, 01/07/09 – rev. 2.3 8015B	Definitive	TPH-DRO – Soil Analysis	Hewlett Packard 5890A (GC/FID)	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
GR-03-128	Polychlorinated Biphenyls by Gas Chromatography, 09/05/09 – rev. 2.5, 8082	Definitive	PCBs - Aqueous and Soil Analysis	Agilent GC 5890 or 6890 Electron Capture Detector (GC/ECD)	TriMatrix	N
GR-04-103	Base/Neutral/Acid Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS), 02/20/09 – rev. 5.5, 8270C	Definitive	SVOCs/Pesticides/ Herbicides - Aqueous and Soil Analysis	Agilent GC 6890 5973N MS or Agilent GC 6890 5975N MS	TriMatrix	N
GR-04-104	Volatile Organic Compounds by Purge & Trap Capillary Gas Chromatography/ Mass Spectrometry, 02/20/09 – rev. 4.5, 8260B	Definitive	VOCs - Aqueous and Soil Analysis	Agilent GC 6890 5973N MS or Agilent GC 6890 5975N MS	TriMatrix	N
GR-04-105	Closed System Purge & Trap Extraction For Volatile Organic Compounds, 02/20/09 – rev. 1.2, 5035A	Definitive	VOCs/TPH-GRO – Soil Extraction	None	TriMatrix	N
GR-05-122	Total Cyanide by Konelab Ultraviolet (UV) Spectrophotometry, 9/25/09 – rev. 0.4, Method 9014	Definitive	Cyanide Reactivity – Aqueous and Soil Analysis	Konelab Automated UV Spectrophotometer	TriMatrix	N
GR-06-105	Organic Carbon Walkley-Black, 03/09/09 – rev 2.4, Agronomy, Methods of Soil Analysis 29-3.5.2	Definitive	Total Organic Carbon – Soil Analysis	NA	TriMatrix	N
GR-06-108	Titrimetric Procedure for Acid Soluble and Reactive Sulfides, 9/12/06 – rev. 2.0, Method 9034/SW-846 Chapter 7 7.3.4	Definitive	Sulfide Reactivity – Aqueous and Soil Analysis	NA	TriMatrix	N
GR-07-113	Potentiometric pH in Soil and Waste, 3/5/09 – rev. 0.3, Method 9045C	Definitive	Corrosivity – Soil Analysis	pH Meter	TriMatrix	N
GR-09-101	Extraction of Base Neutrals and Acids from Water, 03/12/09 – rev. 3.7, 3510C	Definitive	SVOCs – Aqueous Extraction	None	TriMatrix	N
GR-09-107	Extraction of Organochlorine Pesticides and PCBs from Water, 08/12/09 – rev. 5.2, 3510C	Definitive	Pesticides/PCBs – Aqueous Extraction	None	TriMatrix	N
GR-09-108	Extraction of Organochlorine Pesticides and Polychlorinated Biphenyls in Soil, Sludge, and Wipe Samples, 3/27/09 – rev. 4.3, Method 3550C	Definitive	PCBs – Soil Preparation	NA	TriMatrix	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
GR-09-123	DRO Extraction, 04/25/08 – rev. 0.2 3550C	Definitive	TPH-DRO – Soil Extraction	NA	TriMatrix	N
GR-10-104	Chain of Custody, 4/06/09 – rev 2.2	NA	Storage and Custody	NA	TriMatrix	N
GR-15-100	Sample Receipt and Log-in, 12/31/08 – rev 3.2	NA	Login and Storage	NA	TriMatrix	N
GR-15-102	Laboratory Waste Disposal, 4/30/09 – rev 2.1	NA	Waste Disposal	NA	TriMatrix	N
GR-18-106	Amenable and Total Cyanide Extraction and/or Macro-Distillation, 11/25/09 – rev. 3.2, Method 9010C/9013A	Definitive	Cyanide Reactivity – Soil Preparation	NA	TriMatrix	N
GR-18-109	Midi-Distillation for Cyanide, 11.25.09 – rev. 3.4, Method 9010C	Definitive	Cyanide Reactivity – Aqueous Preparation	NA	TriMatrix	N
GR-18-117	Reactive Sulfide, 9/12/06 – rev. 3.0, SW-846 Chapter 7 7.3.4	Definitive	Sulfide Reactivity – Aqueous and Soil Preparation	NA	TriMatrix	N
GR-18-124	Setaflash Closed Cup Flash Point, 12/31/08 – rev. 1.2, Method 1020A	Definitive	Ignitability – Soil Analysis	Koehler Rapid Tester, Closed Cup	TriMatrix	N

SAP Worksheet No. 24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
Dexsil L2000DX Field PCBs	Single point (50 ppm) calibration standard	Every 20 samples or every hour, whichever comes first, and if temperature has changed more than 5 °C.	Electrode output must be within 50-75 millivolts (mV). Temperate must be within acceptable range.	Check electrode output in Diagnostics mode. If not within range, change the calibration solution and refill the electrode with electrode filling solution. Recalibrate. If this does not work, polish the electrode. Recalibrate. Replace electrode.	Tetra Tech Project Chemist	L2000DX User's Manual
GC/MS VOCs	Tuning	Prior to analysis and at the beginning of each 12 hour shift.	See table in SOP and/or method. Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Determine source of problem by re-tuning using auto or manual tune, clean source, rerun tune.	Analyst/ Supervisor	TriMatrix SOP GR-04-104
	Initial Calibration (ICAL) – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	The percent relative standard deviation (RSD) for each Calibration Check Compound (CCC) must be ≤ 30%. The minimum mean response factor (RF) for each System Performance Check Compound (SPCC) must meet the criteria stated in 8260B. If the RSD for an analyte is > 15%, use a linear curve (linear regression correlation coefficient (r) ≥ 0.995) or quadratic curve (coefficient of determination (r^2) ≥0.99, (minimum 6 points) for quantitation.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	ICV - 2nd source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for individual compounds must be within 80-120% of true value.	Identify source of problem, correct, repeat calibration, rerun samples	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12 hour shift after a bromofluorobenzene (BFB) tune.	The minimum RF for all analytes must meet the criteria stated in 8260B. All analytes must be ≤ 20 percent difference or percent drift (%D).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
GC/MS SVOCs/ Pesticides/ Herbicides	Tuning	Prior to analysis and at the beginning of each 12 hour shift.	See table in SOP and/or method. Must meet the ion abundance criteria required by the method. No samples may be accepted without a valid tune.	Determine source of problem by re-tuning using auto or manual tune, clean source, rerun tune.	Analyst/ Supervisor	TriMatrix SOP GR-04-103
	ICAL – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	The %RSD for each CCC must be ≤ 30%. The minimum mean RF for each SPCC must meet the criteria stated in 8270C. If the RSD for an analyte is > 15%, use a linear curve (r > 0.995) or quadratic curve (r^2 >0.99, minimum 6 points) for quantitation.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	ICV - 2nd source	Once after each initial calibration.	%R for individual compounds must be within 80-120% of true value.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning of each 12 hour shift after a Decafluorotriphenyl-phosphine (DFTPP) tune.	The minimum RF for SPCCs must meet the criteria stated in 8270C. All analytes must be ≤ 20 %D.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
GC/ECD PCBs	Breakdown Check	Prior to analysis and at the beginning of each 12 hour shift.	Calculated breakdown for DDT and Endrin must be less than 15%.	Determine the source of the problem, correct and rerun breakdown standard.	Analyst/ Supervisor	TriMatrix SOPs GR-03-120/ GR-03-128
	ICAL – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	Must be ≤20% RSD, or r ≥0.995, or r^2 ≥0.99 with a minimum of 6 points, for all except toxaphene and chlordane. One point calibration for toxaphene and chlordane.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	ICV - 2nd source	Once after each ICAL.	%R for individual compounds must be within 80-120% of true value.	Identify source of problem, correct, repeat.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	%R for individual compounds must be within 80-120% of true value.	If %D > 15% and samples are < PQL, narrate. If %D > 15% only on one column, narrate. If %D > 15% for closing CCV, and is likely due to matrix interference, narrate. Otherwise, reanalyze all samples back to the last acceptable CCV.	Analyst/ Supervisor	
GC/FID TPH-GRO TPH-DRO	ICAL – A minimum of a 5-point calibration curve is analyzed	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	Must be ≤20% RSD or r^2 ≥0.99 with a minimum of 5 points.	Correct problem then repeat ICAL.	Analyst/ Supervisor	TriMatrix SOPs GR-03-121/ GR-03-122
	ICV - 2nd source	Once after each ICAL.	%R for individual compounds must be within 80-120% of true value.	Re-run the sample once. If second analysis fails, prepare a new calibration, run, and repeat ICV. Re-analyze all samples from last acceptable ICV.	Analyst/ Supervisor	
	CCV	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	%D for individual compounds must ≤20%.	Remake CCV solution and re-analyze. If re-analysis is out of control, run a new calibration and all samples analyzed since the last acceptable CCV.	Analyst/ Supervisor	
ICP-AES Metals	ICAL – A minimum of a 1-point calibration is analyzed	The instrument is calibrated at the beginning of each day or if the QC is out of criteria.	The instrument is calibrated by a one point calibration per manufacturer's guidelines. Analytes ran at their calibration levels must fall within 90-110%R of the true values.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	TriMatrix SOP GR-01-100
	ICV - 2nd source	Once after each initial calibration, prior to sample analysis.	%R for individual compounds must be within 90-110% of true value.	Identify source of problem, correct, repeat.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	CCV	At the beginning and end of the sequence and after every 10 samples.	%R for individual compounds must be within 90-110 %R of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every ten samples, and at the end of the sequence.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.	Analyst/ Supervisor	
	Low-Level Check Standard (if using 1-point ICAL)	After the ICAL or CCV and before samples.	%R for individual compounds must be within 80-120% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	Interference Check Standards (ICS - ICSA & ICSB)	At the beginning and end of each run and after every 20 samples.	ICSA recoveries must be within the absolute value of 2x MDL for all non-spiked analytes and ICSB recoveries must be within 80-120%R of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
CVAA Mercury	ICAL – A minimum of a 5-point calibration curve is analyzed	The instrument is calibrated at the beginning of each day or if the QC is out of criteria.	The correlation coefficient, r, must be ≥0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	TriMatrix SOP GR-01-123
	ICV - 2nd source	Once after each ICAL.	%R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	CCV	At the beginning and end of the sequence and after every 10 samples.	%R must be within 80-120% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	
	CCB	After every 10 samples and at the end of the sequence.	Analyte must be < 2x MDL.	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.	Analyst/ Supervisor	
	CCV (undistilled)	The CCV (undistilled) is analyzed at the beginning and end of the sequence and after every 10 samples.	%R must be within 90-110% of true value.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing CCVs.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
TOC Analyzer TOC	NA - Procedure is a titration.	NA	NA	NA	Analyst/ Supervisor	TriMatrix SOP GR-06-105
Ignitability	NA	NA	NA	NA	Analyst/ Supervisor	TriMatrix SOP GR-18-124
Cyanide Reactivity	ICAL- six point calibration	ICAL performed daily prior to sample analysis.	The correlation coefficient, r, must be ≥0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	TriMatrix SOP GR-05-122
Sulfide Reactivity	NA	NA	NA	NA	Analyst/ Supervisor	TriMatrix SOP GR-06-108
Corrosivity	pH meter must be calibrated using three buffers that bracket the expected pH range.	Daily before use.	Slope must be between 90-102%, and each buffer must read within ± 0.05 pH units.	Repeat calibration until acceptance criteria is met. Replace probe if several attempts are unsuccessful.	Analyst/ Supervisor	TriMatrix SOP GR-07-113

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

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

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Dexsil L2000DX	Filling and Polishing Electrode and Electrode Check	Field PCBs	NA	After electrode is filled after empty storage period.	Electrode output must reach at least 140 mV within one minute in rinse solution.	Follow Electrode Care in User's Manual.	Tetra Tech Project Chemist	L2000DX User's Manual
	NA	Calibration	NA	See Worksheet No. 24.	See Worksheet No. 24.	See Worksheet No. 24.		
GC/MS	Clean source, replace vacuum pump oil, replace filament, replace inlet liner, replace septa, and bake out instrument.	VOCs/ SVOCs/ Pesticides/ Herbicides	Inspect vacuum pressure.	Clean source and replace vacuum pump oil annually or as needed. Routine maintenance as necessary. Tune instrument after major maintenance. Inspect vacuum pressure daily.	BFB and DFTPP tunes must meet the criteria listed in the method. Per instrument manufacturer's instructions.	Inspect system and correct problem; Re-tune instrument if necessary. Perform new ICAL.	GC/MS Analyst	TriMatrix SOPs GR-04-103/ GR-04-104
EST Encon Purge &Trap Concentrator	Clean sparge vessel, replace adsorbent trap, bake trap.	Soil VOCs/ TPH-GRO	Check purge flow.	Maintenance as necessary. Purge flow checked weekly.	40 mL/minute +/- 2mL/minute.	Adjust flow rate or perform leak check. If leak or restriction found fix problem.	GC/MS Analyst	TriMatrix SOPs GR-04-104/ GR-04-105
GC/ECD	Replace septa, replace inlet liner, clip column, bake out detectors, recondition column.	PCBs	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions.	Inspect system; correct problem; perform new initial calibration and affected samples.	GC Analyst	TriMatrix SOPs GR-03-120/ GR-03-128
GC/FID	Replace septa, replace inlet liner, clip column, clean or replace FID jet, bake out detector, recondition column.	TPH-GRO TPH-DRO	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks.	Replace liner, septa, and clip column as indicated by instrument change in response and chromatography. If signal is suppressed, clean or replace FID jet. Bake out detectors and columns if signal elevated.	Per instrument manufacturer's instructions.	Inspect system; correct problem; perform new initial calibration and affected samples.	GC Analyst	TriMatrix SOPs GR-03-121 GR-03-122

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
ICP-AES	Replace pump tubing and clean filters.	Metals	Inspect torch visually, disassemble torch assembly and replace worn or damaged parts.	Replace pump tubing and inspect torch daily. Clean filters weekly. Disassemble torch every 6-8 weeks or as needed.	Per instrument manufacturer's instructions.	Inspect torch assembly and replace any worn or damaged parts. Clean or replace nebulizer and aluminum injector.	Metals Analyst	TriMatrix SOP GR-01-100
CVAA	Flush all tubing, replace pump tubing, replace Perma-pure drying system membrane or entire assembly, or replace activated charcoal in exhaust line.	Mercury	Check for discoloration in Perma-pure drying system.	Flush tubing daily. Change tubing at least weekly. Check Perma-pure system for discoloration monthly. Replace Perma-pure inner membrane every six months. Replace charcoal annually.	Per instrument manufacturer's instructions.	Inspect system and correct problem.	Metals Analyst	TriMatrix SOP GR-01-123
TOC	NA - Procedure is a titration.	TOC	NA	NA	NA	NA	Analyst/ Supervisor	TriMatrix SOP GR-06-105
Ignitability	Clean and dry all parts of the cup. If oil residue is present, clean with solvent.	Ignitability	Perform visual inspection of apparatus.	Daily prior to use.	Flashpoint of p-Xylene must be 81° F± 2°F.	If outside acceptance window, inspect apparatus, verify operating conditions, or replace standard.	Analyst/ Supervisor	TriMatrix SOP GR-18-124
Cyanide Reactivity	Change diluent, tubing, and lamp as needed.	Cyanide Reactivity	Clean and check injection syringe and mixer alignment daily.	Perform dilution test daily after initial calibration.	Dilution results must be within 10% of original value.	Recalibrate and perform dilution test again. If test still fails, replace syringe or perform manual dilutions.	Analyst/ Supervisor	TriMatrix SOP GR-05-122
Sulfide Reactivity	NA	Sulfide Reactivity	NA	NA	NA	NA	Analyst/ Supervisor	TriMatrix SOP GR-06-108
Corrosivity	Clean pH electrode as needed. Refill with fluid as needed.	Corrosivity	Inspect pH probe.	Inspect pH probe prior to use and throughout measurement process.	Per manufacturer's instructions.	Replace pH probe.	Analyst/ Supervisor	TriMatrix SOP GR-07-113

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

SAP Worksheet No. 26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL and field personnel / Tetra Tech
Sample Packaging (Personnel/Organization): FOL and field personnel / Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL and field personnel / Tetra Tech
Type of Shipment/Carrier: Overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample custodians / TriMatrix
Sample Custody and Storage (Personnel/Organization): Sample custodians / TriMatrix
Sample Preparation (Personnel/Organization): Preparation laboratory staff / TriMatrix
Sample Determinative Analysis (Personnel/Organization): GC/MS, GC/ECD, GC, GC/FID, ICP-AES, CVAA / TriMatrix
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt (stipulated in Master Services Agreement [MSA])
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days from receipt (stipulated in MSA)
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample custodians / TriMatrix

SAP Worksheet No. 27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures

Following sample collection using the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to TriMatrix for analysis. Samples will be preserved as appropriate based on the analytical method. TriMatrix will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4 (±2) °C until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP SA-6.1 (Appendix A).

Chain-of-Custody Procedures

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently had custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Tetra Tech SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure, which is provided in Appendix A. Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratories and copies of the TriMatrix chain-of-custody form, which are provided in Appendix B.

Laboratory Sample Custody Procedures (receipt of samples, archiving, and disposal)

Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (TriMatrix SOPs GR-15-100 [Sample Receipt and Login], GR-15-102 [Laboratory Waste Disposal], and GR-10-104 [Chain of Custody]), which are provided in Appendix B.

Sample Designation System

Each sample collected for analysis will be assigned a unique sample tracking number. This number will consist of a segmented alphanumeric code that identifies the Site location, the sample type (sample

medium or QC sample designation), the sample location, and the sample depth indicator. The alphanumeric coding system to be used is as follows:

Site Identifier:

BPS1 = Bethpage Site 1

BPS1-TT-MW = Bethpage Site 1 Monitoring Well (Tetra Tech-installed Monitoring Well)

Sample Medium and Sample Location:

SB = Soil Boring Sample

GW = Groundwater Grab Sample

MW = Monitoring Well Sample

Each soil boring and monitoring well will be assigned a three- or four-digit number to identify the location (e.g., BPS1-SB3004, which indicates soil boring number 3004; BPS1-MW303, which indicates monitoring well number 303).

Depth:

For soil samples, the soil sample depth will be indicated by a four digit number (unless the lower limit of the depth interval is greater than 99 feet, where six digits will be necessary). The first two (or three) digits will represent the upper limit of the sample depth interval bgs (rounded to the nearest foot) and the bottom two (or three) digits will represent the lower limit of the depth interval bgs.

BPS1-SB3004-2025 (sample collected from 20-25 feet bgs)
BPS1-SB3004-150155 (sample collected from 150-155 feet bgs)

Groundwater grab samples will also use a four or six digit number for the sample depth.

BPS1-GW3007-7075 (sample collected from 70-75 feet bgs) or
BPS1-GW3007-150155 (sample collected from 150-155 feet bgs)

Monitoring well samples will use an eight digit date to identify the sample event as follows:

BPS1-MW30-06152010 (sample collected on June 15, 2010)

Quality Assurance/Quality Control (QA/QC) Sample Designation:

FB = Field Blank (used for source water blanks)

RB = Equipment Rinsate Blank (if necessary)

TB = Trip Blank

DUP = Field Duplicate

All QC samples will be assigned a sequential sample number. The field duplicate, MS, and sample duplicate/MSD samples will be collected from the same station.

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

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

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Groundwater/QC/ IDW samples
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B TriMatrix SOP GR-04-104

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Trip Blank	One per cooler of VOC samples shipped to laboratory.	No analytes > ½ the QL, except common lab contaminants, which must be < QL.	No corrective action by laboratory.	None	Bias / Contamination	No analytes > ½ the QL, except common lab contaminants, which must be < QL.
Method Blank	One per batch of 20 or less.	No target compounds >½ the QL, except common lab contaminants, which must be < QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds >½ the QL, except common lab contaminants which must be < QL.
System Monitoring Compounds (SMC)/ Surrogates	4 per sample: Dibromofluoromethane 1,2-Dichloroethane Toluene-d8 4-Bromofluorobenzene	85-115%R 70-120%R 85-120%R 75-120%R	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD Quality Systems Manual (QSM) limits for %R.
Laboratory Control Sample (LCS)	One per batch of 20 or less.	Table G-4 DoD QSM Version 4.1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
Laboratory Control Sample Duplicate (LCSD) (Not Required)	Used if insufficient sample supplied for batch MS/MSD.	Table G-4 DoD QSM Version 4.1. LCS/LCSD RPD must be ≤ 30%.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. LCS/LCSD RPD must be ≤ 30%.

Matrix	Soil/Groundwater/QC/ IDW samples
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B TriMatrix SOP GR-04-104

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Internal Standards (IS)	3 per sample: Fluorobenzene Chlorobenzene-d5 1,4-Dichlorobenene-d5	Retention time +/- 30 seconds from retention time (RT) of the ICAL midpoint standard, and the Extracted Ion Current Profile (EICP) area within -50% to +100 % of ICAL midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Retention time +/- 30 seconds from RT of the ICAL midpoint standard, and EICP area within -50% to +100 % of ICAL midpoint standard.
MS/MSD	One per Sample Delivery Group (SDG) or every 20 samples.	Table G-4 DoD QSM Version 4.1. MS/MSD RPD should be ≤ 30%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD should be ≤ 30%.

Matrix	Soil/Groundwater/QC /IDW samples
Analytical Group	PCBs
Analytical Method/ SOP Reference	SW-846 8082 TriMatrix SOP GR-03-128

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	No target compounds > ½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > ½ the QL.
Surrogates	2 per sample: Tetra-chloro-meta-xylene and Decachlorobiphenyl	36-114%R 40-135%R	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
LCS	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
MS/MSD	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1. MS/MSD RPD should be ≤ 30%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD should be ≤ 30%.
Second Column Confirmation	Used to confirm hit on primary column.	RPD must be ≤40%.	Report the primary column result unless obvious matrix interference exists.	Analyst, Laboratory Supervisor and Data Validator	Confirmation	RPD must be ≤40%.

Matrix	Soil samples
Analytical Group	TPH-GRO
Analytical Method/ SOP Reference	SW-846 8015B TriMatrix SOP GR-03-121

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	No target compounds > ½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > ½ the QL.
Surrogates	1 per sample: aaa-Trifluorotoluene	76-113%R	If recovery is outside acceptance limits, verify calculations, dilutions, and surrogate solutions. Verity instrument performance. Re-analyze samples unless matrix interference is verified by MS recovery.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
LCS	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1.	Determine the reason for failure. All samples must be re-analyzed unless the LCS is out-of-control high and there are no reportable sample results in the associated samples.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
MS/MSD	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1. MS/MSD RPD should be ≤ 30%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD should be ≤ 30%.

Matrix	Soil samples
Analytical Group	TPH-DRO
Analytical Method/ SOP Reference	SW-846 8015B TriMatrix SOP GR-03-122

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	No target compounds > ½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > ½ the QL.
Surrogates	1 per sample: o-Terphenyl	44-137%R	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
LCS	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
MS/MSD	One per batch of 20 or less.	Table G-16 DoD QSM Version 4.1. MS/MSD RPD should be ≤ 30%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD should be ≤ 30%.

Matrix	IDW samples
Analytical Group	SVOCs
Analytical Method/ SOP Reference	SW-846 8270C TriMatrix SOP GR-04-103

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	No target compounds > ½ the QL, except common lab contaminants which must be < QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > ½ the QL, except common lab contaminants which must be < QL.
SMCs	6 per sample: 2-Fluorophenol Phenol-d6 2,4,6-Tribromophenol Nitrobenzene-d5 2-Fluorobiphenyl ortho-Terphenyl	 20-110%R 10-115%R 40-125%R 40-110%R 50-110%R 50-135%R	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
LCS	One per batch of 20 or less.	Tables G-2 and G-6 DoD QSM Version 4.1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DOD QSM limits for %R.
IS	6 per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time +/- 30 seconds from RT of the ICAL midpoint standard, and the EICP area within -50% to +100 % of ICAL midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Retention time +/- 30 seconds from RT of the ICAL midpoint standard, and the EICP area within -50% to +100 % of ICAL midpoint standard.

Matrix	IDW samples
Analytical Group	SVOCs
Analytical Method/ SOP Reference	SW-846 8270C TriMatrix SOP GR-04-103

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
MS/MSD	One per SDG or every 20 samples.	Tables G-2 and G-6 DoD QSM Version 4.1. MS/MSD RPD should be ≤ 30%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DOD QSM limits for %R. MS/MSD RPD should be ≤ 30%.

Matrix	IDW samples
Analytical Group	Pesticides/Herbicides
Analytical Method/ SOP Reference	SW-846 8270C TriMatrix SOP GR-04-103

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	No target compounds >½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds >½ the QL.
Surrogates	6 per sample: 2-Fluorophenol Phenol-d6 2,4,6-Tribromophenol Nitorbenzene-d5 2-Fluorobiphenyl ortho-Terphenyl	 30-120%R 20-109%R 23-120%R 20-140%R 35-130%R 34-130%R	Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Lab internal limits for %R.
LCS	One per batch of 20 or less.	Lab internal limits. See attached table in Appendix B.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Lab internal limits for %R.
MS/MSD	One per batch of 20 or less.	Lab internal limits. See attached table in Appendix B.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Lab internal limits and RPD.

Matrix	IDW samples
Analytical Group	Pesticides/Herbicides
Analytical Method/ SOP Reference	SW-846 8270C TriMatrix SOP GR-04-103

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
IS	6 per sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time +/- 30 seconds from RT of the ICAL midpoint standard, and the EICP area within -50% to +100 % of ICAL midpoint standard.	Inspect instrument for malfunctions. Check for possible matrix effects. If none found, reanalyze affected sample if sufficient sample is available. Qualify data as needed.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Retention time +/- 30 seconds from RT of the ICAL midpoint standard, and the EICP area within -50% to +100 % of ICAL midpoint standard.

Matrix	IDW samples
Analytical Group	Metals
Analytical Method/ SOP Reference	SW-846 6010B TriMatrix SOP GR-01-100

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Calibration Blank	After the ICV and after every CCV.	Contaminants in the calibration blank must be < the MDL.	Correct problem. Reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Contaminants in the calibration blank must be < the MDL.
Method Blank	One per digestion batch of 20 or fewer samples.	No target analytes > ½ QL. For common lab contaminants, no analytes > QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target analytes > ½ QL. For common lab contaminants, no analytes > QL.
LCS	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DoD QSM limits for %R.
MS	One per digestion batch of 20 or fewer samples.	Table G-18 DoD QSM Version 4.1.	Check for errors in calculations and spike preparation. Check un-spiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	DoD QSM limits for %R, if sample < 4x spike added.
Duplicate Sample	One per preparatory batch of 20 or fewer samples per matrix.	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Laboratory Supervisor and Data Validator	Precision	The RPD should be ≤ 20% for duplicate samples for both water and soils.
ICP Serial Dilution	One per digestion batch.	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must be ≤10% RPD.	Analyze Post Digestion Spike and narrate the analytes that fail criteria.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must be ≤10% RPD.

Matrix	IDW samples
Analytical Group	Metals
Analytical Method/ SOP Reference	SW-846 6010B TriMatrix SOP GR-01-100

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Post-Digestion Spike	One per digestion batch of 20 or fewer samples.	%R must be within 75-125% of expected value.	Apply J flag to associated sample result. Not necessary if analyte concentration < 50 x MDL.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias in matrix	DoD QSM limits of 75-125 %R.

Matrix	IDW samples
Analytical Group	Mercury
Analytical Method/ SOP Reference	SW-846 7470A TriMatrix SOPs GR-01-123

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per digestion batch of 20 or fewer samples.	Must be < ½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Must be < ½ the QL.
LCS	One per digestion batch of 20 or fewer samples.	%R must be within 80%-120%.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DoD QSM limits for %R.
MS	One per digestion batch of 20 or fewer samples.	%R should be within 80%-120%.	Check for errors in calculations and spike preparation. Check un-spiked sample results for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	DoD QSM limits for %R, if sample < 4x spike added. %R should be within 80%-120%..
Duplicate Sample	One per preparatory batch of 20 or fewer samples per matrix.	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Laboratory Supervisor and Data Validator	Precision	The RPD should be ≤ 20% for duplicate samples for both water and soils.

Matrix	Soil samples
Analytical Group	TOC
Analytical Method/ SOP Reference	Walkley-Black Method TriMatrix SOP GR-06-105

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	Result must be <½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Result must be <½ the QL.
LCS	One per batch of 20 or less.	%R must be within 92-138%.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Lab internal limits for %R.
Laboratory Duplicate	One per batch of 20 or less.	RPD must be ≤ 20%, if sample result is great than four times QL.	Check for errors in calculations and preparation. If no errors are found qualify failing results as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision	RPD must be ≤ 20%, if sample result is great than four times QL.

Matrix	IDW samples
Analytical Group	Ignitability
Analytical Method/ SOP Reference	SW-846 1020 TriMatrix SOP GR-18-124

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	NA	NA	NA	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	NA
Laboratory Duplicate	One per batch of 20 or less.	Duplicate result must be within ± 3 ^{o} F.	If sufficient sample is available, reanalyze. If not, or repeated attempt still fails, qualify results as estimated.	Analyst, Laboratory Supervisor and Data Validator	Bias / Precision	Duplicate result must be within ± 3 ^{o} F.

Matrix	IDW samples
Analytical Group	Cyanide Reactivity
Analytical Method/ SOP Reference	SW-846 9014 TriMatrix SOP GR-05-122

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	Result must be <½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Result must be <½ the QL.
LCS	One per batch of 20 or less.	%R must be within 80-120%.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Lab internal limits for %R. %R must be within 80-120%.
MS/MSD	One per batch of 20 or less.	%R should be within 80-120%, RPD should be ≤ 20%	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Lab internal limits for %R and RPD. %R should be within 80-120%, RPD should be ≤ 20%

Matrix	IDW samples
Analytical Group	Sulfide Reactivity
Analytical Method/ SOP Reference	SW-846 7.7.3.4 TriMatrix SOP GR-06-108

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	One per batch of 20 or less.	Result must be <½ the QL.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply B flag to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Result must be <½ the QL.
LCS	One per batch of 20 or less.	%R must be within 29-113%.	Re-prepare and reanalyze all samples with positive results. If insufficient amount of sample is available, apply qualifier to all affected sample results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Lab internal limits for %R. %R must be within 29-113%.
MS/MSD	One per batch of 20 or less.	%R should be within 29-113%, RPD should be ≤ 20%.	Check for errors in calculations and spike preparation. Check un-spiked sample results and surrogate recoveries for possible matrix effects. If no errors are found and the associated LCS in control, matrix effects are the likely cause. Qualify failing analytes as estimated.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Lab internal limits for %R and RPD. %R should be within 29-113%, RPD should be ≤ 20%.

Matrix	IDW samples
Analytical Group	Corrosivity
Analytical Method/ SOP Reference	SW-846 9045C TriMatrix SOP GR-07-113

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPCs)
Method Blank	NA	NA	NA	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	NA
Standard Reference Materials (SRM)	Two per batch of 20 samples or less.	Must be within ± 0.05 pH units.	Repeat calibration and reanalyze all samples. If insufficient volume, qualify results as estimated.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Must be within ± 0.05 pH units.
Laboratory Duplicate	One per batch of 20 or less.	RPD must be ≤ 20%.	If sufficient sample is available, reanalyze. If not, or repeated attempt still fails, qualify results as estimated.	Analyst, Laboratory Supervisor and Data Validator	Bias / Precision	RPD must be ≤ 20%.

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

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents and Records Field Logbook Field Sample Forms Chain-of-Custody Records Sample Shipment Air Bills Equipment Calibration Logs Sampling Notes Drilling Logs Photographs FTMR Forms This UFP-SAP (which includes all Field Sampling SOPs) Site-Specific HASP	Field documents will be maintained in the project file located in the Tetra Tech King of Prussia, Pennsylvania office.
Laboratory Documents and Records Sample Receipt, Custody, and Tracking Record Standards Traceability Logs Equipment Calibration Logs Sample Preparation Logs and Analysis Run Logs Equipment Maintenance, Testing, and Inspection Logs Corrective Action Forms Reported Field Sample Results Reported Results for Standards, QC Checks, and QC Samples Sample Storage and Disposal Records Telephone Logs Extraction/Clean-up Records Raw Data Data Completeness Checklist	Laboratory documents will be included in the hardcopy and portable document format (PDF) deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech King of Prussia, Pennsylvania project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Regulatory Approval of the UFP-SAP Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech King of Prussia, Pennsylvania project file.
Reports All versions of the submitted PCB Investigation Reports	All reports for the PCB Investigation will be stored in hardcopy in the Tetra Tech King of Prussia, Pennsylvania project file and electronically in the server library.

SAP Worksheet No. 30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil	VOCs	See Worksheet No. 18	SW-846 5035/8260B TriMatrix SOPs GR-04-104/105	21 days	Phil Komar Laboratory PM TriMatrix Laboratories, LLC 5560 Corporate Exchange Court SE Grand Rapids, MI 49512 616-846-9528 www.TriMatrixlabs.com	NA
	PCBs		SW-846 3546/8082 TriMatrix SOPs GR-03-128/09-108			
	TPH-GRO		SW-846 5035A/8015B TriMatrix SOP GR-03-121			
	TPH-DRO		SW-846 3550C/8015B TriMatrix SOPs GR-09-123/GR-03-122			
	TOC		Walkley Black Method TriMatrix SOP GR-06-105			
IDW	TCLP VOCs		SW-846 5035/8260B TriMatrix SOPs GR-04-104/105/ GR-01-119			
	TCLP SVOCs		SW-846 1311/8270C TriMatrix SOPs GR-01-119/04-103			
	TCLP Pesticides/ Herbicides		SW-846 1311/8270C TriMatrix SOPs GR-01-119/03-120			
	Total PCBs		SW-846 8082 TriMatrix SOP GR-03-128			
	TCLP Metals		SW-846 1311/6010B/7470A TriMatrix SOPs GR-01-119/01-100/123/140/147			
	Ignitability		SW-846 1030 TriMatrix SOP GR-18-124			
	Corrosivity		SW-846 1110-9045D TriMatrix SOPs GR-07-100/113			
	Reactivity		SW-846 9010C/9014/Chapter 7 7.3.4 TriMatrix SOPs GR-18-106/109/117			
Groundwater	VOCs		SW-846 8260B TriMatrix SOP GR-04-104			
	PCBs		SW-846 3510C/8082 TriMatrix SOPs GR-09-107/03-128			

SAP Worksheet No. 31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Systems Audit	1 per contract year	Internal	Tetra Tech	Person assigned by Tetra Tech QAM, Tetra Tech	PM and FOL, Tetra Tech	Auditor and PM Tetra Tech	CLEAN QAM, Tetra Tech
Laboratory System Audit[1]	Every 3 years	External	DoD ELAP	DoD ELAP Accrediting Body Auditors	Laboratory QA Manager or Laboratory Manager, TriMatrix	Laboratory QAM or Laboratory Manager, TriMatrix	Laboratory QAM or Laboratory Manager, TriMatrix

[1] TriMatrix has successfully completed the laboratory evaluation process required as part of the DoD and NYSDOH ELAPs. The DoD and NYSDOH ELAP certifications are included in Appendix B.

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

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	**Nature of Deficiencies Documentation**	**Individual(s) Notified of Findings** (name, title, organization)	**Timeframe of Notification**	**Nature of Corrective Action Response Documentation**	**Individual(s) Receiving Corrective Action Response** (name, title, organization)	**Timeframe for Response**
Field Sampling System Audit[1]	Audit checklist and written audit finding summary	Rob Sok, PM, Tetra Tech; TBD, FOL, Tetra Tech; John Trepanowski, Program Manager; and Garth Glenn, Deputy Program Manager, Tetra Tech	Dependent on findings, if major a stop work maybe issued immediately, however if minor within one week of audit	Written memo	Tom Johnston, CLEAN QAM, Tetra Tech; Designee, Field Auditor, Tetra Tech; John Trepanowski, Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Jeff Glaser, Laboratory Manager, TriMatrix	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

[1] Audits are scheduled at the Tetra Tech program level and may or may not include this project.

SAP Worksheet No. 33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

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

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

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign each chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The chain-of-custody forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validator. See SOP SA-6.3 (Appendix A).	Internal	FOL and Field Crew, Tetra Tech
	The TriMatrix Laboratory Sample Custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment. The data validator will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	External Internal	1 - Laboratory Sample Custodian, TriMatrix 2 –Data Validator, Tetra Tech
Field SOPs/Field Logs/Sample Collection	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved, particularly that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken chain of custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the SAP was implemented and carried out as written and that any deviations are documented.	Internal	PM, FOL, or designee, Tetra Tech
Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Internal	Laboratory QAM, TriMatrix
Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact Tetra Tech for guidance prior to report preparation.	Internal	Laboratory QAM, TriMatrix
Sample Tables	Proposed samples verified to have been collected.	Internal	FOL and Field Crew, Tetra Tech
Sample Log Sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	Internal	PM, FOL, or designee, Tetra Tech

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Field QC Samples	Verify that field QC samples listed in Worksheet No. 20 were collected as required.	Internal	FOL or designee, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, TriMatrix
	Verify that the data package contains all the elements required by the functional guidelines and scope of work. Missing information will be requested from the laboratory, and validation will be suspended until missing data are received. This occurs as part of the data validation process.	External	Data Validator, Tetra Tech
Electronic Data Deliverables	The electronic data will be compared to the chain-of-custody form and hard copy data package to verify accuracy and completeness.	External	Data Validator, Tetra Tech

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

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

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Sample Coordinates	Sample locations will be validated to be correct and in accordance with the SAP (compare map of proposed locations to map of actual locations).	PM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded.	Data Validator, Tetra Tech
IIa	Holding Times	Verify that the samples were shipped and stored at the required temperature and that the sample pH values for chemically preserved samples meet the requirements listed in Worksheet No. 19. Verify that the analyses were performed within the holding times listed in Worksheet No. 19.	Data Validator, Tetra Tech
IIa	Data Results	Verify that the summary form results match the raw data.	Data Validator, Tetra Tech
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet No. 28 were analyzed and that the MPCs listed in Worksheet No. 12 were met for all field samples and QC analyses. Verify that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Data Validator, Tetra Tech
IIa/IIb	Field and Laboratory Duplicate Analyses for Precision	Verify the field sampling precision by checking the RPD for field duplicate samples. Verify laboratory precision by checking RPDs or %D values from laboratory duplicate, MS/MSD, MS/Sample Duplicate, and LCS/LCSD analyses. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet Nos. 12 and 28.	Data Validator, Tetra Tech
IIa/IIb	Sample Results for Representativeness	Verify that the laboratory recorded the temperature of each sample at sample receipt and the pH of chemically preserved samples to ensure sample integrity from sample collection to analysis.	Data Validator, Tetra Tech
IIa/IIb	Project Screening Levels	Discuss the impact of matrix interferences or sample dilutions performed, because of the high concentration of one or more contaminants, on the other target compounds reported as not detected. Document this usability issue and inform the Tetra Tech PM.	Data Validator, Tetra Tech

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
		Review and add PSLs to the laboratory electronic data deliverable. Flag samples and notify the Tetra Tech PM of samples that exceed the PSLs as listed in Worksheet No. 15.	PM or designee, Tetra Tech
IIa/IIb	Data Validation Report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all data qualifications. Print a copy of the project database, qualified data depicting data qualifiers, and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	Data Validator, Tetra Tech
IIa, IIb	SAP QC Sample Documentation	Verify that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Verify that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech Project Chemist or PM.	Data Validator, Tetra Tech
IIa, IIb	Documentation of Analytical Reports for Completeness	Ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet No. 36. Verify all data have been transferred correctly and completely to the final SQL database.	Data Validator, Tetra Tech
IIb	Project Quantitation Limits for Sensitivity	Verify that the QLs listed in Worksheet No. 15 were achieved.	Data Validator, Tetra Tech
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods, SOP requirements, and matrix interferences on the analytical results.	Data Validator, Tetra Tech

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

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil and Groundwater	VOCs and PCBs	A (Level III) data validation will be performed using criteria for SW-846 Methods 8260B and 8082 listed in Worksheet Nos. 12, 15, 24, and 28, and the DoD QSM. If not included in the aforementioned, the logic outlined in the National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) will be used to apply qualifiers to data.	Data Validator, Tetra Tech
IIa and IIb	Soil	TPH-GRO and TPH-DRO	A (Level III) data validation will be performed using criteria for SW-846 Method 8015B listed in Worksheet Nos. 12, 15, 24, and 28, and the DoD QSM. If not included in the aforementioned, the logic outlined in the National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) will be used to apply qualifiers to data.	Data Validator, Tetra Tech
IIa and IIb	Soil	TOC	A (Level III) data validation will be performed using criteria for the Walkley Black Method listed in Worksheet Nos. 12, 15, 24, and 28, and the DoD QSM to the extent possible. If not included in the aforementioned, the logic outlined in the National Functional Guidelines for Inorganic Data Review (October 2004) will be used to apply qualifiers to data to the extent possible.	Data Validator, Tetra Tech

IDW data will not be validated.

SAP Worksheet No. 37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

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

Completeness

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the project team will prepare a table comparing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and technical staff will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions. The completeness goal for this project is 90%.

Precision

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets 12 and 28 for 25% of the data. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet 28 for 25% of the data. This assessment will include an evaluation of field and laboratory contamination, instrument calibration variability, and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

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

Comparability

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

Sensitivity

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet No. 15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be

described. The Tetra Tech Project Chemist will enlist the help of the Project Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

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

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

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. A statistical evaluation of the data will not be performed because a biased sampling design was employed that does not support the use of statistics in assessing whether DQOs were attained.

Field Screening/Laboratory PCB Data Correlation

The Tetra Tech Project Statistician will evaluate the correlation of field PCB data to laboratory data for Total PCBs. Factors considered in this evaluation will include the magnitude of the slope and intercept of the correlation equation, the distribution of data points across the plotted concentration range, and the value of the correlation coefficient (commonly denoted as “r”). If the coefficient is less than 0.65 or the plotted data do not appear to be well correlated in accordance with standard statistical principles, limitations on the use of the data will be described in the project report.

The field data will be plotted against the laboratory data (Total PCBs will be determined), and a linear least-squares fit will be applied to generate an estimate of the “best” line through the data. If the data set exhibits more than one region where there appears to be a linear fit to the data but the regions have different slopes, the data may be subdivided into separate regions for fitting. No more than three separate regions of the correlation will be fit in this manner, and as few regions as possible will be used to adequately represent a linear fit. If a correlation coefficient of 0.65 or greater is obtained for a region, the fit will be considered adequate to translate field PCB results to the equivalent laboratory Total PCB concentrations with confidence. A correlation coefficient less than 0.65 will be considered unacceptable for making this translation. An ideal correlation coefficient would be 1.0. Concentrations used in the correlation plot will be limited to values greater than the QL.

The use of subdivisions to represent a best linear fit will be most important for concentrations in the range of about 5 to 60 mg/kg, because the 10 mg/kg screening value for PCBs is within this range.

Identify the personnel responsible for performing the usability assessment:

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

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

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

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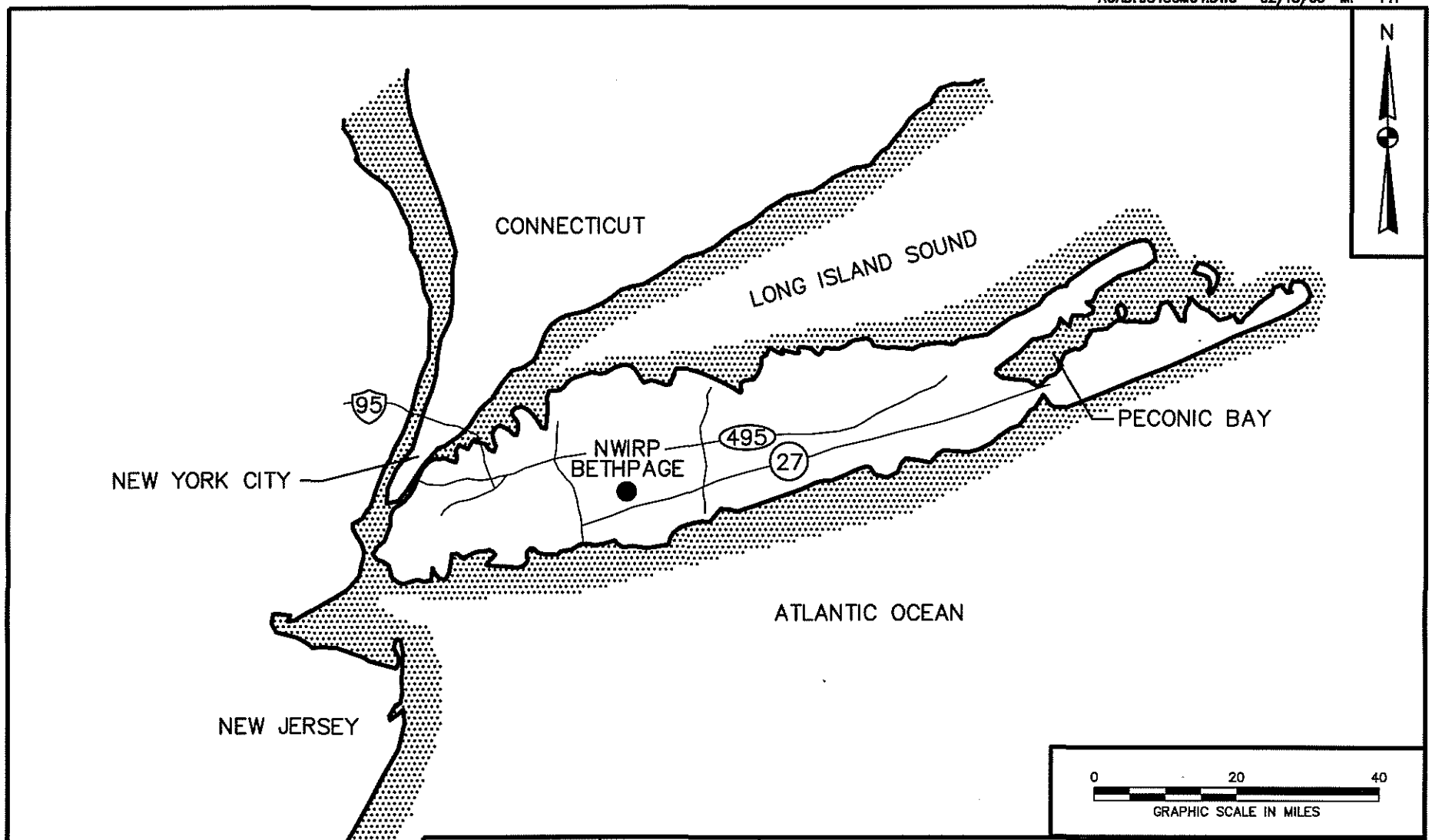
FIGURES

ACAD: 9845CM04.DWG 02/15/06 MF PIT

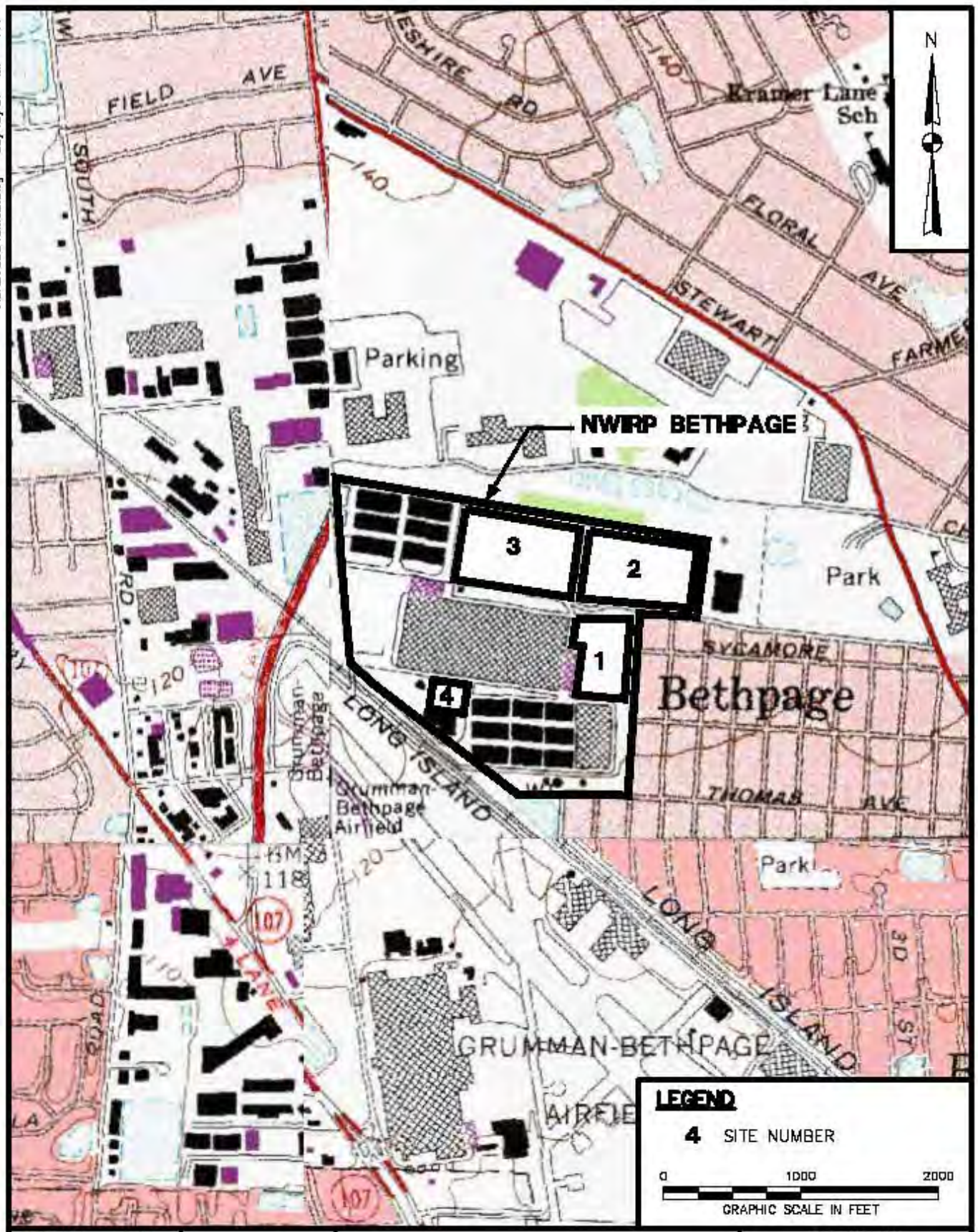


DRAWN BY MF	DATE 12/9/05	Tetra Tech NUS, Inc.	GENERAL LOCATION MAP ESD NWIRP BETHPAGE BETHPAGE, NEW YORK
CHECKED BY	DATE		
REVISED BY	DATE		
SCALE AS NOTED			

CONTRACT NO. 9845	
OWNER NO. 0002	
APPROVED BY	DATE
DRAWING NO. FIGURE 1	REV. 0

FORM CADD NO. TTNUS-AH.DWG - REV 1 -9/10/98

ACAD:0604CM02.dwg 06/13/07 MF PIT



DRAWN BY MF	DATE 6/13/07	Tetra Tech NUS, Inc.	CONTRACT NO. 0604
CHECKED BY	DATE	SITE LOCATION MAP SITE 1 NWIRP BETHPAGE BETHPAGE, NEW YORK	OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE AS NOTED			DRAWING NO. FIGURE 2 / REV. 0

FORM CADD NO. TTNUS-AV.DWG - REV 1 -9/10/98

112G02019/0410/112G02019GM04-1.DWG 01/12/10 MKB

TETRA TECH NUS, INC.

PCBs CONCENTRATIONS
0 – 2 FEET BGS
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GM04–1	AS NOTED
FIGURE NUMBER: FIGURE 3	REV: 0 / DATE: 01/12/10

112G02019/0410/112G02019GM04-2.DWG 01/13/10 MKB

TETRA TECH NUS, INC.

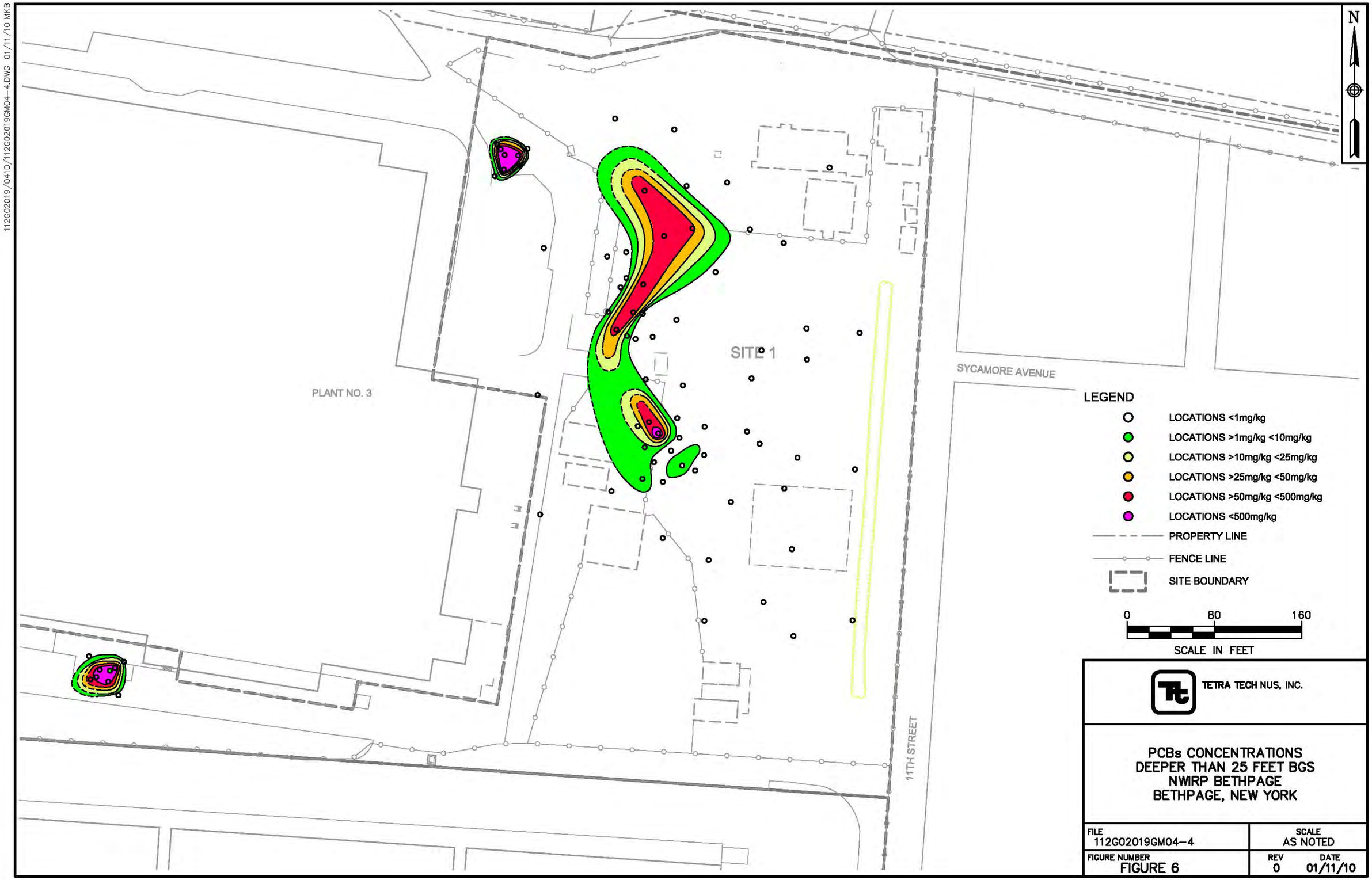
PCBs CONCENTRATIONS
2 – 15 FEET BGS
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GM04–2	AS NOTED

FIGURE NUMBER	REV	DATE
FIGURE 4	0	01/12/10

112G02019/0410/112G02019GM04-3.DWG 01/11/10 MKB

112G02019/0410/112G02019GM04-4.DWG 01/11/10 MKB



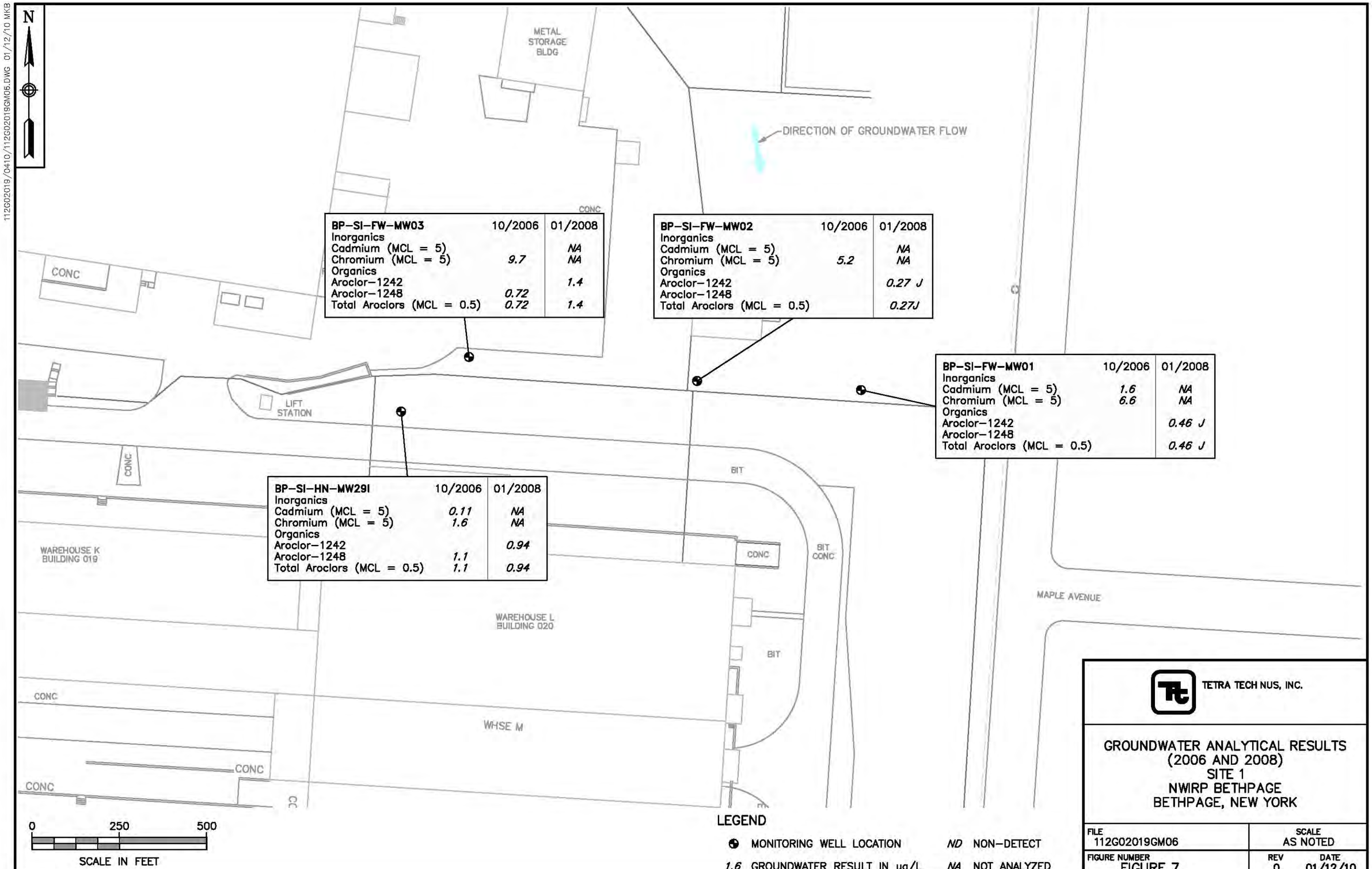
TETRA TECH NUS, INC.

PCBs CONCENTRATIONS
DEEPER THAN 25 FEET BGS
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GM04-4	AS NOTED

FIGURE NUMBER	REV	DATE
FIGURE 6	0	01/11/10

112G02019/0410/112G02019GM06.DWG 01/12/10 MKB



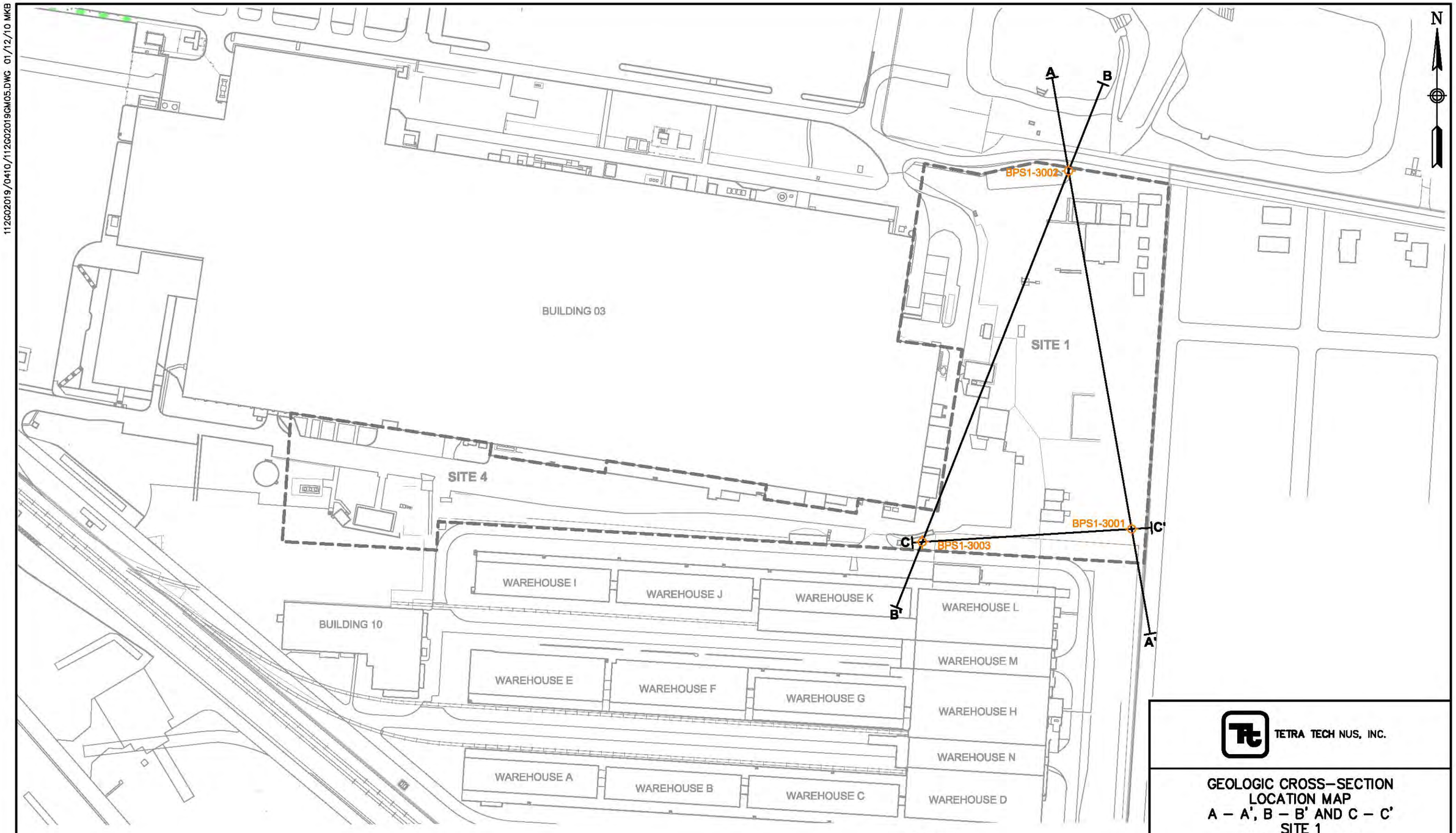
BP-SI-FW-MW03	10/2006	01/2008
Inorganics		
Cadmium (MCL = 5)		NA
Chromium (MCL = 5)	9.7	NA
Organics		
Aroclor-1242		1.4
Aroclor-1248	0.72	
Total Aroclors (MCL = 0.5)	0.72	1.4

BP-SI-FW-MW02	10/2006	01/2008
Inorganics		
Cadmium (MCL = 5)		NA
Chromium (MCL = 5)	5.2	NA
Organics		
Aroclor-1242		0.27 J
Aroclor-1248		
Total Aroclors (MCL = 0.5)		0.27J

BP-SI-FW-MW01	10/2006	01/2008
Inorganics		
Cadmium (MCL = 5)	1.6	NA
Chromium (MCL = 5)	6.6	NA
Organics		
Aroclor-1242		0.46 J
Aroclor-1248		
Total Aroclors (MCL = 0.5)		0.46 J

BP-SI-HN-MW29I	10/2006	01/2008
Inorganics		
Cadmium (MCL = 5)	0.11	NA
Chromium (MCL = 5)	1.6	NA
Organics		
Aroclor-1242		0.94
Aroclor-1248	1.1	
Total Aroclors (MCL = 0.5)	1.1	0.94

112G02019/0410/112G02019GM05.DWG 01/12/10 MKB



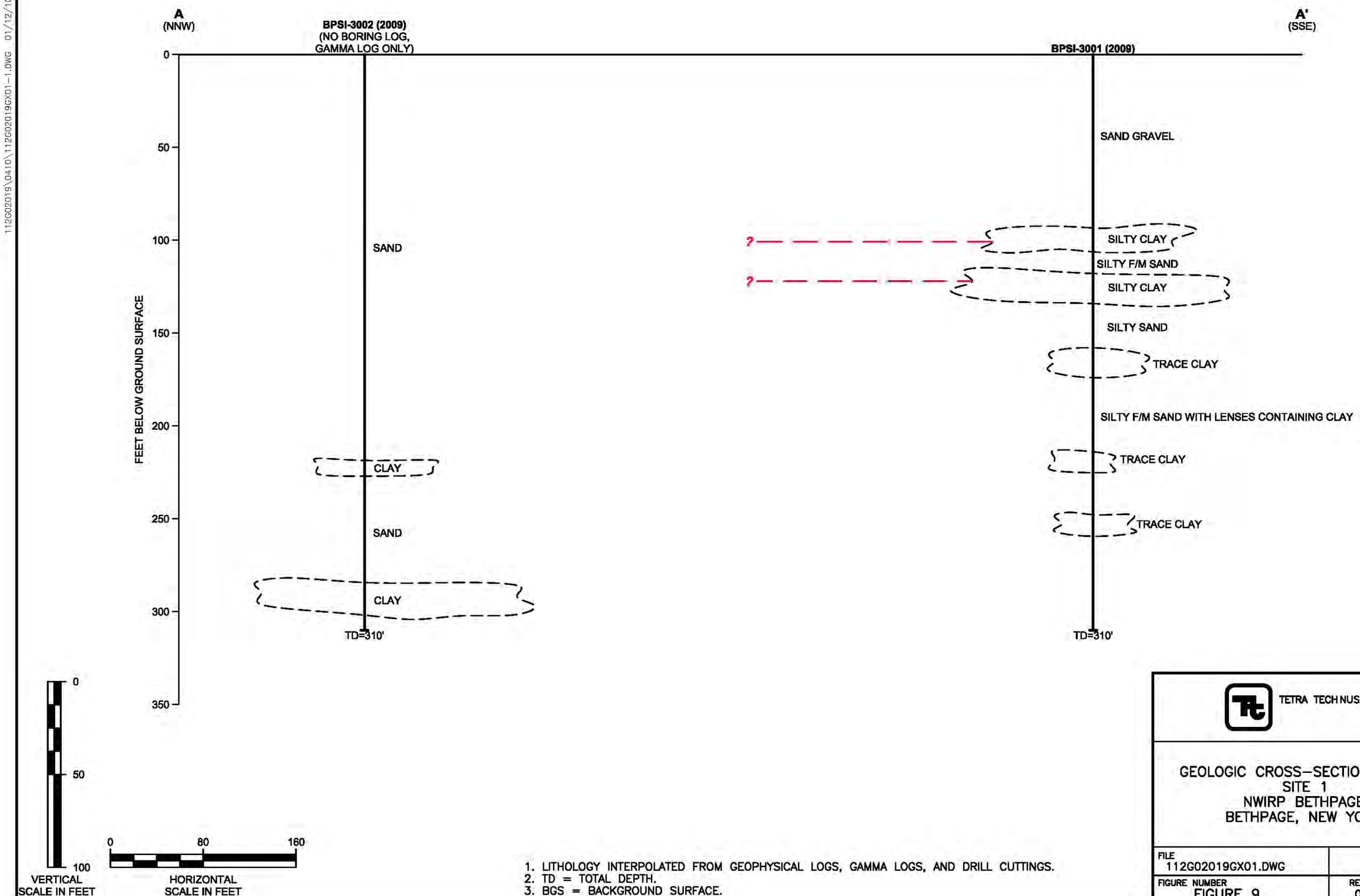
TETRA TECH NUS, INC.

GEOLOGIC CROSS-SECTION
LOCATION MAP
A – A', B – B' AND C – C'
SITE 1
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GM05	AS NOTED

FIGURE NUMBER	REV	DATE
FIGURE 8	0	01/12/10

112G02019\0410\112G02019GX01-1.DWG 01/12/10 MKB



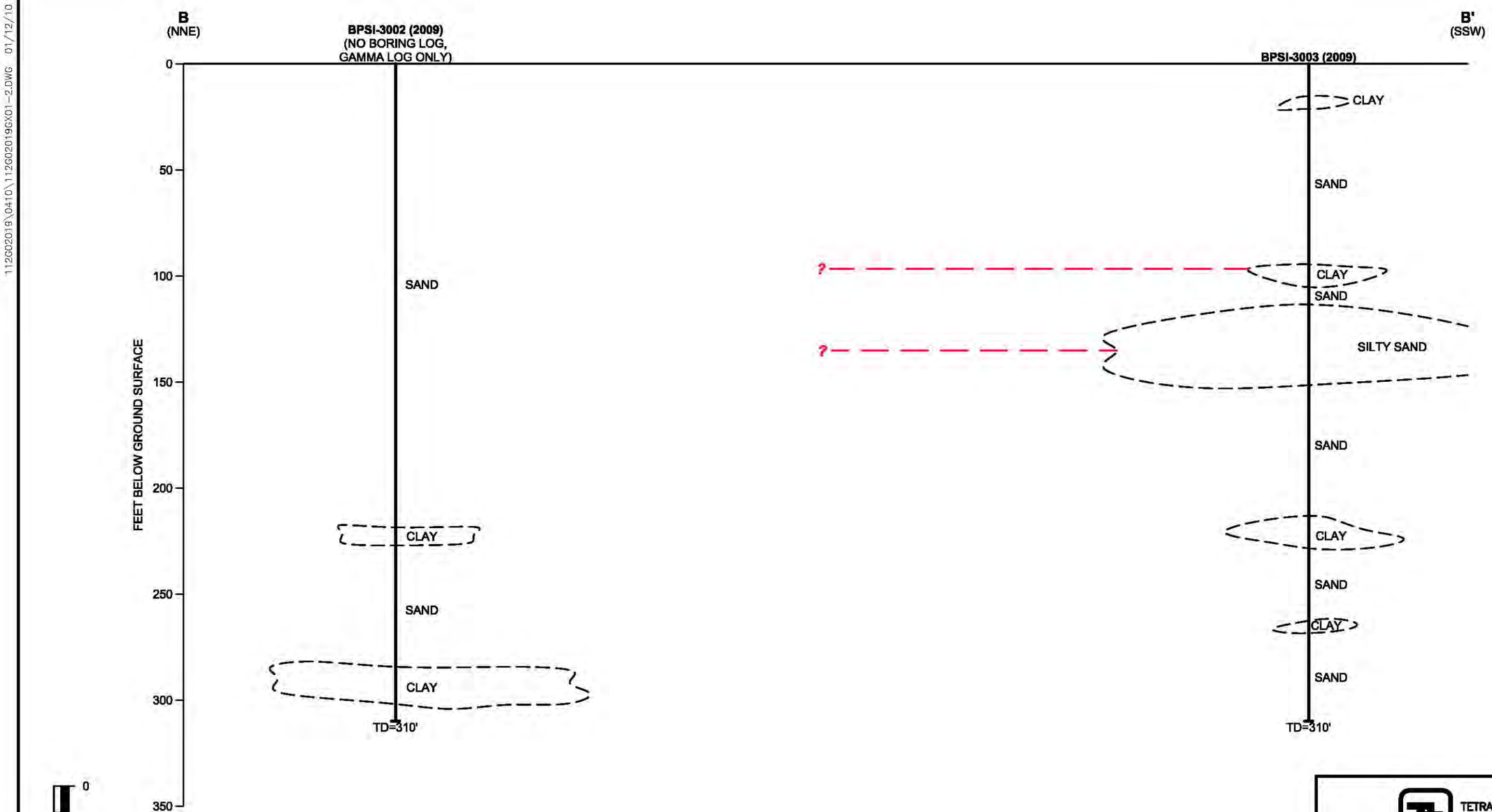
1. LITHOLOGY INTERPOLATED FROM GEOPHYSICAL LOGS, GAMMA LOGS, AND DRILL CUTTINGS.
2. TD = TOTAL DEPTH.
3. BGS = BACKGROUND SURFACE.

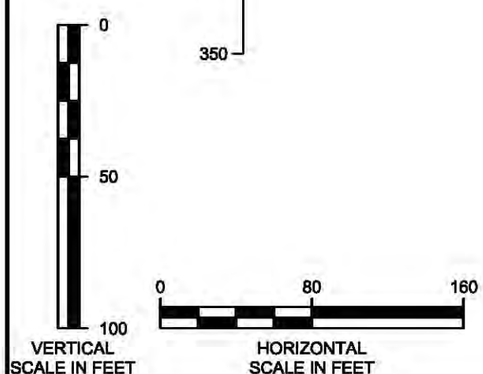
TETRA TECH NUS, INC.

GEOLOGIC CROSS-SECTION A – A'
SITE 1
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GX01.DWG	AS NOTED
FIGURE NUMBER	REV / DATE
FIGURE 9	0 / 01/12/10

112G02019\0410\112G02019GX01-2.DWG 01/12/10 MKB





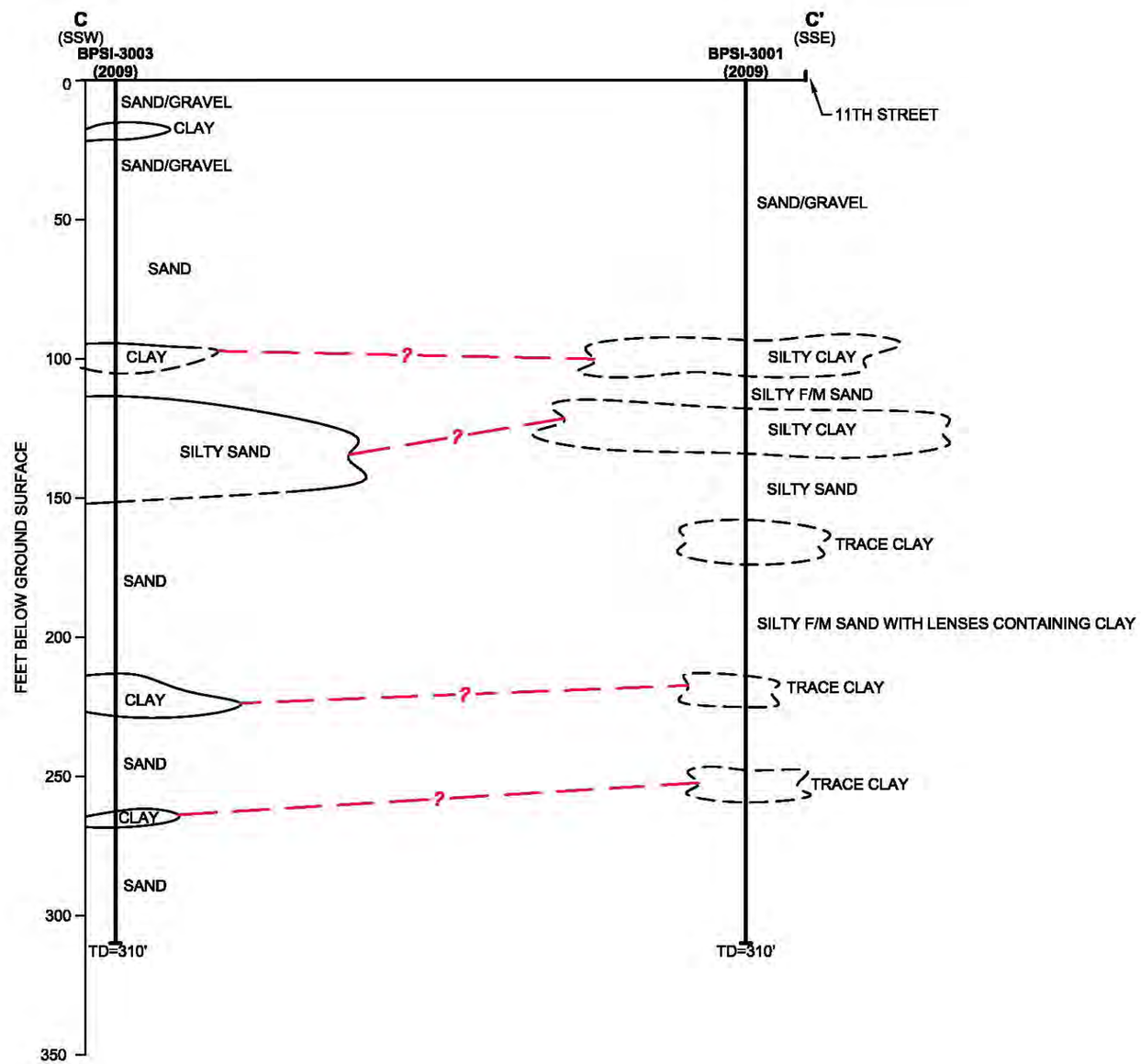
1. LITHOLOGY INTERPOLATED FROM GEOPHYSICAL LOGS, GAMMA LOGS, AND DRILL CUTTINGS.
2. TD = TOTAL DEPTH.
3. BGS = BACKGROUND SURFACE.

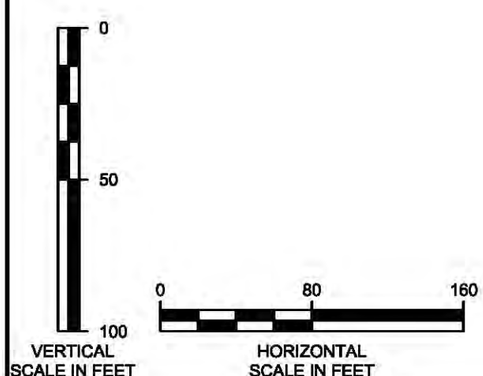
TETRA TECH NUS, INC.

GEOLOGIC CROSS-SECTION B – B'
SITE 1
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GX01-2.DWG	AS NOTED
FIGURE NUMBER	REV / DATE
FIGURE 10	0 / 01/12/10

112G02019\0410\112G02019GX01-2.DWG 01/13/10 MKB





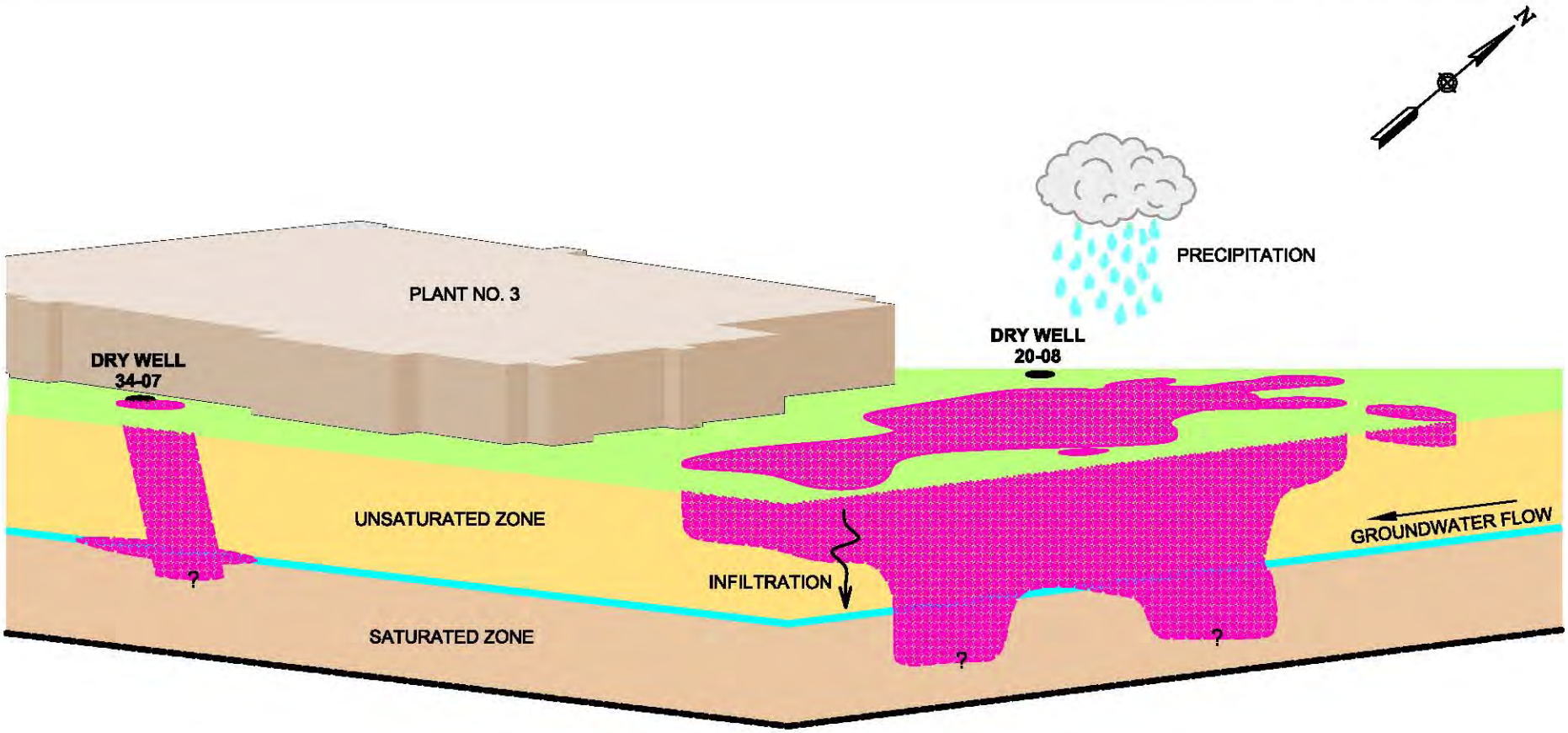
1. LITHOLOGY INTERPOLATED FROM GEOPHYSICAL LOGS, GAMMA LOGS, AND DRILL CUTTINGS.
2. TD = TOTAL DEPTH.
3. BGS = BACKGROUND SURFACE.

TETRA TECH NUS, INC.

GEOLOGIC CROSS-SECTION C – C'
SITE 1
NWIRP BETHPAGE
BETHPAGE, NEW YORK

FILE	SCALE
112G02019GX01-3.DWG	AS NOTED
FIGURE NUMBER	REV / DATE
FIGURE 11	0 / 01/13/10

112G02019/0410/112G02019CS01.DWG 01/11/10 MKB



LEGEND

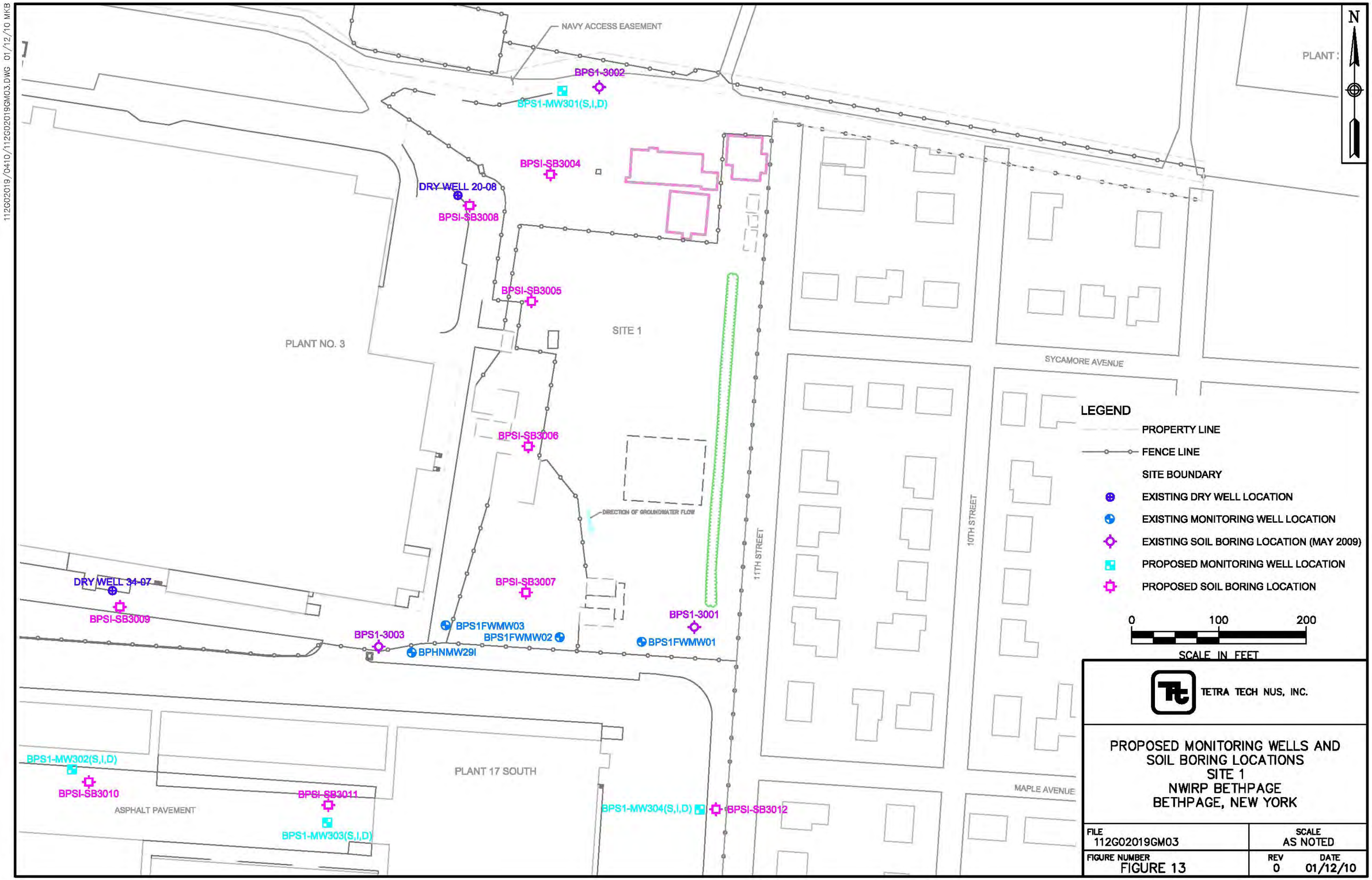
- PCB CONTAMINATED SOILS
- UNSATURATED ZONE
- SATURATED ZONE
- INFERRED CLAY UNIT

TETRA TECH NUS, INC.

CONCEPTUAL SITE MODEL
SITE 1 – FORMER DRUM MARSHALLING AREA
NWIRP BETHPAGE
BETHPAGE, NEW YORK

SCALE	AS NOTED
FILE	112G02019CS01
REV	0
DATE	01/11/10
FIGURE NUMBER	FIGURE 12

112G02019/0410/112G02019GM03.DWG 01/12/10 MKB



APPENDIX A

TETRA TECH SOPS

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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:
CTO:
Project Manager:
Requestor:
Program/Client:
State/EPA Region:

Request Date:
Date Data Available for Production:
Request in Support of:
Database Lead:
GIS Lead:
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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ATTACHMENT

A DRILLING EQUIPMENT SIZES 25

FIGURE

NUMBER **PAGE**

1 STANDARD SIZES OF CORE BARRELS AND CASING 20

1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.

- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.

- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.

- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotosonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

5.2.2 Continuous-Flight Solid-Stem Auger Drilling

This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous-flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.

There would be very few situations where use of a solid-stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.

5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

5.2.4 Rotosonic Drilling

The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.

The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

5.2.5 Reverse Circulation Rotary Drilling

The common reverse-circulation rig is a water or mud-rotary rig with a large-diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud-rotary). This type of rig is used for the construction of large-capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large-diameter hole which is created. A few special reverse-circulation rotary rigs are made with double-wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.

Advantages of the latter method include:

- The formation water is not contaminated by the drilling water.
- Formation samples can be obtained, from known depths.
- When drilling with air, immediate information is available regarding the water-bearing properties of formations penetrated.
- Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air-rotary rig.

Disadvantages include:

- Double-wall, reverse-circulation drill rigs are rare and expensive to operate.
- Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.

5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:

  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."

- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.

- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.

- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.

- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

- Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

- Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.

- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).

- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.

- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.

- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.

- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations			Casing Coupling					Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings	Casing O.D., Inches	O.D., Inches	I.D., Inches	Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	---	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	---	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	---	5.500			5.650	---	---	---	---
SW	---	6.625			6.790	---	---	---	---
UW	---	7.625			7.800	---	---	---	---
ZW	---	8.625			8.810	---	---	---	---
---	AX __|__\	---	---	---	---	1.875	1.750	1.000	---
---	BX __|__\	---	---	---	---	2.345	2.250	1.437	---
---	NX __|__\	---	---	---	---	2.965	2.813	1.937	---

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	---
	7 1/4	6 1/4	3 1/4	---
	13 1/4	12	6	---
Thin Wall Tube Samplers (Ref. 7)	---	2	1 7/8	---
	---	2 1/2	2 3/8	---
	---	3	2 7/8	---
	---	3 1/2	3 3/8	---
	---	4 1/2	4 3/8	---
	---	5	4 3/4	---
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

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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.

Remarks: _____

Drilling Area Background (ppm): ____

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					HINE-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	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)		
GRAVELS (More than Half of Coarse Fraction is Larger than No. 4 Sieve Size)	CLEAN GRAVELS (Little or No 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.	SILTS AND CLAYS Liquid Limit Less than 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.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.		Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	GRAVELS WITH FINES (Appreciable Amount of Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.		Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.	SILTS AND CLAYS Liquid Limit Greater Than 50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
SANDS (More than Half of Coarse Fraction is Smaller than No. 4 Sieve Size)	CLEAN SANDS (Little or No Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.		High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.		Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
	SANDS WITH FINES (Appreciable Amount of Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.	HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.						

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	5-9
Medium Loose	10-29
Dense	30-49
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 0.5	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.5 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 thumb-nail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumb-nail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Terms	Scratcher or Knife Effects	Hammer Effects	Descriptive Terms	Abbreviation	Spacing
Soft	Easily Gouged	Crushes when pressed with hammer	Very Broken	(V.Br.)	0-2"
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges	Broken	(Br.)	2"-1'
Medium Hard	Can be scratched	Breaks (one blow); sharp edges	Blocky	(Bl.)	1'-4'
Hard	Cannot be scratched	Breaks only with difficulty (several blows); sharp edges	Massive	(Ma)	4'-8'

LEGEND:

SOIL SAMPLES - TYPES	ROCK SAMPLES - TYPES	CORE LOSS
1-3" Split Barrel Sample	2-NQ (Conventional) Core (2-1/8" O.D.)	0/9
3-5½" O.D. Undisturbed Sample	3-HQ (Diamond) Core (3-1/8" O.D.)	▽ ▽ ▽ Initial Loss of water & Depth
0 - Other Samples, Specify in Remarks	4 - Other Core Sizes, Specify in Remarks	0/9
		▽ ▽ ▽ Stabilized Loss of water & 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

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

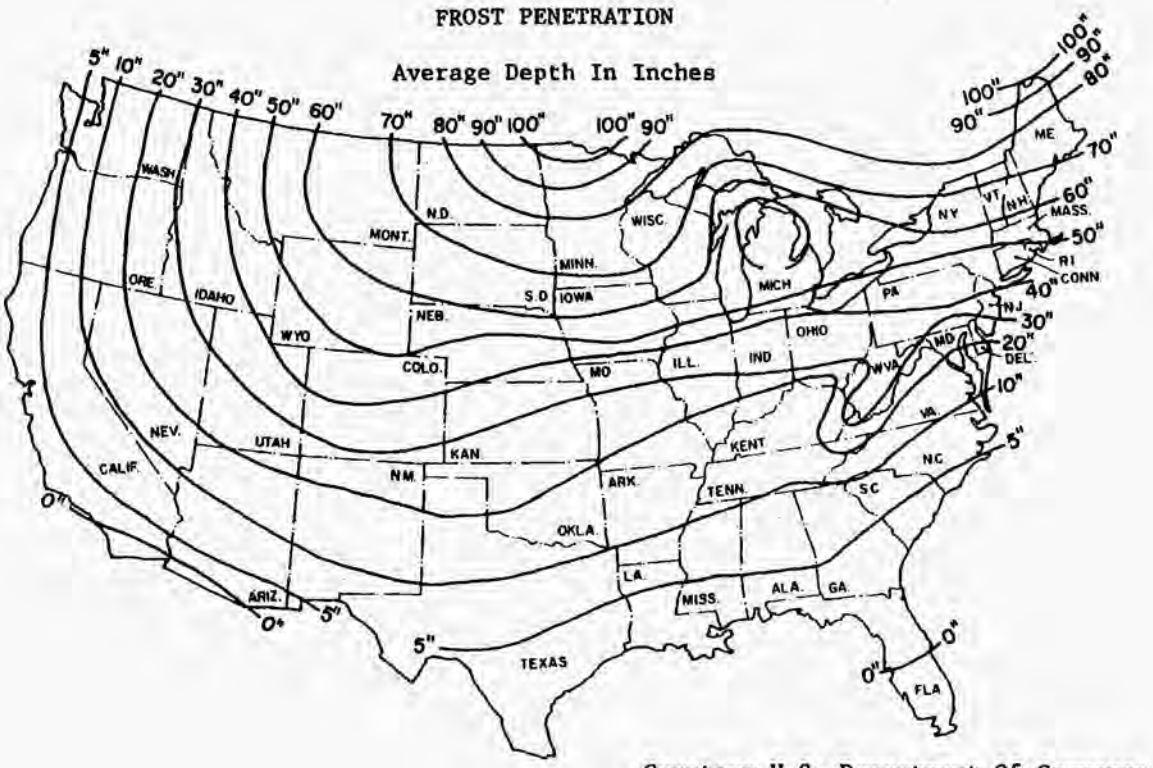
British Columbia
BC One Call
1-800-474-6886

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.

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – 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 collected.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol ($^0/_{00}$) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

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 (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.
General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

Knee injuries – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.

- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.

- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.

- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.

- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.

- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.

- Wear high-visibility vests to increase visual recognition by motorists.

- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.

- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

CAUTION

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

> ***CAUTION***
> Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.

2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.

3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

> ***CAUTION***
> For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:

   - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.

   - While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
  - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
  - pH Paper
  - Camera and film (if appropriate)
  - Appropriate keys (for locked wells)
  - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.
- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).

2. Determine well or inner casing diameter.

3. Measure and record static water level (depth below ground level or top of casing reference point).

4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.

5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).

6. Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

   where: V = Static volume of well in gallons.
   T = Linear feet of water in the well.
   r = Inside radius of well casing in inches.
   0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.

2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.

3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

6.4 Evacuation of Static Water – Purging

6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.
- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.

- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters

used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

6.5.1 Measurement of pH

6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)

- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

6.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.

2. Dip the pH paper into this small portion of sample.

3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.

4. Record the pH value from the chart on the sampling log sheet.

5. Discard the used pH paper as trash.

6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

6.5.2 Measurement of Specific Conductance

6.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

6.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

6.5.3 Measurement of Temperature

6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.

2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.

2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.

3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

6.5.4 Measurement of Dissolved Oxygen

6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between

the two metals, reduction of oxygen to hydroxide ion (OH^-) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.

8. Rinse the probe with deionized water.

9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

6.5.5 Measurement of Oxidation-Reduction Potential

6.5.5.1 General

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

6.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

6.5.5.3 Equipment

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

2. Thoroughly rinse the electrode with deionized water.

3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.

4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

6.5.6 Measurement of Salinity

6.5.6.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

6.5.6.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

6.5.6.3 Equipment

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.6.4 Measurement Techniques for Salinity

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.

2. Check batteries and calibrate the meter before going into the field.

3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.

5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.

6. Rinse the probes with deionized water.

6.5.7 Measurement of Turbidity

6.5.7.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

6.5.7.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

6.5.7.3 Equipment

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Calibration solution and sample tubes, as specified by the manufacturer.

- Manufacturer's operation manual.

6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.

2. Check batteries and calibrate the instrument before going into the field.

3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.

4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.

5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.

6. Immerse the electrode in the sample and measure the turbidity.

7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.

8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.

9. Rinse the electrode or test cell with deionized water.

6.6 Sampling

6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.

- Brief description of area and waste characterization.

- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).

- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.

2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases form contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:

   - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
   - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
   - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

   Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler

during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.

4. Calculate volume of well water to be removed as described in Section 6.3.

5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.

6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water was as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.

7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.

8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.

9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.

10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.

11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this

occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.

14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.

15. Process sample containers as described in SOP SA-6.1.

16. Decontaminate equipment as described in SOP SA-7.1.

6.7 Low-Flow Purging and Sampling

6.7.1 Scope and Application

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

6.7.2 Equipment

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).

- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.

- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).

2 Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.

3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.

4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well

5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.

7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.

9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.

10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.

11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:

    - pH ±0.2 standard units
    - Specific conductance ±10%
    - Temperature ±10%
    - Turbidity less than 10 NTUs
    - DO ±10%

12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.

NOTE: VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.

- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.

- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.

- Prepare samples for shipping as per SOP SA-6.1.

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ATTACHMENT A

PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	$300-400	Requires compressed gas; piezometric level indicator; other materials available.

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

- PE Polyethylene
- PP Polypropylene
- PVC Polyvinyl chloride
- SS Stainless steel
- PC Polycarbonate
- EPDM Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

- NA Not applicable
- AC Alternating current
- DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

ATTACHMENT B
GROUNDWATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[ ] Domestic Well Data
[x] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[X] Low Concentration
[ ] High Concentration

SAMPLING DATA:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	ORP	Other
Time:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	mV	NA
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	ORP	Other
Method:								
Monitor Reading (ppm):			SEE LOW FLOW PURGE DATA SHEET					
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

ATTACHMENT C
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

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

ATTACHMENT D
LOW FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: ____________ WELL ID: ____________

PROJECT NUMBER: ____________ DATE: ____________

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): ____________ PAGE___OF___

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that 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 shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (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 (e.g., 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.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – 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 collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

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 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine 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).

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.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting 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) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection 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 Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

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 that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed 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 sampling operations, particularly noting sampling locations, depths, 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. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, 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 after collection. In addition, to the extent possible, vegetation should be removed from the sample.

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. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

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.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be

obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4˚C ± 2 ˚C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

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.

After the Encore™samples are collected, they should be placed on ice immediately and delivered 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.

6.2.1.2 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) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall 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 shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.

2. Pull the plunger back and insert the syringe into the soil to be sampled.

3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.

4. Weigh the sample and adjust until obtaining the required amount of sample.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. 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.

9. 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):

> ***CAUTION***
> Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

> ***NOTE***
> If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.

2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.

3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.

4. Transfer the mixed soil to the appropriate sample containers and close the containers.

5. Label the sample containers in accordance with SOP SA-6.3.

6. Place the containers in a cooler of ice as soon after collection as possible.

7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

NOTE
Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4˚C.

NOTE
Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

NOTE
Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil 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 soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

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) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

   - Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

REMEMBER
If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential 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.

CAUTION
The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is 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.

4. 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. In addition, the check valve maintains a positive suction within the tube to help retain the sample.

5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.

6. 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 with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. 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.

7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.

8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.

11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.

12. Mark the "up" direction on the side and upper end of the tube with indelible ink.

13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).

14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity 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.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

> ***NOTE***
> Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.
>
> For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:
>
> - Surface soil - 0 to 6 inches bgs
> - Near-surface soil - 6 to 18 inches bgs
>
> If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon's or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- 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 (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. 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.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required 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 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to 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 either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

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 in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.

6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.

7. 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 to be smaller in diameter than the bucket bit employed to initiate the borehole.

8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.

11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.

13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.

14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).

- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.

- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.

- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.

- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists 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-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

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 (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

2. Lower the sampler into the borehole inside the hollow stem auger bits.

3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.

4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.

5. Detach the sampler from the drill rods.

6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

> ***CAUTION***
> Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.

8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) 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.

9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings where encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).

10. 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.

11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).

12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from 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 required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-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 soil data 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.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed 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 power equipment to permit detailed descriptions 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 provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

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 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, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

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 required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. 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 soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. 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.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log 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 as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

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 includes 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 that 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.

6.8.3.2 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 bottle ware 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).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on 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:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.
- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that 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 after it has been deposited on the ground, as follows:

  a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.

  b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.

  c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.

  d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.

  e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.

  f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.

- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be 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.

> ***CAUTION***
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:

  a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.

  b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

  c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

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 soil 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 are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine 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 oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable trough soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall 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.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e,g., sledge hammer) or pushing (e.g., backhoe bucket) equipment 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 soil 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: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled 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 tube 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 6.8.3.4 shall be followed. The thin-walled 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 backhoe may be used to drive or push the 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. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 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 may photograph, if required by the project-specific work plan, 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. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

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.

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. 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. If the project-specific work plan requires photographs, 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 if required
- Groundwater levels
- PID/FID/LEL/O_2 meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists

- Soil type classification

7.0 REFERENCES

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.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

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:

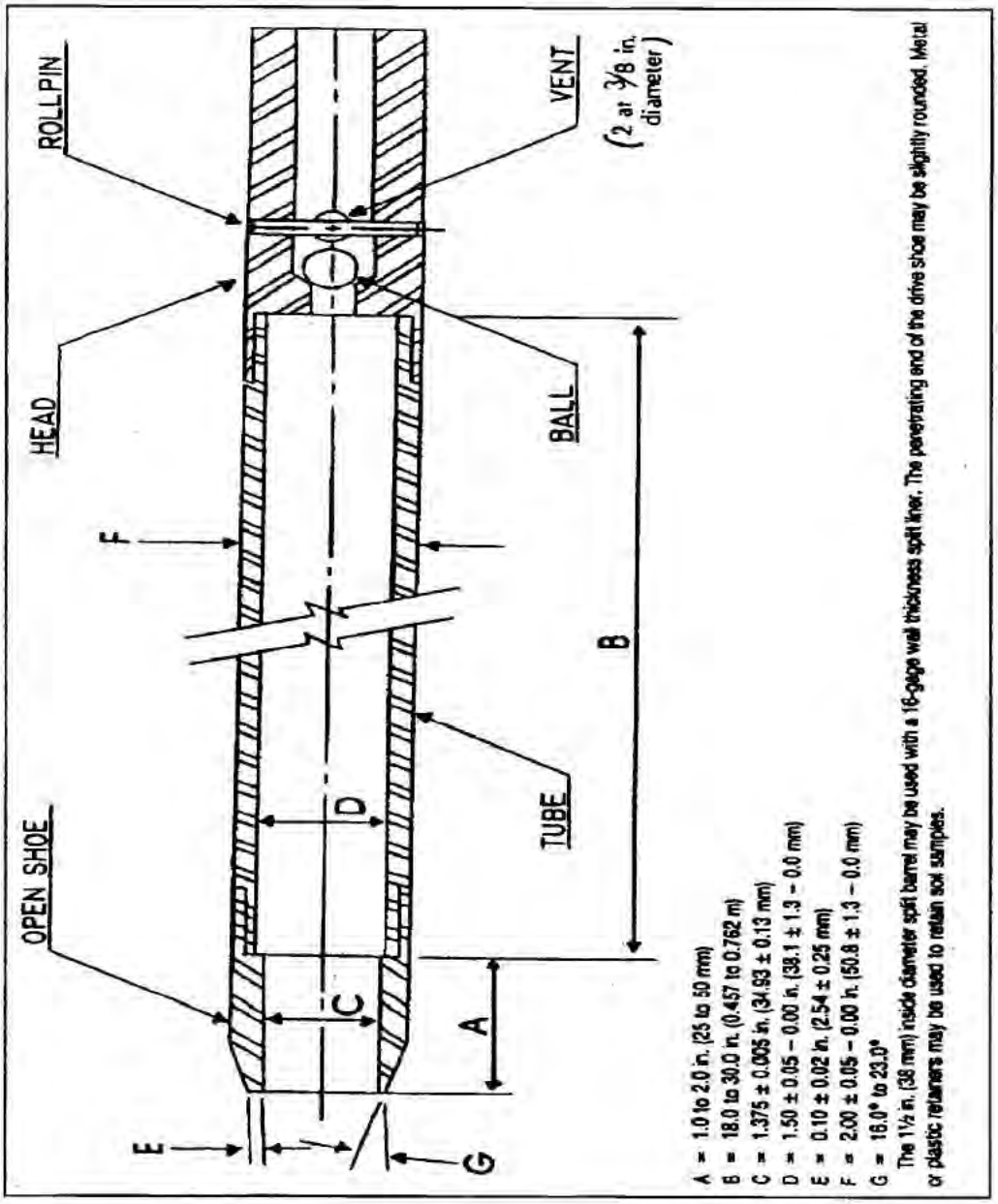
Circle if Applicable:

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 = 18.0° 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: _____

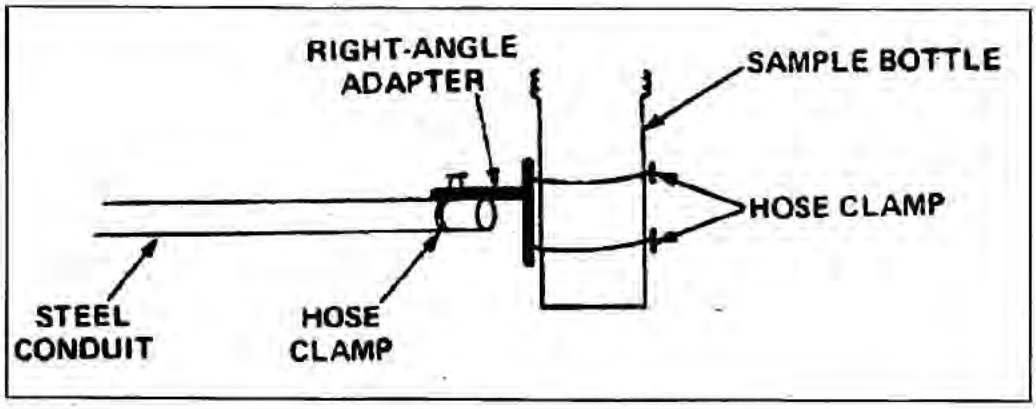
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

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 provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

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

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium- to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

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

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

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U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

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ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon®
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton®*	Silicone	Neoprene	Teflon®*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

1 Teflon®
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton®
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

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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 (y261.30 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 (y261.20 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 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, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS 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 AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM 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 FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

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 on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS 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, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions 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 because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks 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 TtNUS 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, subject to 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 SOPs.

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. Complete a sample log sheet 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. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody 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 as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and 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 that 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. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets 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

Complete a Groundwater Level Measurement Sheet 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. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.

5.3.2.3 Packer Test Report Form

Complete a Packer Test Report Form for each well at which a packer test is conducted.

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

Complete a Monitoring Well Construction Details Form 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

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools 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 ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, 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. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site 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 used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that 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 DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- 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
SAMPLE LABEL

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
CHAIN-OF-CUSTODY RECORD FORM

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

CONTAINER TYPE PLASTIC (P) or GLASS (G)
PRESERVATIVE USED
TYPE OF ANALYSIS

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									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

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in 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) - 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 College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For 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 - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is 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. Improper or incomplete decontamination is 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.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. 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 soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.

  - Avoidance of areas where control/custody cannot be maintained.

  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.

  - Avoidance of potentially contaminated areas.

  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.

- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet 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. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.

  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil 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. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.

  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.

  - Liner – Depending on the types of equipment and decontamination method to be used, 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. The desired thickness may be achieved through layering materials of lighter construction. 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 shall be applied to provide traction as necessary.

  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:

  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is 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 environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and 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. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 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.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents –
> Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

> ***SAFETY REMINDER***
> Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

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. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great 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 if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  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 ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole 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. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 ˚F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0˚ tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40˚ fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to 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 loose soil from the equipment through manual means.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.

3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.

5. Rinse the equipment with deionized water.

6. To the extent possible, allow components to air dry.

7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.

8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device

- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.

- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 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.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

NOTE
Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

NOTE
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.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.

   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.

   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.

   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.

   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.

   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of 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 unless specified differently in the project-specific planning documents:

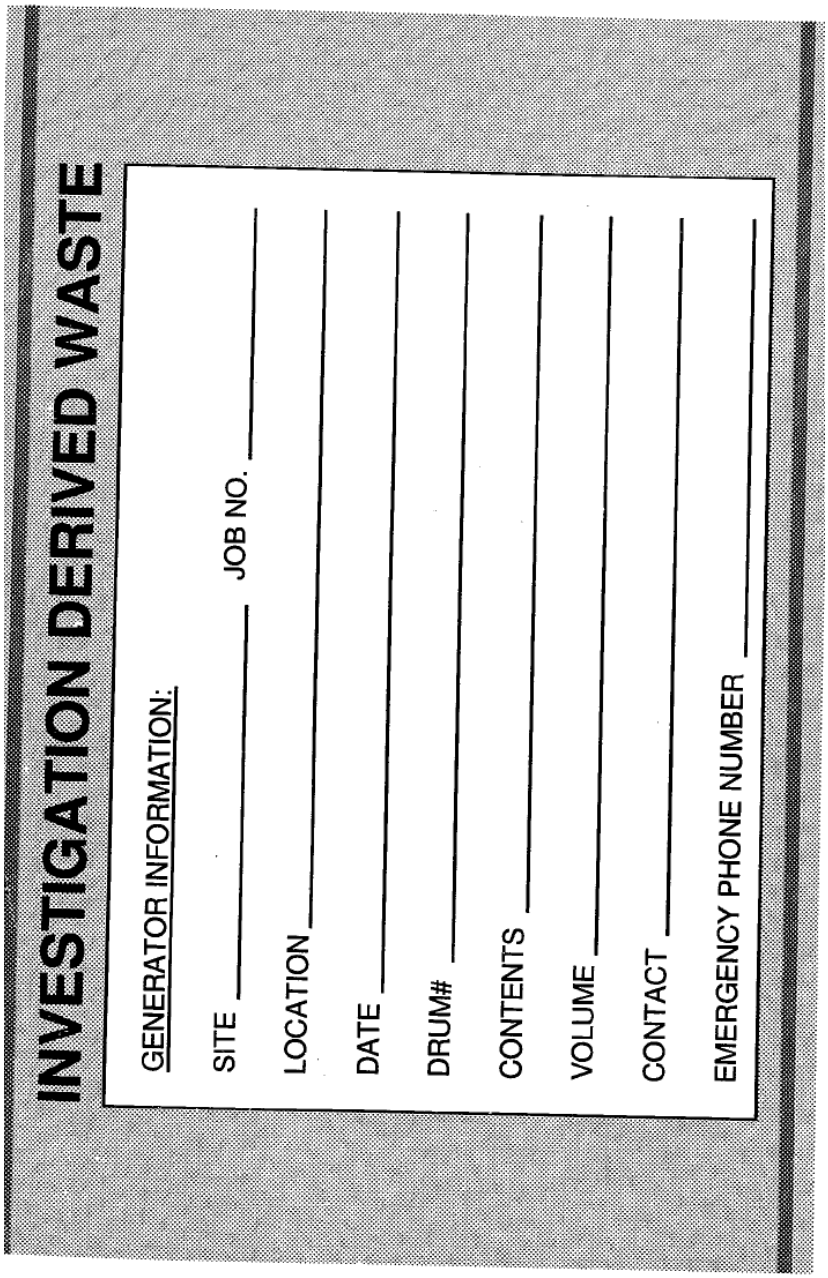
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult 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.

Attachment A
iDW Label



APPENDIX B

LABORATORY SOPS

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Inductively Coupled Plasma Atomic Emission Spectroscopy
Perkin Elmer Optima-3000/3300DV/5300DV

SW-846 Method 6010C

APPROVALS:

Area Supervisor: _____ Date: 1-19-09
Denise S. Coffey

QA Officer: _____ Date: 1-16-09
Tom C. Boocher

Operations Manager: _____ Date: 1/19/09
Jeff P. Glaser

Procedure Number: GR-01-100
Revision Number: 5.7

Date Initiated: 4/4/96
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Date Revised: 1/16/09
Pages Revised: All

By: Denise S. Coffey

Total Number of Pages: 47

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Inductively coupled plasma atomic emission spectroscopy (ICP) determines a wide range of elements in solution.

1.2 The procedure is applicable to a variety of matrices, including but not limited to soil, water, TCLP leachates, EPTOX leachates, SPLP leachates, ASTM leachates, oil, solvents, paint, sludge, pure product or any other matrix that may be extracted or dissolved into an acidic aqueous solution.

1.3 Most matrices require solubilization and/or digestion prior to ICP analysis.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007

2.2 *ICP Winlab Software Guide*, Revision E, June 1997

2.3 *Optima 3000 Family Hardware Guide*, Release B, August 1997

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. Refer to the appropriate SW-846 3000 series methods for this procedure.

3.2 This procedure describes the simultaneous, multi-elemental determination of elements by ICP. The procedure measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the argon plasma torch via an argon carrier gas. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by an eschelle grating polychrometer, and the intensities are monitored by a Segmented Charge Coupled Device (SCD).

3.3 Tables 1 and 1A specify applicable elements, primary wavelengths, and reporting limits. The wavelengths listed in these tables have been selected because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity, and are treated with the same corrective techniques for spectral interference. Other elements may be added as more information becomes available, and as required.

3.4 Quantitation limits are sample dependent and may vary as the sample matrix varies; the quantitation limits provided in Tables 1 and 1A are the current nominal limits for relatively interference-free aqueous and soil matrices. These limits may need to be elevated for more complex sample matrices.

3.5 Scandium (Sc) and Yttrium (Y) are used as internal standards and are not of interest at the quantitation limit. Background correction is required for trace element determination. Background intensities must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity must be free from spectral interferences, and reflect the same change in background intensity that

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.

3.5.1 Refer to Attachment 23.1, Table 1, Analytical Wavelenths and Instrumental Reporting Limits for the Optima 3000.

3.5.2 Refer to Attachment 23.2, Table 1A, Analytical Wavelengths and Viewing for Optima 3300DV.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure may be used for the analysis of the following metals:

Aluminum	Molybdenum
Antimony	Nickel
Arsenic	Phosphorus
Barium	Potassium
Beryllium	Selenium
Boron	Silicon
Cadmium	Silver
Calcium	Sodium
Cerium**	Strontium
Chromium	Sulfur *
Cobalt	Thallium
Copper	Tin
Iron	Titanium
Lead	Vanadium
Lithium	Zinc
Magnesium	
Manganese	

* Optima 3300DV and 5300DV only
**For interference correction only.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.2 TriMatrix SOP GR-01-137, *Acid Digestion of Sediments, Sludges and Soils Using Block Digestion,* latest revision

5.3 TriMatrix SOP GR-01-147, *Acid Digestion of Wastewater, Dissolved Water, Total Water and Extracts, using Block Digestion for the ICP Analysis of Total Metals,* latest revision

5.4 TriMatrix SOP GR-01-119, *Toxicity Characteristic Leaching Procedure (TCLP),* latest revision

5.5 TriMatrix SOP GR-01-117, *Synthetic Precipitation Leaching Procedure (SPLP),* latest revision

5.6 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Spectral Interferences

6.1.1 Spectral interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements.

6.1.2 Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.1.3 Users of simultaneous multi-element instruments, such as the Optima 3000, 3300DV and 5300DV must verify the absence of spectral interference at the analytical wavelength. As an example, Table 2 (Refer to Attachment 23.3, Table 2, Analyte Emission Equivalents Arising from Interference for the Optima 3000, Instrument 101) shows the interferences that have been documented for the Optima 3000. The data in Table 2 is intended as rudimentary guides for indicating potential interferences. This data will periodically change; therefore, these values are only guides. Interelement correction factors are re-evaluated every six months, or following major instrument maintenance, whichever is more frequent.

6.1.3.1 The interference is expressed as apparent false concentration per ppm of interferent. The instrument software automatically calculates and compensates for interelement corrections (IEC). For more information on the topic of interelement corrections, refer to the Optima 3000, Optima 3300DV or Optima 5300DV software manuals.

6.1.3.2 The dashes in Table 2 indicate that no measurable interferences were observed at the concentrations used to create this table.

6.1.4 Interelement interference check solution elements are selected based on the target analyte or analytes interfered with.

6.1.4.1 For example, iron interferes with beryllium and is included in solution IFB-1. Titanium interferes with thallium and is included in solution IFB-2.

6.1.4.2 Multiple elements that interfere with a target analyte are in the solution where the highest degree of interference can be evaluated. For example, copper, iron, manganese, nickel and vanadium interfere with antimony. Iron is in IFB-1 and the rest are in IFB-2 so antimony is in IFB-2.

6.1.4.3 To further validate interelement correction factors, all elements are monitored in each IFA solution if a false positive or negative greater then twice the analyte

reporting limit is observed. If any are observed, locate and correct the problem before reporting analyte results.

6.2 Physical Interferences

6.2.1 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 due to a decrease in nebulization and transport efficiencies. Both the Optima 3000 and 3300DV compensate for the viscosity and solids content by using on-line internal standard addition. The internal standards, in this case Sc and Y, are added via a peristaltic pump to the sample stream as it is pumped into the plasma. If the internal standard intensity changes in a sample relative to a standard, the data for that sample is electronically altered to reflect the assumed change in analyte intensity.

6.2.2 Samples containing large amounts of elements may decrease the forward power available to the plasma for elemental excitation. The RF generator used by Perkin Elmer in both the Optima 3000 and 3300DV is a free running generator, meaning that the generator may compensate for a decrease in the forward power by increasing the amount of power to the RF coil. The result of this compensation is that the forward power available to the plasma will remain at the set level if large quantities of analytes are introduced into the system.

6.2.3 The Optima 3000, 3300DV and 5300DV use a mass flow control for the argon supply which has been reported to improve instrument performance.

6.3 Chemical Interferences

6.3.1 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique.

6.3.2 If observed, chemical interferences can be minimized as follows:

6.3.2.1 By careful selection of operating conditions such as incident power and/or observation position

6.3.2.2 By buffering the sample

6.3.2.3 By matrix matching

6.3.2.4 By using the method of standard additions

6.3.3 Chemical interferences are highly dependent on matrix type and the specific element.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratories, Inc. Laboratory Safety Manual.

7.2 Concentrated acids are used in the preparation of standards and samples for analysis by ICP. Gloves, labcoats, and safety glasses must be worn at all times when handling concentrated acids. Gloves must also be worn when handling samples. Refer to the Material Safety Data Sheets (MSDS) for information on these or any other chemicals utilized in this procedure.

7.3 Check the exhaust hood over the instrument to be sure it is operating correctly. If the ventilation system is not working properly, extinguish the plasma and immediately contact the metals lab supervisor. Under no circumstances should the ICP be used if the exhaust hood is not working.

7.4 The plasma emits UV radiation. Avoid looking directly at the plasma without some type of strong UV protection. The instrument uses a UV resistant material in the viewing port so the analyst may watch the plasma. Do not tamper with this plate. Do not operate the instrument without this plate in place. Do not attempt to view the plasma directly or indirectly in any way. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 The ICP emits a strong RF field. To minimize exposure to this field, Perkin Elmer has included several safety interlocks to prevent exposure of the analyst to harmful radiation. Never override any interlock on the ICP. When working on the instrument, always replace all of the RF shielding using all of the supplied screws. If any safety device has been tampered with, contact the metals lab supervisor.

7.6 The ICP uses argon to sustain the plasma and to nebulize the sample into the plasma. Although argon in and of itself is not hazardous or flammable, it may cause suffocation through oxygen deprivation. It is therefore imperative that all sources of argon be turned off at the valve when not in use. Since argon is colorless and odorless, if you feel lightheaded, evacuate the metals lab at once and notify the metals lab supervisor.

7.7 Many of the chemicals and elements used in this procedure are toxic if ingested. Refer to the MSDS for information on these or any other chemicals utilized in this procedure.

7.8 No food or drink is allowed in the metals lab. Food or drink may become contaminated with acid or metals and may therefore be hazardous.

7.9 Wash hands before starting work. Chemicals may be present on the skin which may interfere with metals analysis. Wash hands before leaving the metals lab. Chemicals and acids may be on the skin which could eventually be ingested or passed on to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Aqueous samples must have been acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, the sample pH must be lowered to <2 during the sample receipt process. A minimum of 24 hours must elapse after adjustment prior to sample digestion.

8.2 Solid samples require no preservation for metals. However, if the same sample container is scheduled for other analyses where refrigeration of the sample is needed, store the sample at 4 ±2° C.

8.3 Samples may be collected in glass or plastic. If Silicon or Boron is to be analyzed, plastic containers must be used. The acidified sample must never come into contact with any metal as this would cause leaching of the metal into the sample.

8.4 For the analysis of dissolved metals, the sample must have been filtered on site and then acidified to pH <2. If the pH was not <2, the sample pH must be lowered to <2 during the sample receipt process and a minimum of 24 hours must elapse after adjustment prior to sample analysis.

8.5 Hold times for all metals for ICP analysis is 180 days. In the case of TCLP or similar leachates, the hold time starts once leachate generation has been completed.

8.6 The minimum sample size for this procedure is 50 mL and 150 mL if samples require Matrix QC. Soils, sediments, and solid waste samples require a minimum of 50 g of sample. This implies that the sample must be solubilized prior to analysis. A smaller minimum amount may be used if the sample may be diluted at the sacrifice of the quantitation limit.

8.7 Digested (solubilized) samples and undigested acidified aqueous samples need not be refrigerated. All samples and digestates must be at room temperature before analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Inductively coupled argon plasma emission spectrometer:

9.1.1 Optima 3000/3300DV/5300DV Inductively Coupled Plasma Optical Emission Spectrometer with an Eschelle based polychrometer and a Segmented-Array Charge-Coupled-Device Detector (SCD). The instrument is capable of taking readings in the UV/VIS spectrum with a wavelength range of 167 to 782 nm. The RF generator employed runs at 40 MHz.

Operating conditions: The analyst must follow the instructions provided by the instrument's manufacturer. For operation with organic solvents, the use of the auxiliary argon inlet is recommended, as is solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental reporting limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within instrument linear range. 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.

9.1.2 Perkin Elmer IEEE autosampler, model AS-90/AS-91. This autosampler must be installed by a Perkin Elmer representative.

9.2 Recirculator/chiller, Polyscience 633 or equivalent. Temperature must be maintained at 18° C and pressure must be at 50 psi. The recirculator is attached directly to the instrument to maintain isothermal conditions within the optics cavity.

9.3 Computer running Perkin Elmer control software. An IBM compatible 80486SX25 or faster computer with at least 64 MB RAM, a 1G hard disk, and a VGA color monitor is needed. The current software used by the instrument is ICP Winlab software version 1.47 running in a Windows NT Environment. The software version may be updated if it performs at least as well as the older version. Major updates, such as moving to another operating platform will require an update to this procedure.

9.4 Appropriate Printer. The printer must be attached to the computer using an appropriate communication cable. Refer to the Instrument manual for further printer specifications.

9.5 15 mL clear centrifuge tubes or equivalent. The tubes must stand upright within the autosampler rack without tipping or leaning.

9.6 Autopipettors capable of delivering 50-1000 µL.

9.7 Class A volumetric flasks, various volumes.

9.8 Class A reusable pipettes, various volumes.

9.9 500 mL plastic bottles for standards and quality control.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 At the beginning of each shift (every 8 hours) the manifold pump tubing must be replaced. Refer to Section 15.5 for a discussion on changing the pump tubing. Update the instrument maintenance logbook.

10.2 There are 3 filters on the Optima 3000 that must be cleaned every week. The first two filters are located on the back of the instrument on the right-hand side. The third filter is located on the left.

10.2.1 Remove the filters from the holders. The right-hand filters have a cover that 'pops' off. The left-hand filter just slides up and out of its holder.

10.2.2 Wash the filters in soap and water to loosen any dirt and dust from the filters.

10.2.3 Squeeze any excess water from the filters. Dry the filters with a paper towel. Allow these filters to air dry.

10.2.4 If there are extra filters, replace the filters on the instrument, otherwise wait until the newly washed filters are dry before replacing them.

10.2.5 Place a note in the instrument maintenance logbook that the filters were cleaned.

10.3 Inspect the waste container on the floor under the instrument every shift. If the container is full, properly dispose of the liquid.

10.4 Visually inspect the torch before lighting the plasma. Do not touch or move the torch. This inspection must be visual only.

10.4.1 Open the doors to the torch chamber.

10.4.2 Observe the torch. If the torch is broken or cracked, replace it. Refer to Section 10.6 for a discussion on how to assemble and disassemble the torch.

10.4.3 Listen for any obvious leaks in the argon lines going to the torch or nebulizer. If leaks are heard, investigate them and tighten the fittings to stop the leak. If the leak cannot be found or cannot be stopped, contact the metals lab supervisor.

10.5 Once every four to eight weeks, disassemble the entire torch assembly and inspect all parts for wear. Replace all worn or damaged parts. Record all changes in the instrument maintenance logbook.

10.5.1 Inspect the drain line at the base of the spray chamber. There must be no leaks or cracks in the tubing. If a problem exists, replace the O-rings in the drain cap. Place a note in the instrument maintenance logbook that the tubing was replaced.

10.5.2 Inspect the nebulizer for wear.

10.5.14.1 Salt deposits, if present, should be removed with water.

10.5.14.2 If a cross-flow nebulizer is used, check the tips for wear. Replace the tips if excessive wear is noted. Record any actions in the instrument maintenance logbook.

10.5.3 Replace the alumina injector with a newly cleaned one. To clean dirty alumina injectors, place them in a plastic 100 mL graduated cylinder. In a hood, cover the injectors with aqua regia. Allow the injectors to soak for 2 hours. Rinse the alumina injectors with tap water, then ASTM Type II water. Place clean injectors in the appropriate drawer for future use. Record the change in the instrument maintenance logbook.

10.5.4 Check all O-rings on the alumina injector holder. Any cracked or chipped O-rings must be replaced with new ones. The book located next to the spare parts drawers has an exploded view of the torch assembly. Use this diagram to identify the exact part number needed for replacement. Note any replacements in the instrument maintenance logbook.

10.5.5 Check the torch for damage. Brown discoloration at the top of the torch is normal. Since the Optima torch has exit windows cut into the torch for light to pass through, this discoloration need not be rectified. Look at the bottom of the torch. There are frequently chips at this spot on the torch. If chips are observed, replace the torch. Never use a torch that is chipped, cracked, or broken. See the metals lab supervisor for help in replacing the torch as this procedure is beyond the scope of this SOP. Note replacement in the instrument maintenance logbook.

10.5.6 Replace the alumina injector support in the spray chamber.

10.5.7 Re-install the spray chamber and alumina injector holder unit in the quick-change mount.

10.5.8 Replace the alumina injector.

10.5.9 Replace the torch on the assembly.

10.5.10 Replace the nebulizer.

10.5.11 Replace the torch assembly on the mounting bracket.

10.5.12 Close the torch box doors.

10.5.13 Note any changes made to the torch during this procedure in the instrument maintenance logbook.

10.6 Inspect the printing quality of the printer. If the print is hard to read or light in appearance, replace the printer cartridge with the appropriate replacement.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Re-distilled acids may be used if it has been demonstrated that the acid is free from contamination.

11.1.1 Concentrated hydrochloric acid, trace metal grade (Fisher Cat #A50B-212) (or equivalent).

11.1.2 Concentrated nitric acid, trace metal grade (Fisher Cat. #A509-212) (or equivalent).

11.2 ASTM Type II water (ASTM D1193). Deionized water is fed into an all glass distillation unit from Barnstead (Model FI-STREEM 2). The resulting distillate is immediately placed into a plastic container. Impurities are measured by the Inorganics group at TriMatrix. This is the only water acceptable for use in the metals laboratory for dilutions or standard preparation.

11.3 Argon gas supply: Welding grade or better. Currently, this is plumbed from a liquid argon tank located outside of the building. Argon is used as the main plasma gas, the auxiliary plasma gas, and the nebulizer carrier gas.

11.4 Nitrogen gas supply: Currently, this is plumbed from a liquid N2 tank located outside of the building. N_2 is used to purge the optics cavity of Oxygen so that measurements below 190 nm may be made accurately.

11.5 Aqua Regia. Three parts hydrochloric to one part nitric. This is used to clean torches and alumina injectors.

12.0 STANDARDS PREPARATION

12.1 All standards are matrix matched as close to sample matrices as possible. Primary Standard stock solutions are purchased from Inorganic Ventures, Absolute Standards and/or SCP Science. All stock solutions are ICP grade single-element (1000 or 10,000 mg/L) or custom multi-element solutions at varied concentrations. All primary standards expire on the supplier's expiration date.

12.2 Secondary Standard stock solutions are purchased from other certified sources. All secondary standards expire on the supplier's expiration date.

12.3 All ICP working standards and dilutions prepared from primary standards expire 3 months after preparation or on the expiration date of the primary standard, whichever is earlier.

12.4 Current purchased custom multi-element standards include: ICP Stock A, ICP Stock B, CRL 1 Stock and

CRL 2 Stock.

12.5 Prepare the working standards.

Note: If Sulfur is a target analyte, refer to Section 12.9.

12.5.1 Rinse four clean 500 mL class A volumetric flasks with ASTM Type II water a minimum of three times.

12.5.2 Place 100 mL ASTM Type II water into each flask.

12.5.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired. Section 12.8.3.1 through Section 12.8.3.3 shows the acid concentrations used for matrix matching.

12.5.3.1 For 10% HCl/5% HNO_3, place 50 mL HCl into the flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.5.3.2 For 5% HCl/5% HNO_3, place 25 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for all digested aqueous samples.

12.5.3.3 For 0.5% HNO_3, place 2.5 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.5.4 Pipette 5.0 mL of ICP Stock A, 5.0 mL of ICP Stock B, 500 µL of 1000 mg/L Co and 500 µL of 1000 mg/L Ag into one of the flasks. This is standard four for the calibration curve.

12.5.5 Pipette 2.5 mL of ICP Stock A, 2.5 mL of ICP Stock B, 250 µL of 1000 mg/L Co and 250 µL of 1000 mg/L Ag into one of the flasks. This is standard three for the calibration curve.

12.5.6 Pipette 0.5 mL of ICP Stock A, 0.5 mL of ICP Stock B, 50 µL of 1000 mg/L Co and 50 µL of 1000 mg/L Ag into one of the flasks. This is standard two for the calibration curve.

12.5.7 Pipette 0.25 mL of CRL 1 Stock and 0.25 mL of CRL 2 Stock into one of the flasks. This is standard one for the calibration curve.

12.5.8 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.5.9 Update the standard numbers in element.

12.5.10 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.6 Sulfur working standards are prepared separately.

12.6.1 Rinse four clean 100 mL class A volumetric flasks with ASTM Type II water a minimum of three times.

12.6.2 Place 25 mL ASTM Type II water into each flask.

12.6.3 Pipette the correct amounts of acid into the flasks depending on the final acid concentration desired.

12.6.3.1 For 10% HCl/5% HNO_3, place 10 mL HCl into the flask and swirl. When the mixing lines are gone from solution, add 5 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.6.3.2 For 5% HCl/5% HNO_3, place 5 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 5 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.6.3.3 For 0.5% HNO_3, place 0.5 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.6.4 Pipette 500 µL of the 1000 mg/L sulfur standard into one of the flasks. This is standard 4 for the curve.

12.6.5 Pipette 250 µL of the 1000 mg/L sulfur standard into a second flask. This is standard 3 for the curve.

12.6.6 Pipette 50 µL of the 1000 mg/L sulfur standard into the third flask. This is standard 2 for the curve.

12.6.7 Pipette 10 µL of the 1000 mg/L sulfur standard into the fourth flask. This is standard 1 for the curve.

12.6.8 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.6.9 Update the standard numbers in element.

12.6.10 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.6.11 Refer to Attachment 23.6, Table 5, Optima Working Standard Concentrations.

12.7 Prepare the stock interelement check standard solutions for IFA-1, IFA-2, IFB-1, and IFB-2.

12.7.1 The solution Interf-1 is purchased from Inorganic Ventures.

12.7.2 Prepare solution ICS-1 Analytes.

12.7.2.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times.

12.7.2.2 Place 2 mL HNO_3 in the flask.

12.7.2.3 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard and never pipette directly out of the primary standard source bottle.

12.7.2.3.1 10 mL each of 1000 mg/L Ag, Cd, Ni, Pb, Zn

12.7.2.3.2 5 mL each of 1000 mg/L Ba, Be, Co, Cr, Cu, Mn, V

12.7.2.4 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.2.5 Update the standard numbers in element.

12.7.2.6 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.7.3 Prepare solution IFA-2 Interferent.

12.7.3.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times. Place 10 mL HNO_3 in the flask.

12.7.3.2 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard, and never pipet directly out of the primary standard source bottle.

10 mL each of 10,000 mg/L Cr, Cu, Mn, Ni, Ti, V

12.7.3.3 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.3.4 Update the standard numbers in element.

12.7.3.5 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents..

12.7.4 Prepare solution IFA-2 Analyte.

12.7.4.1 Rinse a clean 100 mL class A volumetric flask with ASTM Type II water a minimum of three times. Place 2 mL HNO_3 in the flask.

12.7.4.2 Pipette the following amounts of the standards below into the flask, swirling after each addition. Always use a new pipette for each standard and never pipet directly out of the primary standard source bottle.

12.7.4.2.1 10 mL each of 1,000 mg/L Al, As, B, Mo, Sb, Se, Tl

12.7.4.2.2 5 mL each of 10,000 mg/L Ca, Mg, Na

12.7.4.3 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.7.4.4 Update the standard numbers in element.

12.7.4.5 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.8 Prepare the interelement check standard solution IFA-1.

12.8.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.8.2 Place about 100 mL ASTM Type II water into the flask.

12.8.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.8.3.1 For 10% HCl/5% HNO_3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO_3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.8.3.2 For 5% HCl/5% HNO_3. Place 25 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone from the solution. This is for digested aqueous samples.

12.8.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.8.4 Pipette 10 mL of the solution Interf-1 into the flask and swirl.

12.8.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.8.6 Update the standard numbers in element.

12.8.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.9 Prepare the interelement check standard solution IFA-2.

12.9.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.9.2 Place 100 mL ASTM Type II water into the flask.

12.9.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.9.3.1 For 10% HCl/5% HNO_3. Place 50 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.9.3.2 For 5% HCl/5% HNO_3. Place 25 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone from the solution. This is for digested aqueous samples.

12.9.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.9.4 Pipette 10 mL of the solution IFA2 – Interferent into the flask.

12.9.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.9.6 Update the standard numbers in element.

12.9.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.10 Prepare the interelement check standard solution IFB-1.

12.10.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.10.2 Place 100 mL ASTM Type II water into the flask.

12.10.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.10.3.1 For 10% HCl/15% HNO_3. Place 50 mL HCl into the flask and swirl until the mixing lines are gone from the solution. Add 25 mL HNO_3 to the solution. Add 25 mL HNO_3 to the flask and swirl until the mixing lines are gone. This is for solid samples.

12.10.3.2 For 5% HNO_3/5% HCl. Place 25 mL HCl into the flask and swirl until the mixing lines are gone. This is for solid samples.

12.10.3.3 For 0.5% HNO_3. Place 2.5 mL HNO_3 into the flask and swirl. This is for non-digested aqueous samples.

12.10.4 Pipette 10 mL of the solution Interf-1 into the flask and swirl.

12.10.5 Pipette 5 mL of the solution ICS-1 analyte into the flask and swirl.

12.10.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.10.7 Update the standard numbers in element.

12.10.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.10.9 Refer to Attachment 23.7, Table 6 IFB-1, Working Solution.

12.11 Prepare the Interelement Check solution IFB-2

12.11.1 Rinse a clean 500 mL class A volumetric flask with ASTM Type II water a minimum of three times.

12.11.2 Place 100 mL ASTM Type II water into a flask.

12.11.3 Pipette the correct amount of acid into the flask depending on the final acid concentration desired.

12.11.3.1 For 10% HCl/5% HNO_3, place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO_3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.11.3.2 For 5% HCl/5% HNO_3, place 25 mL HCl into each flask and swirl. When the mixing lines are gone from solution, add 25 mL HNO_3 to each flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.11.3.3 For 0.5% HNO_3, place 25 mL HNO_3 into each flask and swirl until the mixing lines are gone. This is for non-digested aqueous samples.

12.11.4 Pipette 10 mL of the solution IFA-2 interferent into the flask. Swirl the flask.

12.11.5 Pipette 5 mL of the solution IFA-2 analyte into the flask. Swirl the flask.

12.11.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.11.7 Update the standard numbers in element.

12.11.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.11.9 Refer to Attachment 23.8, Table 7, IFB-2 Working Solution.

12.12 Prepare the CRL working standard.

12.12.1 Rinse a clean 500 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.12.2 Place about 100 mL ASTM Type II water into the flask.

12.12.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.12.3.1 For 10% HCl / 5% HNO3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone form the solution, add 25 mL HNO3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.12.3.2 For 5% HCl / 5% HNO3. Place 25 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 25 mL HNO3 to the flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.12.3.3 For 0.5% HNO3. Place 2.5 mL HNO3 into the flask and swirl. This is for non-digested aqueous samples.

12.12.4 Pipette 0.5 mL of the CRL 1 Stock and 0.5 mL of the CRL 2 Stock into the flask.

12.11.6 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.11.7 Update the standard numbers in element.

12.11.8 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.13 Prepare the internal standard solution.

12.13.1 Rinse a clean 1000 mL Class A volumetric flask with ASTM Type II water a minimum of three times.

12.13.2 Place 100 mL ASTM Type II water into the flask.

12. 13.3 Pipette the correct amounts of acid into the flask depending on the final acid concentration desired.

12.13.3.1 For 10% HCl / 5% HNO3. Place 100 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 50 mL HNO3 to the flask, and swirl until the mixing lines are gone. This is for solid samples.

12.13.3.2 For 5% HCl / 5% HNO3. Place 50 mL HCl into the flask and swirl. When the mixing lines are gone from the solution, add 50 mL HNO3 to the flask and swirl until the mixing lines are gone. This is for digested aqueous samples.

12.13.3.3 For 0.5% HNO3. Place 5.0 mL HNO3 into the flask and swirl. This is for non-digested aqueous samples.

12.13.4 Use the volumes listed to prepare concentrations for the instrument being used:

12.13.4.1 Pipette 0.50 mL of 1000 mg/L Sc stock standard and 1.0 mL of 1000 mg/L Y stock standard into the flask for the Optima 3000.

12.13.4.2 Pipette 2.0 mL of 1000 mg/L Sc stock standard and 0.075 mL of 1000 mg/L Y stock standard into the flask for the Optima 3300DV.

12.13.4.3 Pipette 2.0 mL of 1000 mg/L Sc stock standard and 0.34 mL of 1000 mg/L Y stock standard into the flask for the Optima 5300DV.

12.13.5 Dilute each flask to the volumetric mark with ASTM Type II water and mix well. Do not transfer to bottles. Adding the water generates heat and a final volume adjustment must be made after the flasks return to room temperature.

12.13.6 Update the standard numbers in element.

12.13.7 After the final volume adjustment, pour the flask contents into clean bottles and label to describe each bottle's contents.

12.14 Prepare the Secondary Calibration Verification (SCV)

12.14.1 There are two SCV stock solutions currently being purchased from Environmental Express.

12.14.1.1 QC Solution A:

12.14.1.1.1 500 mg/L of Al, As, P, Tl, Se

12.14.1.1.2 1000 mg/L each of Ca, K, Mg, Na

12.14.1.1.3 100 mg/L each of V, Ba, B, Cu, Fe, Li, Zn, Cd, Cr, Co, Pb, Mn, Ni, Sr

12.14.1.1.4 10 mg/L of Be

12.14.1.2 QC Solution B:

12.14.1.2.1 500 mg/L of Sb, Sn, Si

12.14.1.2.2 100 mg/L each of Ag, Mo, Ti

12.17.2 The working SCV is prepared fresh daily.

12.17.2.1 Pipet 50 µL of each SCV stock into 9.9 mL of 10% HCl/5% HNO_3 reagent blank. This is for solid samples.

12.17.2.2 Pipet 50 µL of each SCV stock into 9.9 mL of 5% HCl/5% HNO_3 reagent blank. This is for digested aqueous samples.

12.17.2.3 Pipet 50 µL of each SCV stock into 9.9 mL of 0.5% HNO_3 reagent blank. This is for non-digested aqueous samples.

12.17.3 Refer to Attachment 23.12, Table 11, SCV Concentration when diluted according to instructions.

13.0 SAMPLE PREPARATION

13.1 Soil, sludge, oil and waste samples must be digested prior to analysis. Refer to TriMatrix SOP GR-01-137 for the digestion procedure.

13.2 Leachates, such as from the TCLP or SPLP extraction, must be digested. Refer to TriMatrix SOPs GR-01-119, GR-01-117, and GR-01-147 for extraction and digestion.

13.3 Water and groundwater samples for total metals must be digested. Refer to TriMatrix SOP GR-01-147 for the digestion.

14.0 CALIBRATION PROCEDURES

14.1 The Optima 3000, 3300DV and 5300DV must be calibrated for every analytical run or every 24 hours, whichever is more frequent.

14.1.1 Before analysis can begin, the instrument must be profiled using a 10 mg/L Mn solution.

14.1.1.1 Click on the tools drop down menu, then spectrometer control.

14.1.1.2 Aspirate the 10 mg/L Mn solution.

14.1.1.3 Click on "optimize".

14.1.1.4 The instrument will take several readings at different positions in the plasma path.

14.1.1.5 When readings are complete, the instrument will set the viewing position to the highest emission count.

14.1.1.6 Record the highest emission count and position setting in the maintenance logbook.

14.1.1.7 If emission readings vary by more than 30% from the recorded value due to wear or instrument maintenance, re-profile the emission output.

14.1.2 Calibration standards are prepared according to Section 12.0. The final concentrations of the working standards used to create the curve are listed in Table 5.

Note: Concentration of the lowest calibration standard must be at or below the reporting limit.

14.1.3 The mean response of a minimum of two exposures of each standard to the plasma is used in generating a calibration curve.

14.1.4 In all cases, calibration is performed using a multi-point calibration with a calculated zero intercept (not forced). The correlation coefficient must be greater than or equal to 0.995. If correlation is less than 0.995, recalibrate the instrument or postpone analysis for only out-of-control elements, until an acceptable calibration can be attained. If correlation is still less than 0.995, contact the metals laboratory supervisor for assistance.

14.2 After calibration, run an initial calibration verification

14.2.1 Analyze the secondary calibration verification (SCV). The SCV standard must be prepared from an independent (second-source) material at or near the mid-range of the calibration curve. The control window for the SCV is 90% to 110% recovery of the true value. If the percent recovery of any target analyte does not fall within the control window, reanalyze the SCV one time. If the recovery is still out of control, recalibrate the instrument and start the analysis at Section 14.2 or postpone analysis for only the out-of-control elements until an acceptable calibration can be obtained.

14.2.2 Analyze the CCB. The CCB is a reagent blank having the same acid concentration as the standards. The absolute value of the mean ICB reading must be less than the lowest reporting limit required for the analysis. If the CCB is not within the control limit, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2 or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained.

14.2.3 Analyze the Contract Required Detection Limit (CRL). Analyze a low level standard at 2 times the standard quantitation limit. The control window is 50% to 150% recovery. If the recovery is outside of the control window, a narrative must be attached to the LIMS batch.

14.2.4 Analyze the IFA-1 solution. The IFA-1 is comprised of interfering elements only. Depending upon the analytes of interest, a second IFA-2 solution containing additional elements may also need to be analyzed. The purpose of the standard(s) is to demonstrate that, with the current interelement correction factors in place, there are no measurable spectral interferences. The QC acceptance window is 80% to 120% recovery of the true value for the elements spiked and ±2 times the reporting limit for the elements not spiked in the solution.

14.2.5 Analyze the IFB-1 solution (Interelement Correction Standard-1). The IFB-1 solution is a standard that contains four to six elements (i.e., interferents), at high concentrations with remaining elements present at trace levels. Depending upon the analytes of interest, a second IFB-2 solution containing additional elements may also need to be analyzed. The purpose of the standard(s) is to demonstrate that, with the current interelement correction factors in place, there are no measurable spectral interferences. The QC acceptance window is 80% to 120% recovery of the true value.

14.2.6 Follow analysis of the IFB standards with analysis of a Continuing Calibration Verification (CCV) standards with a concentration at the mid-point of the calibration curve. The acceptance window for this QC is 90% to 110% recovery of the true value and <5% RSD. If the CCV is not within the control limits, reanalyze the CCV one time. If the recovery is still out, recalibrate the instrument for the affected analytes and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained.

14.2.7 Analyze the CCB again. If the CCB is not within the control limits, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCB must be reanalyzed for the affected target analyte(s).

14.2.8 Analyze up to 10 samples. If the percent RSD is >20 for any result, the sample immediately prior is to be checked for a high analyte concentration. If the analyte is high, then check the replicates of the sample in question to verify the concentration is not decreasing. If the concentration is decreasing, reanalyze the sample. If the replicates are not decreasing, report the result.

14.2.9 Analyze the CCV again. If the CCV is not within the control limits, reanalyze the CCV one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCV must be reanalyzed for the affected target analyte(s).

14.2.10 Analyze the CCB again. If the CCB is not within the control limits, reanalyze the CCB one time. If the recovery is still out, recalibrate the instrument and start again at Section 14.2, or postpone analysis for only the out-of-control elements until an acceptable calibration can be attained. All samples with an unacceptable CCB must be reanalyzed for the affected target analyte(s).

14.2.11 Repeat Sections 14.2.8 through 14.2.11 until the end of the analytical run.

14.2.12 End the run with a compliant CCV and CCB analysis.

15.0 ANALYTICAL PROCEDURE

Note: The following section assumes the user is familiar with Optima 3000 and 3300DV operating software. If unfamiliar or unsure, consult the instrument manual for additional instructions.

This procedure provides adequate instruction, but is not intended to replace instrument operating manuals.

15.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices. Digestion procedures are presented in the appropriate Sample Preparation Methods.

15.2 Turn on the instrument.

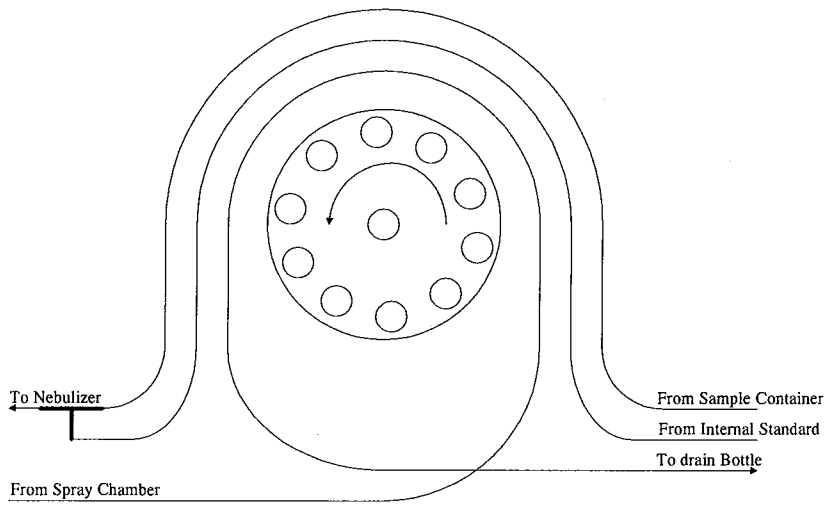
15.3 Boot the computer and ICP WinLab software.

15.4 If the instrument power or RF generator power was not on when the system was booted, an equilibration time will be required by the instrument before the plasma may be lit. The instrument software will alert the operator in this event. Wait until the instrument alerts you that it is ready before proceeding to the next step.

15.5 Change the peristaltic pump tubing. The tubing should be changed every eight hours since the tension exerted on the tubing by the pump will cause the tubing to stretch. Place the waste line on the bottom so that the pump is pumping the waste away from the ICP. Place the internal standard line in the middle so that the pump is pumping the internal standard toward the ICP. Place the sample line on the top so that the pump is pushing the sample to the ICP. Refer to Figure 1 for the correct orientation of the pump tubing. Update the instrument maintenance log to reflect that a change in the pump tubing. Currently, three types of tubing are being used:

Color	Type
Black/Black	Sample line
Orange/Green	Internal standard line
Red/Red	Waste line

Figure 1
Peristaltic Pump Setup



15.6 Recalibrate the spectrometer. The Hg recalibration helps to compensate for drift during the analysis. Although not required, this procedure will greatly increase the stability of the spectrometer, and is therefore highly recommended. Enter the spectrometer mode. Click on the Hg recalibration button. Wait until the computer states that the Hg lamp recalibration is complete. Change the Hg recal time to 2 hours.

15.7 Table 8 lists typical element file conditions for the Optima 3000. Note that any or all of the conditions listed below may change for an element if it has been established that new conditions are at least equivalent to those listed.

15.8 Refer to Attachment 23.9, Table 8, Element Conditions for the Optima 3000. Dashes indicate no Background subtraction point used for this interval.

15.9 Set up the analysis on paper using a sample identification weight sheet (ID/WT sheet). This sheet has columns where the operator enters the sample description, corresponding autosampler position number, digestion dilution (if any), and any subsequent dilutions performed on the sample. This sheet must be filled out before continuing with the procedure, as it is an integral part of the analysis. A typical starting sequence for the ID/WT sheet is given in Table 9. Consult the area supervisor if assistance is required in filling out this sheet.

15.10 Refer to Attachment 23.10, Table 9, ID/WT Sheet Setup for Instrument Calibration and Verification

15.11 Set up the analysis.

15.11.1 Import the analysis sequence from the laboratory information management system Element™ in accordance with TriMatrix GR-10-123.

15.11.2 Enter the analysis sequence.

15.11.3 Analyze samples.

15.11.4 When the analysis is complete, the autosampler will return to position "0" and wait for operator input.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The concentration of each sample is read directly from the computer printout. Dilution factors should be taken into account in the sample information file.

$$C_{Final} = C_{Instr} * DF_{Digest} * DF_{Subseq}$$

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for exporting each run to the laboratory information management system (Element™) in accordance with TriMatrix SOP GR-10-123. It is essential to perform the export correctly to place data from the instrument appropriately and accurately in Element™. Prepare data files for exporting as follows:

17.1.2 Instrument 101 (Optima 3000)

17.1.2.1 Click on "File", "Utilities" and then "Reformat".

17.1.2.2 Browse to select the dataset to be reformatted and exported.

17.1.2.3 Next, save the reformatted file to the archive directory by browsing to select "archive\101\current year\current month\current week". Be sure the reformatted file extension is ".prn".

17.1.2.4 Select the "sample" tab and click each box to show an "x".

17.1.2.5 Select the "mean" tab and click each box to show an "x".

17.1.2.6 Select the "repl" tab and click each box to change the value to the number of replicates used in the analysis.

17.1.2.7 At this point, click "save results" then close the reformatting window.

17.1.3 Instrument 116, and 311 (Optima 3300DV, 5300DV)

17.1.3.1 Click on "File", "Utilities", then "Data Manager".

17.1.3.2 Highlight the dataset to be exported then click "Export".

17.1.3.3 Click "New Design", then "Next" and then "Next" again on the two following windows without making any changes.

17.1.3.4 Change the dataset file name extension to ".prn".

17.1.3.5 Change the directory to archive\me116(311)\current year\export\current month\current week then click "next".

17.1.3.6 Highlight all available parameters and click "Add" then "Next". Repeat for the following page.

17.1.3.7 Finally, click "Export Data" and when complete, close the window.

17.2 If internal chain-of-custody is required, it is very important that the COC form be filled in correctly and returned to the COC file location.

17.3 All hardcopy generated from the analysis must be archived appropriately.

17.4 All logbooks must be filled in completely and correctly. Corrections are to be made with a lineout, not a writeover, and must be dated and initialed. Blank lines on a logbook page must be Z'd out.

18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection.

18.2 Linear range studies must be performed quarterly (or more frequently if specified by project DQO's), or when there is a significant change in instrument response.

18.2.1 To perform a linear range study, calibrate the instrument as it would be calibrated for an actual analysis.

18.2.2 Run standards at high concentrations and calculate the percent recovery for each element. A good place to start is 100 mg/L.

18.2.3 If the percent recovery is acceptable (90%-110%), run a standard at a higher concentration. If the percent recovery is not acceptable, run a standard at a lower concentration.

18.2.4 The last standard concentration that had a percent recovery within the acceptance window will be designated as the linear range for that element.

18.3 IDL studies must be performed after interelement correction factors are updated. Calculate the IDL as follows:

18.3.1 Analyze seven sequential aliquots of metals reagent water. Repeat on two more non-consecutive days to obtain 21 results in 3 datasets.

18.3.2 Input these data to the IDL spreadsheet located on the laboratory intranet to calculate the IDL which is the calculated average standard deviation.

18.3.3 The spreadsheet compares the calculated IDL to the calculated method detection limit (MDL). The IDL must be less than the MDL. If not, locate the source of the problem and repeat the IDL/MDL studies successfully. Repeat only for elements that failed.

18.3.4 Consult the metals supervisor and/or the quality assurance department if repeated studies indicate the IDL is not less than the MDL.

18.4 Interelement correction factors must be updated every 6 months.

18.4.1 Interelement correction factors must be used.

18.4.2 To update or create factors, pure single-element standard solutions of interfering elements are aspirated into the instrument at levels equivalent to those found in samples. The instrument measures the intensity of all of the analytes, and the intensity of the interfering element. The ratio of false analyte intensity to interfering element intensity is called the interelement correction factor. The factors may be positive or negative depending on if the interfering element causes a false positive or false negative reading for the analyte.

18.5 Dilution is not required for samples/digestates exceeding the calibration range until 90% of instrument linearity is exceeded (Section 18.2).

18.5.1 If a project specifically requires quantitation within the calibration range, analyze a single-point standard more concentrated than the sample/digestate. If the single-point result is between 90%

and 110% of the expected concentration based on the existing calibration, sample/digestate results between the highest calibration point and the single-point standard may be quantitated without diluting.

18.5.2 The single-point standard concentration must remain equal to or less than 90% of the established linear range. The high level standard must be analyzed prior to analysis of the closing CCV/CCB.

18.5.3 Any sample/digestate concentration exceeding 90% of the linear range must be diluted.

18.6 Include a minimum of one Method Preparation Blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK must be carried through all sample preparation. Results must be at or below the reporting limit.

18.7 Include a minimum of one Laboratory Fortified Blank (BS) per sample batch. The BS must be carried through the sample preparation procedure. The BS is reagent water spiked with each analyte between the low and mid-level standard except minerals, which are spiked at a higher level. The acceptance window is not to exceed 80 to 120% recovery of the prepared concentration. When historical limits are available, they may be used if within the 80 to 120% maximum. If the BS is outside the acceptance window, re-run once. If still outside the acceptance window, all samples associated with that BS must be re-prepared and re-analyzed.

18.8 Analyze one matrix spike (MS), and one matrix spike duplicate (MSD), at a frequency of at least 1 in 20 (5%). MS and MSDs are aliquots of sample to which is added a known quantity of analyte between the low and mid-level standard except minerals, which are spiked at a higher level. The percent recovery of the analyte is calculated. The percent recovery is not to exceed 75% to 125% recovery (or in-house established limits) with an RPD <20%. This measures the accuracy of the sample preparation method as well as the effect of the matrix on the analysis. The relative percent difference is calculated from the MS and MSD concentrations. The MSD checks the precision of the method.

18.8.1 Pretreated samples must be spiked before the digestion has begun. Refer to the specific digestion procedure for a discussion on spiking.

18.8.2 Samples not requiring pretreatment are spiked before they are physically loaded on the tray.

18.8.2.1 Currently, there are two 'spiking solutions' used to perform the addition of analyte to the sample. These are designated as SSW-1A and SSW-SSS-2A. These solutions are stable by themselves, but due to the high concentrations involved would not be compatible together.

18.8.2.2 Refer to Attachment 23.11, Table 10, Spiking Solutions Final Concentrations

18.8.3 Calculate the percent recovery of the MS and MSD as follows:

$$\%R = \frac{C_{Spike} - C_{Orig}}{\text{Spike Quantity}} \times 100$$

where:
%R is the percent recovery of the spike
C_{Spike} is the concentration of the spiked sample

C_{Orig} is the original concentration of the sample (the unspiked sample concentration)
Spike Quantity is the quantity of the element spiked into the sample

18.8.4 Calculate the relative percent difference between the MS and MSD as follows,

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

where:

%RPD is the relative percent difference between the MS and MSD
C_{MSD} is the concentration of the MSD as read from the raw data printout
C_{MS} is the concentration of the MSD as read from the raw data printout

18.8.5 The MS must recover within 75-125% (or within laboratory established limits) of the true value. The precision between the two spikes must be ≤20%.

18.9 The following tests outlined below are performed with each batch of samples and will ensure that neither positive nor negative interferences are affecting the measurement of any of the elements or distorting the accuracy of the reported values.

18.9.1 Post-Digestion Spike (PS): The same sample from which the MS/MSD aliquots were prepared should also be spiked with a post digestion spike. An analyte spike between the low and mid range standard is added to a portion of a prepared sample, or its dilution. The acceptance window is 80% to 120% of the true value. If the MS/MSD recovery was acceptable and the PDS was unacceptable then a serial dilution should be performed on this sample. If the MS/MSD recovery was unacceptable, and the PDS recovery was unacceptable then a matrix effect is confirmed. An alternative test method should be considered.

18.9.2 Serial Dilution: A serial dilution should be performed on the same sample as the MS/MSD. If the analyte concentration is ≥50 times the MDL, the analysis of a 1:5 dilution should agree within ±10% of the original determination. If not, then a chemical or physical interference effect should be suspected. The use of standard-addition analysis can usually compensate for this effect.

18.9.3 Duplicate (DUP) Analysis: If specified for the project, a DUP is analyzed in substitution for, or in addition to, the MSD. A DUP is a second aliquot of sample prepared and analyzed in the same way as the sample. The acceptance window for the DUP is ≤10% RPD.

18.10 Method of Standard Additions (MSA)

18.10.1 The MSA technique must be performed on all TCLP extracts that have spike recoveries less than 50% or if the concentration is within 20% of the regulatory limit. Regulatory limits are as follows:

Arsenic	5.0 mg/L
Barium	100 mg/L
Cadmium	1.0 mg/L
Chromium	5.0 mg/L

Copper	100 mg/L
Lead	5.0 mg/L
Selenium	1.0 mg/L
Silver	5.0 mg/L
Zinc	500 mg/L

18.10.2 This technique is used when interferences in the sample enhance or depress the analytical signal, resulting in a biased high or low quantitated value. The MSA technique will not correct additive interferences that cause a baseline shift.

18.10.3 The standard addition technique involves adding known amounts of standard to three aliquots of the sample solution. The sample aliquots are then quantitated as discussed in the SOP. The quantitated values of the sample and the three spikes are used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x axis. The absolute value of this point is the analyte concentration in the sample.

18.10.4 Prepare three aliquots with spikes at 50%, 100%, and 150% of the amount found in the sample.

18.10.5 Quantitate the samples following the SOP. Using the regression program located on the computer in the metals lab, plot the absorbance vs. the concentration to construct a curve using the four results. The extrapolated x-intercept point is the concentration of the analyte. An example of a plot so obtained is shown in Attachment 23.4.

Note: For results of this technique to be valid, the following limitations must be taken into consideration:

18.10.5.1 The curve constructed must be linear, with a correlation coefficient of ≥0.995.

18.10.5.2 The chemical form of the analyte added must respond the same way as the analyte in the sample.

18.10.5.3 The interference effect must be constant over the working range of concern.

18.10.5.4 The signal must be corrected for any additive interference.

18.11 Internal Standardization must be used in all analysis to correct for instrument drift, and physical interferences. The recovery requirement for the internal standard is 80% to 120% of the true value.

18.11.1 If the internal standard recovery is low, the sample must be diluted 1:5 and reanalyzed. If the recovery is still low, a further 1:5 dilution is required. This process is repeated until satisfactory recoveries are achieved.

18.11.2 If the internal standard recovery is high, then the internal standard may naturally be present in the sample, and a different internal standard must be used.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of metals reagent water so concentration is in the lower half of the calibration range.

19.1.1.2 Digest and analyze the four spikes as samples following every step in the procedures.

19.1.1.3 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must be within blank spike control limits. Relative standard deviation must be ≤20%.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.1.1.5 Repeated failure will confirm a general problem with the procedure and/or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure and/or techniques uses then repeat the study successfully.

19.1.1.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed.

19.1.2 A Continuing Demonstration of Capability (CDC) study must be performed annually by one of the following:

19.1.2.1 By repeating the IDC study.

19.1.2.2 By using four consecutively analyzed blank spikes obtained from routine sample analysis if run exclusively by the analyst.

19.1.2.3 By successfully analyzing a performance testing sample during the course of routine sample analysis if run exclusively by the analyst.

19.1.2.4 By using the last four of seven results from an MDL study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.3 Submit copies of all successful demonstrations of capability spreadsheets to the quality assurance department for data and training documentation.

19.2 A Method Detection Limit (MDL) study must be performed on every instrument using this procedure and for each digestion procedure. Refer to TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectrometry", Revision 3, February 2007

22.2 *ICP Winlab Software Guide*, Revision E, June 1997

22.3 *Optima 3000 Family Hardware Guide*, Release B, August 1997

23.0 ATTACHMENTS

23.1 Table 1, Analytical Wavelengths and Instrumental Reporting Limits for Optima 3000.

23.2 Table 1A, Analytical Wavelengths and Viewing for Optima 3300DV.

23.3 Table 2, Analyte Emission Equivalents Arising from Interference for the Optima 3000, Instrument 101.

23.4 Table 3, 100X Concentration OPT A

23.5 Table 4, 100X Concentration OPT B

23.6 Table 5, Optima Working Standard Concentrations

23.7 Table 6, IFB-1 Working Solution

23.8 Table 7, IFB -2 Working Solution

23.9 Table 8, Element Conditions for the Optima 3000

23.10 Table 9, ID/WT Sheet Setup for Instrument Calibration and Verification

23.11 Table 10, Spiking Solutions Final Concentrations

23.12 Table 11, SCV Concentration When Diluted According to Instructions

23.13 Table 12, 100X Concentration CRL 1

23.14 Table 13, 100X Concentration CRL 2

Attachment 23.1
Table 1
Analytical Wavelengths And Nominal Reporting Limits For The Optima 3000

Element	Elemental Symbol	Wavelength (nm)	Reporting Limit (ug/L)	Reporting Limit (mg/Kg)
Aluminum	Al	396.140	100	10
Antimony	Sb	206.831	100	10
Arsenic	As	188.979	100	10
Barium	Ba	455.403	10	1.0
Beryllium	Be	234.861	1	1.0
Boron	B	249.773	100	10
Cadmium	Cd	214.438	10	1.0
Calcium	Ca	315.887	500	50
Chromium	Cr	205.558	10	1.0
Cobalt	Co	230.786	10	1.0
Copper	Cu	324.754	10	1.0
Iron	Fe	238.204	10	1.0
Lead	Pb	220.353	50	5.0
Lithium	Li	670.781	8	1.0
Magnesium	Mg	279.074	500	50
Manganese	Mn	257.610	10	1.0
Molybdenum	Mo	202.031	100	10
Nickel	Ni	231.603	10	1.0
Phosphorus	P	213.618	250	25
Potassium	K	766.514	500	50
Scandium	Sc	357.253	N/A	N/A
Selenium	Se	196.026	100	10
Silicon	Si	251.611	500	50
Silver	Ag	328.065	10	1.0
Sodium	Na	588.995	500	50
Strontium	Sr	407.771	50	5.0
Thallium	Tl	190.800	100	10
Tin	Sn	189.933	1000	100
Titanium	Ti	334.941	100	10
Vanadium	V	292.402	10	1.0
Yttrium	Y	360.071	N/A	N/A
Zinc	Zn	206.200	10	1.0

Attachment 23.2
Table 1A
Analytical Wavelengths And Viewing For Optima 3300DV

Element	Elemental Symbol	Wavelength (nm)	View	Reporting Limit (ug/L)	Reporting Limit (mg/Kg)
Aluminum	Al	396.154	Radial	100	10
Antimony	Sb	217.580	Axial	100	10
Arsenic	As	188.979	Axial	100	10
Barium	Ba	455.401	Radial	10	1.0
Beryllium	Be	313.100	Axial	1	1.0
Boron	B	182.576	Axial	100	10
Cadmium	Cd	214.435	Axial	10	1.0
Calcium	Ca	315.887	Radial	500	50
Chromium	Cr	205.557	Axial	10	1.0
Cobalt	Co	228.613	Axial	10	1.0
Copper	Cu	324.754	Axial	10	1.0
Iron	Fe	238.201	Radial	10	1.0
Lead	Pb	220.351	Axial	50	5.0
Lithium	Li	670.782	Radial	8	1.0
Magnesium	Mg	279.074	Radial	500	50
Manganese	Mn	257.606	Radial	10	1.0
Molybdenum	Mo	202.030	Axial	100	10
Nickel	Ni	231.603	Axial	10	1.0
Phosphorus	P	178.222	Axial	250	25
Potassium	K	766.494	Radial	500	50
Scandium	Sc	357.253	Radial	N/A	N/A
Selenium	Se	196.027	Axial	100	10
Silicon	Si	251.609	Radial	500	50
Silver	Ag	328.067	Axial	10	1.0
Sodium	Na	588.998	Radial	500	50
Strontium	Sr	407.769	Radial	50	5.0
Sulfur	S	181.971	Axial	200	20
Thallium	Tl	190.792	Axial	100	10
Tin	Sn	189.927	Axial	1000	100
Titanium	Ti	334.939	Radial	100	10
Vanadium	V	292.399	Axial	10	1.0
Yttrium	Y	360.071	Axial	N/A	N/A
Zinc	Zn	206.198	Axial	10	1.0

Attachment 23.3
Table 2
Analyte Emission Equivalents Arising From Interference For The Optima 3000
Instrument 101

Analyte	□	Al	B	Be	Ca	Cd	Co	Cu	Fe	K	Li	Mg	Mn
Ag	328.065	0.000050	0.000010	-0.000002	-0.000186	-0.000009	-0.000026	-0.000004	-0.000841	-0.000002	---	-0.000914	0.000056
Al	396.140	N/A	0.000015	---	0.000601	0.000015	0.000002	0.000001	-0.000144	0.000005	-0.000001	0.000034	---
As	193.694	-0.000015	-0.000003	---	-0.000001	0.000026	0.000022	-0.000002	-0.000010	---	---	-0.000014	-0.000001
B	249.773	0.000017	N/A	-0.000013	0.000026	0.002253	-0.000094	---	0.002248	0.000008	0.000001	-0.000027	0.000004
Ba	455.403	0.000103	-0.000139	0.000084	0.000055	0.000177	0.008286	0.000013	-0.000429	0.000177	0.000015	0.000229	0.000014
Be	234.861	0.000260	0.000032	N/A	0.000004	0.000047	0.000153	0.000003	0.001322	0.000158	0.000005	-0.000152	-0.000008
Ca	315.887	0.000046	-0.000416	-0.000041	N/A	-0.000459	0.008221	0.000016	0.000264	-0.000099	-0.000011	0.000794	0.000019
Cd	214.438	0.000114	-0.000001	-0.000038	0.000001	N/A	0.000053	0.000022	0.000109	0.000001	---	0.000145	0.000001
Co	238.890	---	-0.000061	0.000002	-0.000005	-0.000046	N/A	---	0.001418	0.000003	---	0.000006	0.000001
Cu	324.764	0.000025	0.000061	0.000013	-0.000030	0.000041	0.000496	N/A	-0.001169	0.000014	0.000005	0.005583	0.000041
Fe	238.204	0.000037	0.000025	0.000004	0.000008	0.000009	0.000098	0.000032	N/A	0.000007	0.000001	0.000396	0.000024
K	766.514	0.000021	0.000098	0.000001	0.000010	0.000017	-0.000066	0.000012	0.000770	N/A	0.000014	0.000108	0.000040
Li	670.781	0.000018	0.000438	-0.000003	-0.000053	0.000251	0.000042	-0.000009	0.000619	0.000028	N/A	0.001026	-0.000007
Mg	279.074	0.000006	0.000005	-0.000001	-0.000026	-0.000004	-0.000183	---	0.000100	-0.000002	---	N/A	0.000053
Mn	257.610	0.000145	-0.000031	0.000001	0.000016	-0.000045	0.000053	0.000004	-0.000116	0.000011	---	0.001103	N/A
Mo	202.031	-0.000007	0.000002	---	-0.000002	---	-0.000005	0.000005	-0.000005	---	---	0.000005	---
Na	588.995	0.000530	0.002112	0.000129	0.000310	0.002348	0.002388	0.000069	-0.002495	0.000726	0.000111	0.001083	0.000116
P	213.618	0.000008	-0.000006	---	-0.000005	-0.000001	-0.000004	0.000202	-0.000018	-0.000002	---	0.000011	-0.000001
Sb	206.831	-0.000027	0.000005	---	-0.000002	-0.000005	0.000006	-0.000002	-0.000008	---	---	0.000019	-0.000002
Sc	357.253	0.000268	-0.000450	-0.000056	0.000136	-0.000875	-0.001421	-0.000010	-0.000510	-0.000126	-0.000023	0.001730	0.000001
Se	196.026	-0.000001	-0.000010	---	0.000001	0.000002	-0.000049	---	-0.000022	---	---	-0.000006	---
Si	251.611	0.000554	-0.000130	-0.000009	0.000319	-0.000278	-0.000358	0.000028	-0.000091	-0.000055	-0.000006	0.009041	0.000015
Sr	407.771	0.000007	0.000227	0.000002	0.006871	0.000242	-0.001019	0.000002	-0.000223	0.000010	0.000292	-0.000004	0.000002
Ti	334.941	0.000092	0.000081	0.000006	-0.000424	0.000159	0.000752	0.000102	0.000262	0.000028	0.000003	0.001648	0.000007
V	292.402	-0.000012	0.000028	-0.000013	0.000002	0.000028	0.000019	0.000001	0.000401	0.000003	0.000001	0.000052	0.000004
Pb	220.353	0.000002	-0.000003	-0.000006	0.000003	-0.000008	0.000072	0.000005	-0.000011	---	---	-0.000062	-0.000001
Ni	231.603	-0.000011	0.000007	---	---	0.000023	0.001143	-0.000002	-0.000007	---	---	-0.000046	---
Cr	205.558	0.000005	0.000007	-0.000535	0.000001	0.000010	0.000020	-0.000001	0.000010	---	---	-0.000014	-0.000003
Zn	213.858	0.000035	0.000020	-0.000005	0.001138	0.000076	0.000062	0.000494	0.000146	0.000008	0.000002	0.000049	0.000006
Sn	189.930	-0.000005	0.000003	---	0.000029	-0.000005	0.000009	-0.000001	-0.000020	0.000001	---	0.002130	-0.000004
Tl	190.794	0.000066	-0.000131	-0.000001	0.000026	-0.000210	-0.000102	-0.000001	-0.000557	-0.000030	-0.000004	-0.000844	0.000007

Attachment 23.3
Table 2 Continued
Analyte Emission Equivalents Arising From Interference For The Optima 3000
Instrument 101

Analyte	□	Mo	P	Sb	Sc	Se	Ti	V	Pb	Ni	Cr	Ag	Tl
Ag	328.065	0.001737	0.000341	0.000406	0.000030	0.001644	0.000045	0.000229	0.003710	-0.000024	-0.000049	-0.000243	0.000172
Al	396.140	0.050458	0.000335	0.000426	-0.000006	0.000179	0.000017	0.000036	0.000070	-0.000520	0.000060	-0.000001	-0.000035
As	193.694	0.000032	-0.000084	-0.000118	-0.000004	0.000177	---	0.000015	0.000062	0.000026	0.000140	0.000002	---
B	249.773	-0.000677	0.000744	0.000266	-0.000004	0.044662	0.000010	-0.000148	0.016232	0.000263	-0.000138	0.000372	0.047492
Ba	455.403	0.004130	0.019290	0.007999	0.000193	0.001338	-0.000023	0.000194	0.003324	0.000472	0.000780	0.000004	0.000881
Be	234.861	-0.005137	0.005041	0.001900	-0.000008	0.001585	0.000007	-0.000001	0.000537	0.000059	-0.001748	0.000006	0.000281
Ca	315.887	0.010015	-0.005816	-0.004457	0.000089	-0.018792	0.000036	-0.003852	-0.005777	0.000046	-0.001528	-0.000099	-0.003067
Cd	214.438	-0.000123	0.000197	0.000387	-0.000004	0.000335	---	-0.000051	0.000228	0.000817	-0.000039	0.000021	0.000032
Co	238.890	0.000090	0.000323	-0.000024	0.000004	0.000070	---	0.000246	0.001663	-0.000037	-0.000054	-0.000003	0.000043
Cu	324.764	0.022074	0.001118	0.000404	0.000076	0.003799	-0.000125	0.000038	0.000981	0.005341	0.000108	0.000014	0.000200
Fe	238.204	0.000118	0.001063	0.001195	0.000007	0.001845	0.000165	-0.001028	0.000438	0.000094	0.004045	0.000007	0.000276
K	766.514	0.002393	0.011369	0.002821	0.000143	0.006884	0.000014	0.000286	0.000204	0.000179	-0.000065	0.000014	0.000156
Li	670.781	-0.000933	0.002374	0.010223	-0.000068	0.006715	-0.000005	0.000108	0.001937	-0.000191	-0.000305	0.000041	0.001957
Mg	279.074	0.000157	-0.000098	0.000029	-0.000004	-0.000059	0.000028	0.000041	-0.000072	-0.000038	-0.000412	-0.000003	-0.000070
Mn	257.610	-0.000385	-0.000633	-0.000299	0.000002	-0.000978	0.000047	-0.000066	-0.000433	0.001016	0.002430	-0.000003	-0.000190
Mo	202.031	N/A	0.000052	0.000011	0.000001	-0.000038	0.000001	0.000004	0.000025	0.000074	0.001457	---	0.000014
Na	588.995	0.003140	0.053565	0.044226	0.000513	0.101270	0.000033	0.000344	0.027038	0.001390	0.002427	0.000416	0.014623
P	213.618	0.001570	N/A	-0.000099	0.000003	-0.000327	-0.000001	0.000002	-0.000166	0.000112	0.000037	-0.000002	-0.000048
Sb	206.831	0.001094	-0.000108	N/A	-0.000070	0.000026	---	0.000001	-0.000033	0.000093	0.001717	---	0.000061
Sc	357.253	0.003392	-0.015891	-0.009545	N/A	-0.030062	-0.000006	0.000634	0.009755	-0.002416	-0.000594	-0.000137	-0.004557
Se	196.026	0.000015	0.000109	0.000032	-0.000002	N/A	---	---	0.000018	0.000004	0.000014	---	0.000009
Si	251.611	0.017841	-0.007392	-0.004843	0.000492	-0.010804	0.000385	0.000471	-0.004126	0.000438	0.000570	-0.000045	-0.001029
Sr	407.771	0.001288	0.002436	0.096894	0.000113	0.007340	-0.000041	0.000046	0.003409	0.000069	0.002546	0.000037	0.004749
Ti	334.941	0.001154	0.002839	0.002701	0.000386	0.005029	N/A	0.002462	0.001298	0.000036	0.011714	0.000027	0.000660
V	292.402	-0.016818	0.000583	0.000402	0.000101	0.001176	0.000049	N/A	0.000325	0.000067	0.001418	0.000003	0.000099
Pb	220.353	-0.001211	-0.000046	-0.000153	0.000007	0.001118	-0.000008	-0.000010	N/A	0.000046	-0.000004	---	-0.000034
Ni	231.603	-0.000540	0.000047	0.001160	0.000003	-0.000118	0.000003	-0.000009	0.000001	N/A	-0.000047	---	0.002266
Cr	205.558	0.000666	0.000083	0.000052	0.000002	0.000532	0.000014	0.000012	0.000068	0.000925	N/A	0.000002	0.000062
Zn	213.858	-0.000080	0.001070	0.000971	0.000004	0.003067	0.000006	0.000086	0.000838	0.010323	0.000151	0.000017	0.000464
Sn	189.930	-0.000047	0.000181	0.000122	0.000005	0.000463	-0.000010	0.000002	0.000086	-0.000099	-0.000059	0.000002	0.000278
Tl	190.794	0.000124	-0.003174	-0.002041	-0.000012	-0.007422	0.000038	0.000352	-0.002062	-0.000201	0.000172	-0.000029	N/A

Attachment 23.4
Table 3
100x Concentration OPT A

Element	Concentration (mg/L)
Ca	1000
Mg	1000
Na	1000
K	1000
As	500
Se	500
P	500
Al	500
Tl	500
V	100
Ba	100
Be	10
B	100
Cu	100
Fe	100
Li	100
Zn	100
Cd	100
Cr	100
Co	100
Pb	100
Mn	100
Ni	100
Sr	100
Ce	100

Attachment 23.5
Table 4
100x Concentration OPT B

Element	Concentration (mg/L)
Sb	500
Sn	500
Si	500
Mo	100
Ti	100

Attachment 23.6
Table 5
Optima Working Standard Concentrations (mg/L)

Element	Standard 4	Standard 3	Standard 2	Standard 1
Ca	10	5	1.0	0.5
Mg	10	5	1.0	0.5
Na	10	5	1.0	0.5
K	10	5	1.0	0.1
S	5	2.5	0.5	0.1
As	5	2.5	0.5	0.10
Se	5	2.5	0.5	0.1
P	5	2.5		0.25
Al	5	2.5	0.5	0.05
Tl	5	2.5	0.5	0.1
V	1	0.5	0.1	0.01
Ba	1	0.5	0.1	0.01
Be	0.1	0.05	0.01	0.001
B	1	0.5	0.1	
Cu	1	0.5	0.1	0.01
Fe	1	0.5	0.1	0.01
Li	1	0.5	0.1	0.008
Zn	1	0.5	0.1	0.01
Cd	1	0.5	0.1	0.005
Cr	1	0.5	0.1	0.01
Co	1	0.5	0.1	0.01
Pb	1	0.5	0.1	0.05
Mn	1	0.5	0.1	0.01
Ni	1	0.5	0.1	0.01
Sr	1	0.5	0.1	0.05
Ag	1	0.5	0.1	0.005
Sb	5	2.5	0.5	0.1
Sn	5	2.5	0.5	
Si	5	2.5	0.5	0.1
Mo	1	0.5	0.1	
Ti	1	0.5	0.1	

Attachment 23.7
Table 6
IFB-1 Working Solution

Element	Concentration (mg/L)
Al	100
Ba	0.5
Be	0.5
Ca	100
Cd	1.0
Co	0.5
Cr	0.5
Cu	0.5
Fe	40
Mg	100
Mn	0.5
Ni	1.0
Pb	1.0
V	0.5
Zn	1.0

Attachment 23.8
Table 7
IFB-2 Working Solution

Element	Concentration (mg/L)
Al	1.0
As	1.0
B	1.0
Ca	5.0
Cr	20
Cu	20
Mg	5.0
Mn	20
Mo	1.0
Na	5.0
Ni	20
Sb	1.0
Se	1.0
Ti	20
Tl	1
V	20

Attachment 23.9
Table 8
Element Conditions For The Optima 3000

Analyte	λ (nm)	Peak Window (nm)	Lower BGC (nm)	Upper BGC (nm)	Viewing Height (mm)	Internal Standard	Standard Concentration	Units	Subarray Number
Ag	328.065	0.0302	0.020	---	12	Sc	1000	ug/L	147
Al	396.140	0.0361	0.048	0.060	12	Sc	5030	ug/L	236
As	188.979	0.0177	0.012	0.012	12	Sc	5050	ug/L	8
B	249.773	0.0230	---	0.031	12	Sc	1005	ug/L	82
Ba	455.403	0.0843	---	0.169	12	Sc	999	ug/L	179
Be	234.861	0.0072	---	0.036	12	Sc	1001	ug/L	74
Ca	315.887	0.0288	0.019	---	12	Sc	10.060	mg/L	137
Cd	214.438	0.0196	0.046	0.020	12	Sc	996	ug/L	40
Co	230.786	0.0220	---	0.022	12	Sc	1000	ug/L	66
Cr	205.558	0.0189	0.019	0.019	12	Sc	1000	ug/L	20
Cu	324.754	0.0396	0.030	0.030	12	Sc	1004	ug/L	153
Fe	238.204	0.0217	---	0.036	12	Sc	1004	ug/L	93
K	766.514	0.1395	0.116	0.093	12	Sc	9.959	mg/L	125
Li	670.781	0.1225	0.061	0.082	12	Sc	999	ug/L	138
Mg	279.074	0.0256	---	0.017	12	Sc	10.030	mg/L	101
Mn	257.610	0.0235	---	0.023	12	Sc	999	ug/L	91
Mo	202.031	0.0186	0.037	0.012	12	Sc	1004.5	ug/L	3
Na	588.995	0.1093	0.055	0.036	12	Sc	10.000	mg/L	148
Ni	231.603	0.0213	0.028	0.021	12	Sc	1000	ug/L	52
P	213.618	0.0196	0.033	0.013	12	Sc	5014	ug/L	38
Pb	220.353	0.0202	0.027	0.020	12	Sc	992	ug/L	54
Sb	206.831	0.0252	0.025	---	12	Sc	4987	ug/L	28
Sc	357.253	0.0326	---	0.043	12	N/A	100	%	186
Se	196.026	0.0179	0.012	0.012	12	Sc	5004.5	ug/L	15
Si	251.611	0.0230	---	0.023	12	Sc	4990	ug/L	89
Sn	189.933	0.0174	0.012	0.023	12	Sc	4992	ug/L	4
Sr	407.771	0.0749	0.037	0.050	12	Sc	996.5	ug/L	216
Ti	334.941	0.0306	---	0.041	12	Sc	1000	ug/L	169
Tl	190.800	0.0174	0.017	0.023	12	Sc	1000	ug/L	14
V	292.402	0.0270	---	0.027	12	Sc	1006	ug/L	113
Zn	206.200	0.0196	0.026	0.026	12	Sc	999	ug/L	39

Approved By: M 1-16-09 **Approved By:** DAL 1-16-09
QA Officer **Area Supervisor**

Attachment 23.10
Table 9
ID/WT Sheet Setup For Instrument Calibration And Verification

Sequence Number	Autosampler Position	Sample ID	Solution Name
1	1	is_init	Blank
2	1	Cal. Blank	Blank
3	2	Std. 1	Standard 1
4	3	Std. 2	Standard 2
5	4	Std. 3	Standard 3
6	5	Std. 4	Standard 4
7	9	SCV	SCV
8	1	ICB	ICB
9	10	CRL	CRL
10	11	IFA-1	IFA-1
11	12	IFA-2	IFA-2
12	13	IFB-1	IFB-1
13	14	IFB-2	IFB-2
14	4	CCV	Standard 3
15	1	CCB	Blank

Attachment 23.11
Table 10
Spiking Solutions Final Concentrations

Element	Aqueous Samples (ug/L)	Solid Samples (ug/L)
Aluminum	1250	1250
Antimony	1250	1250
Arsenic	1250	1250
Barium	250	250
Beryllium	25	25
Boron	250	250
Cadmium	250	250
Calcium	12500	12500
Chromium	250	250
Cobalt	250	250
Copper	250	250
Iron	250	250
Lead	250	250
Lithium	250	250
Magnesium	12500	12500
Manganese	250	250
Molybdenum	250	250
Nickel	250	250
Phosphorus	1250	1250
Potassium	12500	12500
Selenium	1250	1250
Silicon	1250	1250
Silver	250	250
Sodium	12500	12500
Strontium	1250	1250
Sulfur	2500	1250
Thallium	1250	1250
Tin	1250	1250
Titanium	250	250
Vanadium	250	250
Zinc	250	250

Attachment 23.12
Table 11
SCV Concentration When Diluted According To Instructions

Element	SCV (ug/L)
Aluminum	2500
Antimony	2500
Arsenic	2500
Barium	500
Beryllium	50
Boron	500
Cadmium	500
Calcium	5000
Chromium	500
Cobalt	500
Copper	500
Iron	500
Lead	500
Lithium	500
Magnesium	5000
Manganese	500
Molybdenum	500
Nickel	500
Phosphorus	2500
Potassium	5000
Selenium	2500
Silicon	2500
Silver	500
Sodium	5000
Strontium	500
Thallium	2500
Tin	2500
Titanium	500
Vanadium	500
Zinc	500

Attachment 23.13
Table 12
100x Concentration CRL 1

Element	Concentration (mg/L)
Ca	100
Mg	100
Na	100
K	20
As	20
Se	20
P	50
Al	10
Tl	20
V	2
Ba	2
Be	0.2
B	20
Cu	2
Fe	2
Li	1.6
Zn	2
Cd	1
Cr	2
Co	2
Pb	10
Mn	2
Ni	2
Sr	10
Ag	1

Attachment 23.14
Table 13
100x Concentration CRL 2

Element	Concentration (mg/L)
Sb	20
Sn	200
Si	20
Mo	20
Ti	20

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Toxicity Characteristic Leaching Procedure (TCLP)

SW-846 Method 1311

APPROVALS:

Area Supervisor: Marge A. Scott Date: 09-10-09
Marge A. Scott

QA Officer: Date: 9-10-09
Tom C. Boocher

Operations Manager: Date: 9/14/09
Jeff P. Glaser

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By: Marge A. Scott

Total Number of Pages: 27

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure determines the mobility of specific organic and inorganic analyte in liquid, solid, and multiphasic waste by extraction with an acetic acid solution.

1.2 A TCLP need not be run if analytes are not detected or are detected but calculated to be below the regulatory limit. This determination must be based on a total compound analysis of the sample.

1.3 If analysis of any liquid fraction of a TCLP extract indicates a concentration equal to or greater than the regulatory limit, the waste is hazardous by association and analysis of the remaining fractions is not required. This determination applies to multiphasic extracts.

1.4 If the bottle tumbler concentration of a volatile analyte exceeds the regulatory limit, the waste is hazardous and ZHE extraction is not required. However, bottle extraction cannot demonstrate any volatile analyte concentration is below the regulatory limit. ZHE must be performed for that purpose.

1.5 This procedure is applied to TCLP analytes only, except by client request.

1.6 Oily waste samples that filter are assumed to be 100% liquid (filterable), and a total analysis is performed to determine TCLP results (refer to Attachment 20.6 - US Environmental Protection Agency Memorandum #35 dated 06/12/92, page 10). Oily waste and paint waste samples that do not filter are assumed to be 100% solid (nonfilterable) even if appearing to be liquid, and a TCLP extraction is performed.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Revision 0, July, 1992, Method 1311, "Toxicity Characteristic Leaching Procedure".

3.0 SUMMARY OF PROCEDURE

3.1 For filterable waste containing less than 0.5% dry solids, the filtrate becomes the TCLP extract analyzed. A 0.7 µm glass fiber filter is used for all TCLP filtrations.

3.2 For filterable waste containing greater than or equal to 0.5% solids, an aliquot is filtered and the filtrate stored while the solid phase (after any particle size reduction) is extracted in a rotary tumbler. An extraction fluid volume equal to 20 times the mass of the solid phase is used. One of two extraction fluids are employed, based on the solid phase alkalinity. A special zero headspace extractor (ZHE) is used when extracting volatile TCLP analytes. After the solid phase has tumbled for 18 ± 2 hours, the extraction fluid is separated from the solid phase by filtration.

3.3 If multiple phases will not form when mixed, the initial waste filtrate is added to the extraction fluid filtrate, which becomes the TCLP extract analyzed. If incompatible, the two filtrates are analyzed separately. Results of the two analyses are reported as a volume-weighted average concentration in mg/L.

4.0 PARAMETER OR COMPOUND LIST

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4.1 Refer to Attachment 20.1 for parameters and analytes associated with TCLP regulatory limits.

5.0 REFERENCED STANDARD OPERATING PROCEDURES

5.1 TriMatrix Laboratories SOP GR-15-102, Laboratory Waste Disposal, most current revision

5.2 TriMatrix Laboratories SOP GR-09-104, Waste Dilution for Semi-Volatile BNA Compounds, most current revision

5.3 TriMatrix Laboratories SOP GR-04-103, Semi-volatiles Gas Chromatrography Mass Spectrometry(GC/MS) Base Neutral Acids(BNA), most current revision

5.4 TriMatrix Laboratories SOP GR-03-126, Chlorinated Herbicides by Capillary Column Gas chromatography, most current revision

5.5 TriMatrix Laboratories SOP GR-09-113, Extraction of Chlorinated Herbicides from Water and Wastewater, most current revision

5.6 TriMatrix Laboratories SOP GR-09-101, Extraction Base Neutrals Acids(BNA) from Water, most current revision

5.7 TriMatrix Laboratories SOP GR-09-107, Extraction Organochlorine Pesticides and PCBs from Water, most current revision

5.8 TriMatrix Laboratories SOP GR-03-120, Gas Chromatography(GC) Organochlorine Pesticides, most current revision

5.9 TriMatrix Laboratories SOP GR-01-147, Acid Digestion of Total Metals in Aqueous Samples by Block Digestion for ICP, most current revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Potential interferences that may be encountered during analysis are discussed in individual analytical standard operating procedures.

6.2 This is an empirical procedure and must be consistently performed without modification, to attain precise and accurate results.

7.0 SAFETY PRECAUTIONS

7.1 Refer to the TriMatrix Chemical Hygiene Plan and Laboratory Safety Manual for routine precautions and safety instructions.

7.2 Gloves and approved safety glasses must be worn at all times when handling acids and working in the TCLP laboratory. A plastic apron must also be worn when handling large volumes of concentrated acid.

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Concentrated acetic acid fumes are a lung irritant. A Fume hood must be used when handling. Rinse all pipette tips and beakers before disposal or cleaning.

CAUTION: Be certain TCLP tumbler vessels are well vented before tumbling. Each jar must be checked periodically for internal pressure and leakage. Tumbler vessels must be vented cautiously to prevent injury or explosive sample loss. Venting must be performed in a hood.

7.3 If a jar leaks or breaks while tumbling, notify the laboratory supervisor and clean up the spill.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 For metals and semivolatiles TCLP extractions, at least 100 g sample aliquots are required. For volatiles TCLP extractions, a minimum of 25 g is required. These minimum requirements are based on samples with no filterable liquids. At least 100 g is also needed for a percent solids determination, except for samples with no filterable liquids.

8.2 Larger sample aliquots must be extracted based on lower percent solids. Enough extract must be generated to support all required analyses. For multiphase extracts, enough volume must be generated for each phase.

8.3 For fibrous materials like cellulose, cloth or other relatively low density waste, the sample size collected must be greater to meet the minimum mass required for extraction.

8.4 Sample preservation is not required for TCLP samples, but refrigeration may be used if the low temperature will not initiate irreversible physical changes to sample integrity.

8.5 TCLP samples for volatiles analysis must be collected and stored in PTFE-lined capped jars, and stored at 4° C until extraction. Volatiles samples must only be opened immediately before extracting.

8.6 TCLP extract preservation is addressed in section 13.10.8.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 The TCLP extractor rotates extraction vessels end-over-end at 30 ± 2 rpm. Five extractors and three zeroheadspace extractors are located in a room adjacent to the organic prep laboratory. This room is monitored and has an ambient temperature of 23 ± 2° C.

9.2 Zero-Headspace Extraction Vessels

9.2.1 Zero-headspace extraction (ZHE) vessels are used only when waste is tested for volatile analyte mobility. A ZHE is designed to perform liquid phase/solid phase separation, the introduction of extraction fluid, the TCLP extraction and the extract filtration without opening. The ZHE has an internal volume of 500 mL to 600 mL, and is equipped to accommodate a 90-110 mm, 0.7 µm glass fiber filter.

9.2.2 The piston within a ZHE must move with no higher than 15 psi. If not, the Viton o-rings within the vessel must be replaced (Millipore or equivalent). If replacing o-rings does not decrease the

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pressure needed to move the piston, the ZHE must be repaired by the manufacturer before using or be replaced.

9.2.3 A ZHE must be tested for leaks after every extraction by pressurizing to 50 psi and letting sit unattended for one hour. If pressure is lost check all fittings, and inspect or replace o-rings. Repeat the leak test. If leakage problems cannot be solved, the ZHE must be repaired by the manufacturer before using or replaced.

9.3 Bottle Extraction Vessels

9.3.1 Bottle extraction jars are used for TCLP metals and semi-volatile extractions. Jars with sufficient volume to hold sample solids and extraction fluid must be used. Headspace is not an issue. Jars used must be inert to the waste being extracted however.

9.3.2 Soda lime flint glass jars are used when evaluating metals and semi-volatiles mobility from the same extraction.

9.3.3 High density polyethylene (HDPE) jars are used when extracting only TCLP metals.

9.4 Filtration Equipment

9.4.1 Filtration equipment must be inert to the waste being extracted. If using PTFE-coated stainless steel, the coating must unbroken.

9.4.2 All filtrations MUST be performed in a hood.

9.4.3 When waste is evaluated for volatiles, the ZHE vessel is used for filtration. A ZHE supports and holds a 0.7 µm glass fiber filter. The vessel is designed to withstand up to 50 psi without rupturing the filter.

Note: If a filter ruptures, a gas-tight syringe and syringe filter must be used to remove TCLP filtrate and store in a Tedlar® bag.

9.4.4 A positive pressure filter holder is used when waste is evaluated for metals and semivolatiles. The holder has an internal capacity of 1.5 L, accommodates a 142 mm diameter 0.7 µm glass fiber filter, and is capable of exerting 50 psi or greater. Vacuum filtration is not used.

9.4.5 Assemble and operate pressure filters and ZHE vessels per manufacturer's instructions.

9.5 Borosilicate glass fiber filters not containing binder material, with an effective pore size of 0.7 µm (Millipore or equivalent) are used for all TCLP filtrations. Prefilters must not be used. TCLP filters are fragile and must be handled with care.

9.5.1 When evaluating metals mobility, filters must be acid-washed before using, by washing with 1N nitric acid followed by three consecutive washes with deionized distilled water (a minimum of 1 L per rinse).

9.6 The pH meter used must be accurate to ±0.05 units at 25° C.

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9.7 A Tedlar® bag or 500 mL glass gas-tight syringe must be used to collect any initial liquid phase and final TCLP extract when using ZHE, according to the following scenario:

9.7.1 If a waste contains an aqueous liquid phase and less than 1% filterable nonaqueous liquid, a Tedlar® bag or 500 mL glass gas-tight syringe is used to collect and combine the initial filtrate and TCLP extract.

9.7.2 If a waste contains greater than 1% nonaqueous filterable liquid(such as oil) in the initial liquid phase, only a Tedlar® bag is used to collect and combine the initial solid/liquid separation and the final extraction.

9.7.3 If a waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), a Tedlar® bag is used.

9.8 A ZHE extraction fluid transfer device is used to transfer extraction fluid into the ZHE without changing the nature of the extraction fluid.

9.9 The laboratory balance used is accurate to within ±0.01 g.

9.10 Beaker and/or Erlenmeyer flask, glass, or Pyrex, 500 mL.

9.11 Watchglass, glass. Appropriate diameter to cover breaker or Erlenmeyer flask.

9.12 9.5 mm (0.375 inch) standard sieve

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 TCLP Filtration Devices

10.1.1 Change PTFE and rubber gaskets as needed.

10.1.2 Frequently examine the PTFE coating on interior surfaces of each ZHE. Scored or abraded surfaces inhibit effective cleaning of the device. If scored or abraded, return to the manufacturer for recoating.

10.2 ZHE Devices

10.2.1 Replace Viton O-rings before each extraction.

10.2.2 Replace worn gaskets as needed.

10.2.3 Maintain pressure gauges and replace as needed.

10.3 Rotary Tumblers

10.3.1 Monitor and record revolutions per minute (rpm) for each rotary tumbler. Tumbler velocity must be 30 ±2 rpm. If velocity exceeds 30 ±2 rpm perform maintenance and repair of the motor and/or bearings.

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11.0 CHEMICALS AND REAGENTS

11.1 Trace metal grade acids must be used. All reagents must conform to specifications of the Committee on Analytical Reagents of the American Chemical Society (ACS), where such specifications are available.

11.2 Reagent Water: For nonvolatiles extractions, ASTM Type II water or equivalent must be used. For volatiles extractions, the reagent water must also be volatile-free.

11.3 Hydrochloric acid (HCl): 1.0 N

11.4 Nitric acid (HNO_3): 1.0 N

11.5 Sodium hydroxide (NaOH): 1.0 N

11.6 Glacial acetic acid(CH_3CH_2OOH)

11.7 Extraction Fluids:

11.7.1 Extraction fluid #1: Add 285 mL glacial CH_3CH_2OOH to ~25 L of reagent water, add 3.215 L of 1.0 N NaOH, and dilute to 50 L. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

11.7.2 Extraction fluid #2: Dilute 285 mL glacial CH_3CH_2OOH with reagent water to a volume of 50 liters. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

Note: Extraction fluid pH must be checked initially to verify the fluids have been made correctly, and before each extraction use. Additionally, both extraction fluids must be monitored for impurities by preparing method performance blanks. If contamination is found or pH is not within specification, the fluid must be discarded and prepared again. These checks must be documented by recording appropriately.

12.0 STANDARDS PREPARATION

12.1 TCLP extracts for metals, designated as Laboratory Control Samples (LCS) or Matrix Spikes (MS) must be spiked before preservation according to the following:

ICP Analysis				
Analyte	**Stock (ug/mL)**	**Spike added (mL)**	**Sample Volume (mL)**	**Final Spike Conc. (ug/mL)**
Arsenic	500	1.00	100	5.00
Barium	50	1.00	100	0.50
Cadmium	50	1.00	100	0.50
Chromium	50	1.00	100	0.50
Copper*	50	1.00	100	0.50

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Lead	50	1.00	100	0.50
Selenium	500	1.00	100	5.00
Silver	50	1.00	100	0.50
Zinc*	50	1.00	100	0.50

ICP/MS Analysis				
Analyte	**Stock (ug/mL)**	**Spike added (mL)**	**Sample Volume (mL)**	**Final Spike Conc. (ug/mL)**
Arsenic	5.0	1.00	100	0.050
Barium	5.0	1.00	100	0.050
Cadmium	5.0	1.00	100	0.050
Chromium	5.0	1.00	100	0.050
Copper*	5.0	1.00	100	0.050
Lead	5.0	1.00	100	0.050
Selenium	5.0	1.00	100	0.050
Silver	5.0	1.00	100	0.050
Zinc*	5.0	1.00	100	0.050

CVAA Analysis				
Analyte	**Stock (ug/mL)**	**Spike added (mL)**	**Sample Volume (mL)**	**Final Spike Conc. (ug/mL)**
Mercury	0.100	0.200	4.00	0.005

* Additional State of Michigan regulated metals

12.2 Additional elements may be spiked/analyzed by client request.

13.0 ANALYTICAL PROCEDURE

13.1 Perform preliminary TCLP evaluations on a minimum 100 gram sample aliquot. This aliquot does not undergo TCLP extraction.

13.2 Preliminary evaluations include:

13.2.1 Determination of percent solids.

13.2.2 Determination of percent solids to decide whether a waste is extracted or filtered.

13.2.3 Determination of whether the solid portion of a waste requires particle size reduction.

13.2.4 Determination of which extraction fluid must be used for a nonvolatile TCLP extraction. Use the TCLP Extraction Worksheet (Attachment 20.4) as a step-by-step guide and as a logbook for recording sample evaluation results.

13.3 Percent solids (section 2 on the TCLP Extraction Worksheet) are defined as the sample fraction (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

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13.3.1 If a sample will obviously yield no liquid when subjected to pressure filtration (is 100% solids) proceed to section 13.4.

13.3.2 If a sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the pressure filter holder described in section 9.4.4 and is outlined below.

13.3.2.1 Pre-weigh the filter disc and container receiving the filtrate.

13.3.2.2 Assemble the filter holder and filter by placing the filter on the support screen and securing.

13.3.2.3 Weigh out a sample aliquot (100 g minimum) and record the mass.

13.3.2.4 Let slurries stand to permit the solid phase to settle out before filtering. Wastes that settle slowly may be centrifuged. However, centrifugation must be used only as a filtration aid. If used, decant any liquid into the filter before adding the solid phase. Filter the entire aliquot by making a quantitative transfer into the filter holder but do not rinse.

13.3.2.5 Spread unfilterable solids evenly over the surface of the filter. Filtration must be at room temperature to prevent viscosity changes from affecting results.

Note: If greater than 1% of the original mass obviously adheres to the transfer container, determine the mass of this residue and subtract it from the original aliquot mass recorded. This subtraction determines the actual mass transferred.

13.3.2.6 Gradually apply gentle pressure up to 10 psi, until nitrogen gas moves through the filter. If no gas moves through after being at 10 psi for 2 minutes, and if no additional liquid passes through the filter, slowly increase the pressure in 10 psi increments at 2 minute intervals, to a maximum of 50 psi. When the nitrogen begins to move through the filter, or when liquid flow ceases after 2 minutes at 50 psi, stop the filtration.

Note: Instantaneous application of pressure degrades the glass fiber filter and causes premature plugging. Pressure increases must be gradual. Especially below 10psi.

13.3.2.7 Any sample remaining on the filter is defined as the solid phase which must be TCLP extracted. The liquid filtrate is the initial liquid phase.

Note: Certain oily waste and paint waste, may contain liquid material that will not filter, even after pressure filtration to 50 psi. Such unfilterable liquid is a TCLP solid and must be extracted. No attempt must be made to continue filtering by using more than one filter disc or higher pressure.

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13.3.2.8 Determine liquid phase mass by subtracting the filtrate container mass from the total mass of the filtrate/container. Determine the solid phase mass by subtracting the liquid phase mass from the waste aliquot used. Record the liquid and solid phase masses. Calculate percent solids as follows:

Percent Solids = $(M_s / M_w)*100$

Where:
M_s = solid phase mass in grams
M_w = waste aliquot mass in grams

13.4 Calculation of percent solids must be done on dry solids:

13.4.1 To dry a solid phase, remove the solid phase and filter from the filtration apparatus.

13.4.2 Dry the filter and solid phase at 100 ± 20° C until two successive weighings yield the same value within ± 1%. Record the final dry mass.

Note: Caution must be taken to ensure a solid phase will not flash when heated. The drying oven must be vented to a hood.

13.4.3 Calculate percent dry solids as follows:

$S_d = (M_{w+f} - M_f)*100/M_w$

Where:
M_{w+f} = dry waste+filter mass (g)
M_f = tared filter mass (g)
M_w = total waste aliquot mass (g)
S_d = Dry Solids (%)

13.5 When percent dry solids are less than 0.5%, proceed to section 13.10 if a nonvolatiles extraction is required. Proceed to section 13.11 if a volatiles TCLP is required. If solids are equal to or greater than 0.5%, use a new sample aliquot to determine if particle size reduction is necessary and to determine which extraction fluid is required.

13.6 A filterable oil with apparent solids greater than 0.5% due only to an oily residue on the filter because of premature clogging, is considered 100% liquid and its own leachate. TCLP results will be obtained from the filtered oil only. The oily filter/residue will not be TCLP extracted. Volatiles TCLP results will also be obtained from analysis of the filtered oil only.

13.7 Solid phase particle size reduction:

13.7.1 Particle size reduction is required before TCLP extraction unless a solid has a surface area equal to or greater than 3.1 cm^2/g or is smaller than 1 cm in its narrowest dimension. It must be capable of passing through a 9.5 mm standard sieve.

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13.7.2 If a solid phase will not pass through the sieve, reduce particle size by crushing, cutting, or grinding until it does. Record all steps taken to reduce particle size on the TCLP Extraction Worksheet in sections 2.3, 3.2, and 4.4.

Note: Surface area criteria are meant for filamentous paper, cloth, and similar waste materials. Actual measurement of surface area after particle size reduction is not required, nor is it recommended.

13.8 If a solid phase is greater than or equal to 0.5% and will be extracted for nonvolatiles, determine the appropriate extraction fluid as follows (complete section 3 on the TCLP Extraction Worksheet):

Note: TCLP extraction for volatile constituents uses only extraction fluid #1. If TCLP extraction for nonvolatiles is not required, proceed to section 13.11.

13.8.1 Reduce a small subsample of solid phase to particles of approximately 1 mm in diameter or less. Weigh 5.0 g into a 500 mL beaker or Erlenmeyer flask.

13.8.2 Add 96.5 mL of reagent water, cover with a watchglass and record the temperature. Stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the initial pH. The pH must be taken at room temperature (25° C). If the initial pH is less than 5.0, use Extraction Fluid #1. Proceed to section 13.10.

13.8.3 If pH is greater than 5.0, add 3.5 mL 1N HCl and slurry briefly. Cover with a watchglass and heat at 50° C for 10 minutes.

13.8.4 Let the solution cool to room temperature (25° C) then measure and record the pH. If pH is less than 5.0, use extraction fluid #1. If pH is greater than 5.0, use extraction fluid #2.

13.9 If a sample aliquot used for preliminary evaluation is 100% solid, then it can be used for nonvolatiles TCLP extraction (assuming at least 100 g remain). The aliquot used for percent solids determination is also appropriate for nonvolatiles extraction if enough solid was obtained to support all analyses required. If an adequate amount remains, proceed to section 13.10. If a sample is not a 100% solid waste, and a solids determination was done, another aliquot must be taken for TCLP volatiles extraction.

13.10 <u>Procedure to use when volatiles are not involved</u>

13.10.1 For nonvolatiles TCLP extractions, 100 g of sample is required (complete section 4 through 7 on the TCLP Extraction Worksheet). A larger aliquot may be needed depending on percent solids content, on whether any initial liquid phase is miscible with the solid phase extract and on which analytes are required. Enough solid phase extract volume must be obtained to support all analyses requested. If an extract volume generated by a single extraction is insufficient to support all analyses, another sample aliquot must be extracted and the extracts combined before analysis.

13.10.2 If a waste yields no liquid when subjected to pressure filtration (is 100% TCLP solids), weigh out at least 100 g and proceed to section 13.2.9.

13.10.3 If a waste is liquid or multiphasic, pressure filtration is required as follows:

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13.10.3.1 Pre-weigh the container receiving filtrate.

13.10.3.2 Assemble the filter holder and filter. Place the filter on the support screen and secure. Acid-wash the filter if evaluating metals mobility.

Note: Acid-washed filters may be used for any nonvolatile extraction, even when metals are not of concern.

13.10.3.3 Weigh out at least 100 g of waste sample and record the mass. If a waste contains < 0.5% dry solids, the liquid filtrate is defined as the TCLP extract. Enough sample must be filtered so the volume can support all required analyses. For wastes containing ≥ 0.5% dry solids, use the percent solids value obtained earlier to determine the minimum sample mass over 100 g needed to support all TCLP analyses required.

13.10.3.4 Let sedimentary samples settle out before filtering. Wastes that settle slowly may be centrifuged. However, centrifugation must be used only as a filtration aid. If used, decant any liquid into the filter before adding the solid phase. Filter the entire aliquot by making a quantitative transfer into the filter holder, without rinsing.

13.10.3.5 Spread unfilterable solids evenly over the surface of the filter. Filtration must be at room temperature to prevent viscosity changes from affecting results.

Note: If greater than 1% of the original mass obviously adheres to the transfer container, determine the mass of this residue and subtract it from the original aliquot mass recorded. This determines the actual mass transferred to the filter.

13.10.3.6 Gradually apply gentle pressure up to 10 psi, until air moves through the filter. If no gas moves through after being at 10 psi for 2 minutes, and if no additional liquid passes through the filter, slowly increase the pressure in 10 psi increments at 2 minute intervals, to a maximum of 50 psi. When air begins moving through the filter, or when liquid flow ceases after 2 minutes at 50 psi, stop the filtration.

Note: Instantaneous application of pressure degrades the glass fiber filter and causes premature plugging. Pressure increases must be gradual. Especially below 10psi.

13.10.3.7 Waste remaining on the filter is defined as the solid phase which must be TCLP extracted. Any filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase is analyzed as the TCLP extract if dry solids are less then 0.5% or stored at 4° C until the solid phase is extracted, if solids are equal to or greater than 0.5%.

Note: Certain oily waste and paint waste, may contain liquid material that will not filter, even after the pressure filtration described above. Such unfilterable liquid is defined as a TCLP solid and must be

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extracted. No attempt must be made to continue filtering by using a fresh filter. Use only one filter.

13.10.3.8 If a waste contains < 0.5% dry solids proceed to section 13.10.8. If a waste passes the 9.5 mm sieve as received, quantitatively transfer the solid phase and filter into an extraction bottle, then proceed to section 13.10.4.

13.10.3.9 Prepare solid phases for extraction by crushing, cutting, or grinding to a surface area or particle size that will pass through the 9.5 mm sieve. When surface area or particle size has been reduced, quantitatively transfer into an extraction bottle. Include the filter used to separate the initial liquid from the solid phase.

13.10.4 Determine the amount of extraction fluid to add to the extraction bottle as follows:

$$M_{ext\ fluid} = (20/100)*S_d*M_w$$

Where:

$M_{ext\ fluid}$ = Extraction fluid mass needed (g)
M_w = Total waste aliquot mass (g)
S_d = Dry Solids (%)

13.10.5 Check the extraction fluid pH and record in the appropriate TCLP Extraction Fluid Logbook (Attachments 20.2 and 20.3). Slowly add extraction fluid to the extraction bottle. Close the lid tightly using PTFE tape to ensure a tight seal. Secure in the tumbler, and rotate at 30 ± 2 rpm for 18 ± 2 hours. The extraction room temperature must be maintained at 23 ± 2° C during the extraction.

Note: While tumbling, pressure may build within extractor bottles containing lime or calcium carbonate waste from carbon dioxide. To relieve excess pressure, bottles must be periodically opened after extracting 15 minutes, 30 minutes and 1 hour. Vent the bottles into a hood.

13.10.6 Following extraction, filter the extract fluid through a new 0.7 µm glass fiber filter, as outlined previously. The filter may be changed if necessary, to speed up filtration. Filters must be acid-washed if evaluating metals mobility.

13.10.7 Complete TCLP extracts as follows:

13.10.7.1 If a waste contains no initial liquid phase, the extraction bottle filtrate is the complete TCLP extract.

13.10.7.2 If multiple phases will not result, combine the entire extraction bottle filtrate with the initial liquid phase separated from the waste aliquot used. The combined liquid becomes the complete TCLP extract.

13.10.7.3 If an initial liquid phase from the waste does not appear compatible with extraction bottle filtrate, do not combine. Each must be analyzed separately and final TCLP results obtained mathematically.

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13.10.8 Following collection of a complete extract, the pH must be taken and recorded. Immediately aliquot the extract for each analysis and preserve.

13.10.8.1 Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed when nitric acid is added to a small volume, do not acidify and digest as soon as possible.

13.10.8.2 All aliquots must be stored by refrigeration (4° C) unless prepared for analysis immediately.

13.10.8.3 Extracts must be prepared and analyzed according to appropriate analytical procedures. Aliquots for metals must be acid digested except when digestion causes analyte loss. If an undigested extract analysis result exceeds a regulatory limit, the waste is hazardous and extract digestion is not required. However, data from undigested extracts cannot be used to demonstrate a waste is not hazardous. If individual phases are analyzed separately, determine each phase volume to ± 0.5%, conduct the appropriate analysis, and combine individual phase results using a volume-weighted average:

Final Analyte

$$\text{Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = Volume of the first phase (L)
C_1 = Concentration of analyte in the first phase (mg/L)
V_2 = Volume of the second phase (L)
C_2 = Concentration of analyte in the second phase (mg/L)

13.10.9 Report TCLP analyte concentrations with the regulatory limits listed in Attachment 20.1.

13.11 <u>Procedure to use when volatiles are involved</u>

When TCLP volatiles analysis is required, a ZHE must be used to obtain the TCLP extract. Extracts obtained from a ZHE must not be analyzed for metals, pesticides or base-neutrals. The TCLP/ZHE Worksheet (Attachment 20.5) must be completed when volatiles are extracted.

13.11.1 A ZHE has approximately 500 mL internal capacity. No more than 25 g of solid phase can be extracted, due to adding extraction fluid equal to 20 times the solid's mass. (Only the sample fraction from which no additional liquid is filterable at 50 psi is extracted)

13.11.2 Introduce sample into a ZHE for extraction once and do not open until the solid phase extract has been collected. Repeated filling to obtain more extractable solid from a low solids waste is not permitted.

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13.11.3 Do not expose waste samples, initial liquid phases or extracts to the air for any more time than absolutely necessary. These materials must be handled while cold (4° C), to minimize volatiles loss.

13.11.4 Pre-weigh an evacuated Tedlar® bag and set it aside. Express liquid directly into the bag.

13.11.5 Place a ZHE piston within ZHE body (it may be helpful to moisten the piston O-rings with extraction fluid). Adjust the piston to a height that minimizes the piston's movement once the ZHE is charged with sample. Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body. Secure the glass fiber filter between the support screens and set aside. Set the liquid inlet/outlet flange (top flange) aside.

13.11.6 If a waste is 100% solids, weigh out no more than 25 g and record the mass.

13.11.7 If a waste contains < 0.5% dry solids, the liquid filtrate is the TCLP extract. Weigh out up to 500 g for charging and record the mass.

13.11.8 If a waste contains ≥ 0.5% solids, weigh out a calculated sample charging mass up to 500 g and record. Calculate the charging mass to weigh out as follows:

Sample charging mass (g) = (25*100)/percent solids

13.11.8 If solid phase particle size reduction is required, crush, cut, or grind to a surface area or particle size as previously described. The solid phase and reduction equipment must be cooled to 4° C before performing the reduction. The technique used must not generate heat in and of itself. Air exposure during particle reduction must be avoided to the highest extent possible.

Note: Do not sieve due to the possibility that volatiles will be lost. Use a graduated ruler instead.

13.11.9 Do not let slurries stand for solid phase settling. Do not centrifuge waste before charging the ZHE.

13.11.10 Rapidly and quantitatively transfer the entire sample (all phases) into the ZHE without rinsing. Secure the filter and support screens onto the top flange and secure the top flange to the ZHE body. Tighten all ZHE fittings and place in a vertical position (gas inlet/outlet flange on the bottom).

Note: If greater than 1% of the original sample mass obviously adheres to the transfer container, determine the residue's mass and subtract from the charging mass.

Attach a gas line to the gas inlet/outlet valve (bottom flange), then with the liquid inlet/outlet valve (top flange) open begin applying gentle pressure of 1-15 psi to force all headspace out of the ZHE device. Perform the expulsion in a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If a 4° C sample temperature inhibits filtration, let the sample warm to room temperature in the ZHE before filtering.

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13.11.11 Attach the evacuated pre-weighed Tedlar® bag to the liquid inlet/outlet valve, then open the valve. Begin applying gentle pressure of 1-10 psi to force any liquid phase into the bag. If no additional liquid is expelled in any two minute interval, slowly increase the pressure in 10 psi increments or less to a maximum of 50 psi. Continue at the same pressure until no additional liquid is expelled after two minutes, then proceed to the next higher pressure increment. When liquid flow has ceased once 50 psi is reached and no additional filtrate is expelled after two minutes, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, then disconnect and weigh the Tedlar® bag.

Note: Instantaneous application of pressure degrades glass fiber filters and causes premature plugging. Always increase pressure gradually.

13.11.12 Particulate material remaining in a ZHE after sample filtration is the solid phase. The filtrate is the liquid phase. Place Tedlar® bags containing liquid phase in the walk-in cooler until the solid phase has been tumbled and the two phases can be combined. All Tedlar® bags containing liquid phase or extract must be labeled.

Note: Some wastes, such as oily sludges and paint waste, will obviously contain material that appears to be a liquid. Even after applying maximum pressure, this material may not filter. If such is the case, the material within the ZHE is defined as a TCLP solid and is extracted.

If a waste contains < 0.5% dry solids, the ZHE filtrate is defined as the TCLP extract and is ready for analysis. Otherwise, determine the mass of extraction fluid #1 to add to the ZHE as follows:

Extraction fluid #1 (g) = (20/100)*percent solids*sample charging mass (g)

13.11.13 Add extraction fluid to the ZHE and tumble to extract, as follows (fluid #1 is used in all cases):

13.11.13.1 With the ZHE in the vertical position, attach a line from the fluid reservoir to the liquid inlet/outlet valve. The line used must be pre-flushed with fluid to eliminate air pockets. Release pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve and pump the calculated amount of extraction fluid into the ZHE.

13.11.13.2 After adding, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure all valves are closed. Manually rotate the ZHE end-over-end 2 or 3 times. Reposition in the vertical position with the liquid inlet/outlet valve on top. Pressurize enough to move the piston (about 5-15 psi) then slowly open the liquid inlet/outlet valve to bleed any headspace from introducing extraction fluid (do in a hood). The bleeding must be done quickly and stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE to 5-10 psi and check all fittings to ensure each is closed.

13.11.13.3 Place in a ZHE rotary tumbler and rotate at 30 ± 2 rpm for 18 ± 2 hours. The extraction room temperature must be monitored and maintained at 23 ± 2° C during extraction.

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13.11.14 Following extraction, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas.

13.11.14.1 If no gas release is observed, the extractor has leaked. Check for leaks as specified in section 9.2.3, repeating the extraction with a new sample aliquot after repairs are made.

13.11.14.2 If a ZHE is still pressurized after extraction, the liquid in the extractor is filtered out. If the waste contained a compatible initial liquid filtrate, the solid phase extract is filtered directly into the same Tedlar® bag.

13.11.14.3 A separate Tedlar® bag must be used if combining the solid phase extract and liquid filtrate forms multiple phases, or if one Tedlar® bag will not hold the entire volume of both liquids.

13.11.14.4 Filter extract fluid through the glass fiber filter already in the ZHE by increasing pressure gradually, as discussed previously. All extract fluid must be filtered and collected if a Tedlar® bag is used, if an extract is multiphasic, or if the waste contains an initial liquid phase.

Note: An in-line glass fiber filter may be used if it is suspected the glass fiber filter in the ZHE has ruptured.

13.12.15 If a sample contains no initial liquid phase, the extract filtrate is the TCLP extract. If a sample contains an initial liquid phase, the extract filtrate and the initial liquid phase filtrate combined are the TCLP extract.

13.12.15 Immediately prepare TCLP extracts for analysis and store with minimal headspace at 4° C until analyzed. Analyze extracts according to the appropriate analytical procedure. If individual phases are not miscible, determine the volume of each phase to ± 0.5%, analyze both and combine results mathematically as follows:

$$\frac{(C_1)(V_1)+(C_2)(V_2)}{V_1 + V_2}$$

where:
V_1 = Volume of the first phase (L)
C_2 = Concentration of analyte in the first phase (mg/L)
V_2 = Volume of the second phase (L)
C_2 = Concentration of analyte in the second phase (mg/L)

13.12.16 Report TCLP analyte concentrations with the regulatory limits listed in Attachment 20.1.

14.0 DATA REPORTING AND DELIVERABLES

14.1 There are no data reporting or deliverable requirements applicable to this procedure.

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15.0 QUALITY ASSURANCE

15.1 Extraction fluid Blanks:

15.1.1 A minimum of one extraction blank using fluid #1 must be prepared after 20 sample extractions in the same ZHE extractor. There are fourteen extractors labeled A-N.

15.1.2 A minimum of one extraction blank must be performed for each extraction fluid #1 batch, for all analytes.

15.1.3 An extraction blank is set up for each new lot of tumbler vessels. The extraction blank is extraction fluid tumbled and filtered as if it were a sample. Analyze the tumbled extraction blank to document the absence of contamination from the tumbler vessels. If contamination is found, the lot must be rejected.

15.1.4 Perform a filtration blank each day semi-volatiles/metals samples are tumbled. Perform the filtration blank with extraction fluid #2. Treat the solution as a tumbled leachate and pass through the filter. Analyze the filtered extraction fluid to document the absence of carryover from previous filtrations. If carryover is found, all associated samples with positive results must be re-extracted and re-filtered. If unable to re-extract/re-filter affected samples, the associated sample results must be qualified and narrated.

15.2 A post-extraction matrix spike must be performed for each waste type (including wastewater treatment sludge, soils and waste) unless a result exceeds the regulatory limit, and data is used solely to demonstrate a waste is hazardous. At least one matrix spike must be analyzed for each analytical batch. Use matrix spiking concentrations listed in individual volatiles and semi-volatiles analytical procedures, but for metals refer to section 12.0.

15.2.1 Matrix spikes are added to TCLP extracts before preserving for analysis. Do not spike before the TCLP extraction.

15.2.2 In most cases, extracts must be spiked at regulatory limit concentrations. If analyte concentration is less than half a regulatory limit, spike concentration can be as low as half the analyte concentration but not less than five times the method detection limit. To avoid matrix variability, a matrix spike analysis must be done using the same extract volume as an unspiked analysis.

15.2.3 Matrix spikes monitor procedures used for TCLP analysis, and document matrix interference.

15.2.4 Matrix spike recovery is calculated as follows:

$$\%R = 100*(X_s - X_u)/K$$

where:
X_s = Spiked extract concentration (mg/L)
X_u = Unspiked extract concentration (mg/L)
K = Spike concentration (mg/L)
%R = percent recovery

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15.3 Samples must undergo TCLP extraction within the following hold times:

Parameter	MAXIMUM SAMPLE HOLD TIMES (DAYS) From: Sample Collection: To: TCLP Extraction	From: TCLP extraction To: Preparative Extraction	From preparative extraction To: Analysis
Mercury	28	not applicable	28
Metals, except mercury	180	not applicable	180
Volatiles	14	not applicable	14
Semi-volatiles	14	7	40

If any holding time is exceeded, all results obtained must be considered minimal concentrations. Exceeding a holding time can not establish a waste as nonhazardous when results are below the regulatory limit. However, results obtained out of holding time can still establish a waste characterization as hazardous when concentration is above the regulatory limit.

15.4 TCLP logbook entries must be completed for both volatiles and nonvolatiles/metals during initial set-up/percent solids, extraction fluid and pH determinations. Entries must also be made for all filtration steps such as pH measurements and meter identification number, including times completed.

15.5 If an organics/metals extraction bottle breaks and the leachate does not tumble for 18 ±2 hours, the sample must be set up again. If a rotary tumbler is shut off for longer than a few minutes or a ZHE loses **all** (not some) pressure, the sample must be set up again.

15.6 To generate enough leachate for all nonvolatile organics, metals analysis and QC, three liters of leachate must be prepared. Two liters for base-neutrals, pesticides and herbicides. One liter for metals. For only base-neutrals and metals, two liters of leachate must be prepared where one and a half liters goes for base-neutrals, and a half-liter goes for metals. Leachates for non-volatiles organic analysis must be stored in 4 L jars with PTFE-lined lids.

15.7 Extraction fluid is made in 50 L quantities and given a Batch I.D. number. A TCLP extraction blank must be performed on each batch made.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 There is no quantitative demonstration of capability associated with this procedure.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

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17.3 Conserve use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

18.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 0, July, 1992, Method 1311, "Toxicity Characteristic Leaching Procedure".

20.0 ATTACHMENTS

20.1 TCLP Regulated Analytes List

20.2 TCLP Extraction Fluid Number 1 Logbook

20.3 TCLP Extraction Fluid Number 2 Logbook

20.4 TCLP Extraction Worksheet

20.5 TCLP/ZHE Worksheet

20.6 US Environmental Protection Agency Memorandum 35, Pg 10

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Attachment 20.1
TCLP Regulated Analytes List

ANALYTE	CAS NUMBER	REGULATORY LIMIT (mg/L)
VOLATILE ORGANIC COMPOUNDS		
Benzene	71-43-2	0.5
2-Butanone (Methyl Ethyl Ketone)	78-93-3	200
Carbon Tetrachloride	56-23-5	0.5
Chlorobenzene	108-90-7	100
Chloroform	67-66-3	6.0
1,2-Dichloroethane	107-06-2	0.5
1,1-Dichloroethene	75-35-4	0.7
Tetrachloroethene	127-18-4	0.7
Trichloroethene	78-01-6	0.5
Vinyl Chloride	75-01-4	0.2
SEMI-VOLATILE ORGANIC COMPOUNDS		
1,4-Dichlorobenzene	106-46-7	7.5
2,4-Dinitrotoluene	121-14-2	0.13
Hexachlorobenzene	118-74-1	0.13
Hexachlorobutadiene	87-68-3	0.5
Hexachloroethane	67-72-1	3.0
2-Methylphenol	95-48-7	200
3-Methylphenol	108-39-4	200
4-Methylphenol	106-44-5	200
Nitrobenzene	98-95-3	2.0
Pentachlorophenol	87-86-5	100
Pyradine	110-86-1	5.0
2,4,5-Trichlorophenol	95-95-4	400
2,4,6-Trichlorophenol	88-06-2	2.0
METALS		
Arsenic	7440-38-2	5.0
Barium	7440-39-3	100
Cadmium	7440-43-9	1.0
Chromium	7440-47-3	5.0
Copper *	7440-50-8	100
Lead	7439-92-1	5.0
Mercury	7439-97-6	0.2
Selenium	7782-49-2	1.0
Silver	7440-22-4	5.0
Zinc *	7440-66-6	500

* additional State of Michigan regulated metals

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Attachment 20.2
TCLP Extraction Fluid Number 1 Logbook

TriMatrix Laboratories, Inc.

TCLP Extraction Fluid #1
pH=4.93±0.05

Batch I.D. _____ **Date Made** _____ **Analyst** _____
Amt./Lot# NaOH Used _____ **Amt. Glacial Acetic Acid Used** _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / / / / /
_____ / / / / /
_____ / / / / /

TCLP Extraction Fluid #1
pH=4.93±0.05

Batch I.D. _____ **Date Made** _____ **Analyst** _____
Amt./Lot# NaOH Used _____ **Amt. Glacial Acetic Acid Used** _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / / / / /
_____ / / / / /
_____ / / / / /

TCLP Extraction Fluid #1
pH=4.93±0.05

Batch I.D. _____ **Date Made** _____ **Analyst** _____
Amt./Lot# NaOH Used _____ **Amt. Glacial Acetic Acid Used** _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / / / / /
_____ / / / / /
_____ / / / / /

*If pH is out of the acceptable range, note it and make a new batch of fluid

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Attachment 20.3
TCLP Extraction Fluid Number 2 Logbook

TriMatrix Laboratories, Inc.

TCLP Extraction Fluid #2
pH=2.88±0.05

Batch I.D._____ **Date Made**_____ **Analyst** _____
Amount Glacial Acetic Acid Used _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____

TCLP Extraction Fluid #2
pH=2.88±0.05

Batch I.D._____ **Date Made** _____ **Analyst** _____
Amount Glacial Acetic Acid Used _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____

TCLP Extraction Fluid #2
pH=2.88±0.05

Batch I.D._____ **Date Made** _____ **Analyst** _____
Amount Glacial Acetic Acid Used _____
Amount of Reagent Water Used _____ **Total Liter Amt. of Batch** _____

Initial pH of Batch _____ **pH Meter Used/Date Calibrated** _____

Additional pH Readings*/Date Rechecked/Analyst/pH meter used/Date Calibrated
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____
_____ / _____ / _____ / _____

*If pH is out of the acceptable range, note it and make a new batch of fluid

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Attachment 20.4
TCLP Extraction Worksheet

TriMatrix Laboratories, Inc. **TCLP Extraction Worksheet**

Client				
Sample Number				
1.8 Sample Description (Liquid, solid, multi-phasic)				

% Solids Determination

All samples must undergo % solids determination. Only re-use the solids from this section for the fluid determination. Discard any other solids/filtrates from this section!

2.1 Analyst /Date	/	/	/	/
2.2	g	g	g	g
2.3	g	g	g	g
Particle Size Reduction (Y/N)	Y N	Y N	Y N	Y N
2.4	g	g	g	g
2.5 Actual Sample Weight (100g min.)	g	g	g	g
2.6 Total Volume Filtrate(s)	mL	mL	mL	mL

Label the phases of the same number, A=Aqueous, B=Oil, C=Organic Phase, D= Other Phase. Make sure ALL Lab areas are aware of any Multi-phasic samples!

Phase A: Density/Volume	d= / mL	d= / mL	d= / mL	d= / mL
Phase B: Density/Volume	d= / mL	d= / mL	d= / mL	d= / mL
Phase C: Density/Volume	d= / mL	d= / mL	d= / mL	d= / mL
Phase D: Density/Volume	d= / mL	d= / mL	d= / mL	d= / mL
2.7 Weight Of Wet Solids (Filter paper wt. + wet solids)	g	g	g	g
2.8 % Wet Solids [(wt. wet solids- wt. filter paper)/ act. samp. wt.] x100	%	%	%	%

- If % wet solids are >0.5%, but < 100%, then use equation: (100g/ % wet solids) x 100 to determine the actual amount of sample to set up the TCLP in Section 4.3. Also, do the % dry solids to complete all sample records
- If sample is 100% solid, proceed to Section 4.2. If sample is oily and % wet solids are low, then proceed with the % dry solids.

2.9 Weight of Dry Solids (Wt. of filter paper + dry solids)	g	g	g	g
- Sample must be dried at 100C +/- 20C for two readings within +/- 1%.				
2.10 % Dry Solids [(wt. dry solids- wt filter paper)/Act. samp. wt.] x100	%	%	%	%

-If % dry solids are <0.5%, then the filtrate = the TCLP leachate. Sample is 100% liquid. Filter enough sample for all lab areas (see Section 6.3.4 for actual amounts).

-If the sample is oily, the % dry solids are low but not <0.5%, and if there are no "visible" solids on the filter paper, then the oil filtrate = the TCLP leachate (since there is only oil caught in the filter paper.)

Make sure all lab areas receive adequate aliquots of filtrate: Micro-40 mL vial, Prep Lab-40 mL vial. PLEASE, record in Narrative (Section 7) that sample was oily and filtrate=TCLP leachate.

Fluid Determination

3.1 Analyst / Date	/	/	/	/
3.2 Particle Size Reduction (Y/N). If Yes, explain in Narrative, Section 7.	Y N	Y N	Y N	Y N
3.3 Initial Temp(C) (Before stirring 5g of sample/96.5mL of DI water for 5 minutes)	C	C	C	C
3.4 Initial pH (after 5 minutes of stirring)	pH	pH	pH	pH

If initial pH is <5.0, then use Extraction Fluid 1 and record in 3.7. If initial pH is >5.0, then proceed to 3.5.

3.5 Add 3.5 mL of 1N HCL to the sample and water, heat to 50C for 10 minutes, and cool to room temp.

3.6 Final pH of sample (once cooled to room temperature)	pH	pH	pH	pH

If final pH is <5.0, then use extraction fluid 1.
If final pH is >5.0, then use extraction fluid 2.

3.7 Record extraction fluid type (1 or 2)				
3.8 pH meter used				
3.9 Date calibrated				

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Attachment 20.4 (Cont.)
TCLP Extraction Worksheet

TriMatrix Laboratories, Inc. **TCLP Extraction Worksheet**

Sample Number				
Sample Size For TCLP Set-up				
4.1 For 100% solid sample, weight used (100 g minimum)	g	g	g	g
4.2 If % solids were >0.5% and <100%, then record:				
4.2.1 Adjusted amount of sample to yield 100g (from Section 2.)	g	g	g	g
4.2.2 Actual weight of sample (sample - weight of filter papers)	g	g	g	g
4.2.3 Total Amount of Miscible Filtrate(s) (from Section 2.6)	mL	mL	mL	mL
4.3 For 100% liquid sample, record filtrate volumes for all lab areas in Section 6.3.4	mL	mL	mL	mL
4.4 Particle Size Reduction (Y/N)	Y N	Y N	Y N	Y N
Amount of Extraction Fluid (added by weight) For TCLP Set-up.				
5.1 Extraction Fluid (1, 2 or 3) and Batch I.D.	EF	EF	EF	EF
5.2 pH of Extraction Fluid	pH	pH	pH	pH
5.3 20 x Actual solid weight (from Section 4.1 or 4.2.2)	g	g	g	g
TCLP Extraction Record				
6.1 Analyst @ set-up/ Date (MM-DD-YY)	/	/	/	/
6.1.1 Initial Temperature @ set-up (23+/- 2 C)	C	C	C	C
6.1.2 Tumbler I.D. / RPM (30+/-2 RPM)				
6.2 Tumbling Times				
6.2.1 Time started				
6.2.2 Time completed				
6.3 Analyst @ completion/ Date (MM-DD-YY)	/	/	/	/
6.3.1 Final Temperature @ completion (23+/-2 C)	C	C	C	C
6.3.2 Initial miscible phase added back to filtered leachate(Y/N). If Y, explain in section 7.0	Y N	Y N	Y N	Y N
6.3.3 Final pH of leachate (Not preserved)				
6.3.3.1 pH meter I.D.				
6.3.3.2	/	/	/	/
6.3.4 Final Volume of Leachate Collected (mL)	mL	mL	mL	mL
6.3.4.1 Metals Lab (normal aliquot =1L ; min aliquot =200 mL)	mL	mL	mL	mL
6.3.4.2 Prep Lab (normal aliquot=500 mL to 1L ; min aliquot = 500mL)	mL	mL	mL	mL
6.3.4.3 Inorganic Lab (Check with Supervisor for aliquot amount)	mL	mL	mL	mL
6.3.5 Volume of non-miscible phase collected (see Section 2.6)	mL	mL	mL	mL
6.3.5.1 Metals Lab (normal aliquot= 20 mL)	mL	mL	mL	mL
6.3.5.2 Prep Lab (normal aliquot = 40 mL)	mL	mL	mL	mL
6.3.5.3 Inorganic Lab (Check with Supervisor for aliquot amount)	mL	mL	mL	mL
6.3.6 Metals Only				
6.3.6.1 Analyst/ Date	/	/	/	/
6.3.6.2 Metals spikes used, please list				
6.3.6.3 Preservation (pH < 2), Record actual pH	pH	pH	pH	pH

7.0 **Narrative/ Comments:**

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Attachment 20.5
TCLP/ZHE Worksheet

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TCLP/ZHE WORKSHEET

ZHE Identification*	A	B	C	D	E	F	G	H
Client								
Sample Number								
Date Started								
Time Started								
Analyst (Set Up)								
Room Temp. (C)								
Percent Solids								
Wet Sample Weight (g)								
Residue Weight (g)								
Actual Sample Wt (g)								
Filter Weight (g)								
Filter + Solid Wt. (g) wet								
Grams Filtrate								
% Wet Solids								
Adjusted amount of sample to yield 25 grams (See formula below)								
Extraction Fluid Information								
Extraction Fluid Type								
pH of fluid								
Batch Number								
TCLP/ZHE Set-up								
Solid Wt. (g) (25 grams Max.)								
mL initial filtrate (If miscible and back to final filtrate. If not, call initial filtrate Part A and final filtrate Part B of the same sample #)								
mL of extraction fluid								
Speed of Rotary Device (rpm)								
TCLP/ZHE Completion								
Final pH of Leachate								
Date Completed								
Time Completed (18±2 hours)								
pH Meter Used								
Date Calibrated								
Analyst @ Completion								
Location								
Additional Notes								

*Note: A blank must be leached after every 20th sample has been extracted in each ZHE unit.

Adjusted amount of sample to yield 25grams = (25grams/percent wet solids) x 100

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Attachment 20.6
US Environmental Protection Agency Memorandum 35, Pg 10

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US Environmental Protection Agency Memorandum #35, Page 10

Oily Waste Analysis

One of the most frequently asked questions on the RCRA Service concerns the application of the TCLP, Method 1311, to oily wastes. Many callers request technical guidance on the extraction of oily wastes due to the difficulty in the filtration on these types of waste. In many cases, an oily waste does not filter completely due to premature clogging of the glass fiber filter. This can result in the retention of standing liquid on the glass fiber filter. Material that do not pass through the glass fiber filter at the conclusion of the filtration step is defined by the method as the solid phase of the waste. The solid phase is then subjected to the leaching procedure of the TCLP. For oily wastes, clogging of the glass fiber filter can result in an overestimation of the amount of solid material available for leaching.

To solve this problem, the Agency recommends a conservative approach, one that probably will overestimate the amount of leaching. Rather than performing the TCLP extraction on the unfiltered portion of the oily waste, assume the waste is 100% liquid (e.g., will pass through the glass fiber filter) and perform a totals analysis on the oily waste to determine if the oil exceeds the appropriate regulatory level.

Filterable waste oil generated during the TCLP must be analyzed for a variety of organic and inorganic analytes. The OSW recognizes the difficulty in achieving acceptable performance for the analysis of waste oil using methods currently provided in SW-846. As a result, the Agency will provide several new methods for the preparation and analysis of oil samples to the Organic Methods Workgroup in July. In addition, a microwave assisted digestion procedure should improve the analysis of metals and will be proposed as part of the Second Update of the Third Edition of SW-846. Brief descriptions of these techniques are provided below, for additional information on the organic procedures contact Barry Lesnik at (202) 260-7459. For additional information on microwave digestion contact Ollie Fordham (202) 260-4778.

The use of purge-and-trap (Method 5030) for volatiles in oil generally results in severe contamination of analytical instrumentation. Traps, transfer lines and chromatography columns may become contaminated with oil. This leads to elevated baselines, hydrocarbon background in subsequent analyses, and cross-contamination. Headspace (Method 3810) is currently allowed only as a screening procedure in SW-846. The Agency is evaluating the use of headspace in conjunction with isotope dilution mass spectrometry for the quantitative analysis of volatiles in oil. Headspace reduces interference problems encountered with purge-and-trap. However, headspace quantitation can be questionable because the distribution of analytes is not

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STANDARD OPERATING PROCEDURE

Mercury by semi-Automated Cold Vapor Atomic Absorption

EPA Method 245.1
SW-846 Method 7470A
SW-846 Method 7471A

APPROVALS:

Area Supervisor: Denise S. Coffey Date: 1-19-09

QA Officer: Tom C. Boocher Date: 1-15-09

Operations Manager: Jeff P. Glaser Date: 1/19/09

Procedure Number: GR-01-123
Revision Number: 5.6

Date Initiated: 5/6/93
Effective Date: 2/1/09

Date Revised: 1/15/09
Pages Revised: All

By: David W. Johnson
Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure includes analysis for total mercury (inorganic/organic) in mobility extracts, aqueous wastes, groundwaters, drinking waters, surface waters, saline waters, wastewaters, domestic industrial wastes, soils, wastes, sludges and oils. All samples must undergo a preliminary digestion.

1.2 The typical reporting limit is 0.2 ug/L mercury for aqueous and 0.10 mg/kg for solid sample matrices.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

2.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision I, September 1994, Method 7471A, *"Mercury in Solid and Semi Solid Waste* (Manual Cold-Vapor Technique)"

3.0 SUMMARY OF PROCEDURE

3.1 Before analysis, all analytical and quality control samples must be pretreated according to the references discussed in Section 5.0. These digestions reduce organo-mercury compounds to inorganic mercury. Inorganic mercury is then converted to mercury in the Hg^{2+} state. All Hg^{2+} with an HCl carrier enters a gas liquid separatory where it is mixed with stannous chloride to form elemental mercury vapor (Hg^0) according to the following equation:

$$Hg^{2+} + Sn^{2+} \rightarrow Hg^0 + Sn^{4+}$$

3.2 The mixture then flows into a liquid-gas separator where argon is introduced to carry the mercury vapor through a drying tube. The dried mercury vapor then enters one path of a heated double-path optical cell that has been optimized for fast response time. A mercury source powered by a constant current power supply delivers a stable source of absorbance at 253.7 nm. Absorbance by the mercury vapor is measured using a solid-state detector with a wide dynamic range. The resulting signal is referenced to the simultaneous absorbance of the pure carrier gas flowing through the second optical path under identical conditions.

3.3 Absorbance of standards is plotted against known concentrations to build a calibration curve. Absorbance of unknown samples are read against the calibration curve, and regressed to concentration in ug/L. The mercury vapor is vented up a hood to minimize contamination of other samples.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-140, *Preparation Procedure for Mercury in Water, Wastewater, and Liquid Wastes,* latest revision

5.3 TriMatrix SOP GR-01-139, *Preparation Procedure for Mercury in Soils, Wastes, and Oils,* latest revision

5.4 TriMatrix SOP GR-10-106, Inorganic *and Metals Laboratories Corrective Actions,* latest revision.

5.5 TriMatrix SOP GR-10-123, *Element Data Transfer and Review*, latest revision

5.6 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.8 TriMatrix SOP GR-09-128, Homogenization, Grinding and Drying of Solid Samples, latest revision

5.9 *PSA 10.045 Millenium Merlin/Galahad System User Manual,* Issue Number 1.1, Issue Date July 10, 1999

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil samples often contain mercury at low levels. When soils are mixed and ground, there exists the possibility that liquid samples may become contaminated by dust from the air. Therefore, preparation areas for mercury must be kept free of dust and dirt.

Note: Mixing and grinding samples must be done in a fume hood for safety.

6.2 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory so precautionary measures against contamination can be taken.

6.3 Mercury may be lost if the digestion temperature goes above 95° C. The digestion temperature must be monitored and recorded with a digital thermometer on the digestion benchsheet.

6.4 Reagents used may become contaminated over time. If contamination is suspected, the reagent must be discarded following proper laboratory disposal guidelines and fresh reagent made.

6.5 Potassium permanganate is added to eliminate possible interference from sulfide as sodium sulfide.

6.6 Copper interferes at concentrations above 10 mg/L.

6.7 Samples high in chlorides and volatile organic materials require additional permanganate.

Note: The maximum permanganate added to a sample in the digestion batch must be added to all associated quality control samples.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix laboratory safety manual and chemical hygiene plan.

7.2 Concentrated acids are used. Disposable gloves, safety glasses and a laboratory coat must be worn at all times when handling concentrated acids. Gloves, safety glasses and laboratory coats must also be worn when handling digested samples or performing any work within the metals laboratory. Refer to the MSDS located on the library drive for information on any chemical used.

7.3 Make sure the venting system is hooked up and running. Do not operate the instrument if it is not properly ventilated.

7.4 Mercury lamps emit UV radiation. Avoid looking directly at the lamp without some type of strong UV protection. Failure to follow this policy may cause very serious and immediate damage to the retina of the eye.

7.5 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Mercury compounds can also be absorbed through the skin. Wear disposable gloves at all times. Elemental Hg^0 and Hg^{2+} are toxic if ingested. Always wash hands after handling mercury standards, samples or reagents, and when leaving the laboratory.

7.6 No food or drink is allowed in the metals laboratory. Food and drink may be consumed in the laboratory office but not stored there.

7.7 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed on to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples should be acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, lower to pH <2 during the sample receipt process. A minimum of 24 hours must elapse after pH adjustment by the laboratory before performing the sample digestion.

8.2 Store non-aqueous and solid samples at 4 ±2° C.

8.3 The maximum holding time for all mercury samples is 28 days from the collection date. Analyze non-aqueous samples as soon as possible within that time.

8.4 All samples must be subjected to digestion before analysis.

8.5 The minimum collected aqueous sample volume required is 100 mL. If quality control is also required, collect at least 300 mL. The minimum sample size for solid samples is 10 g.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Laboratory analytical balance capable of weighing 0.0001 g

9.2 Laboratory balance capable of weighing 0.1 g

9.3 Macropipettor capable of delivering 50 - 1000 µL

9.4 Class A volumetric flasks, various volumes

9.5 Class A reusable pipettes, various volumes

9.6 Macropipettes capable of delivering 5 - 10 mL of solution

9.7 Wooden laboratory spatulas

9.8 Calibrated digital thermometer capable of reading to 100° C. Mercury thermometers must never be used in this procedure.

9.9 PSA low-level absorbance mercury analyzer, Millenium system, model 10.045

9.10 Computer running Avalon software. An IBM compatible computer running Windows NT 4.0 service pack 5.0. The software version may be updated without notice if it performs at least as well as the older version.

9.11 Filter, 0.45 µm glass fiber.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Check the calibration on autopipettors daily. Remove from service and send out for recertification when daily checks indicate unacceptable accuracy or performance. Perform an annual inspection to determine overall condition.

10.2 Verify balance calibration daily. Remove from service and call for repair when daily checks indicate unacceptable accuracy or performance. Recertify annually through a contracted service representative.

10.3 Inspect the waste bottle under the instrument before starting. If the bottle is full, properly dispose of the liquid.

10.4 Under normal conditions, the PSA requires very little maintenance. However, attention to the maintenance schedule below must be followed:

10.4.1 Daily:

10.4.1.1 Before shutting down, flush all tubing and the sample valve with laboratory reagent water. Finally, flush with air.

10.4.1.2 It may necessary to replace all tubing. However, flushing of the sample valve must still take place.

10.4.1.3 Replace the pump tubing if required. When in doubt, replace it.

10.4.1.4 Ensure that glassware is clean. When in doubt, clean it.

10.4.1.5 Clean all outer surfaces of the instrument.

10.4.1.6 Also after shutting down, release any tension on the pump tubing.

10.4.2 Weekly:

10.4.2.1 Perform daily maintenance activities.

10.4.2.2 Replace all pump tubing if not already replaced as part of the daily maintenance.

10.4.3 Monthly:

10.4.3.1 Perform daily and weekly maintenance activities.

10.4.3.2 Replace all pump tubing if not already replaced as part of daily/weekly maintenance.

10.4.3.3 Check for discoloration in the Perma-pure drying system and replace as necessary.

10.4.4 Six Months:

10.4.4.1 Perform daily, weekly and monthly maintenance.

10.4.4.2 Replace the inner membrane of the Perma-pure drying system if needed.

10.4.5 Yearly:

10.4.5.1 Perform daily, weekly, monthly and six month maintenance.

10.4.5.2 Change the inner membrane of the Perma-pure drying system if needed.

10.4.5.3 Replace the activated charcoal in the exhaust line.

10.4.6 Cleaning:

10.4.6.1 Before undertaking any cleaning, disconnect the main supply cable from the instrument. Cleaning is ONLY recommended for the outer case.

10.4.6.2 Clean with a damp cloth or sponge which has been previously immersed in warm soapy water. Alternatively, a suitable laboratory cleaning agent may be used. Avoid copious amounts of liquid coming into contact with the instrument.

10.4.6.3 Finish off by wiping down with a dry lint-free cloth or sponge.

10.4.7 Wipe acid spills up with a suitable neutralizing agent IMMEDIATELY. However, be careful to avoid a copious volume of liquid coming into contact with the instrument.

11.0 CHEMICALS AND REAGENTS

11.1 Acids used in standards preparation and for samples must be trace metal grade or better.

11.1.1 Concentrated hydrochloric acid, trace metal grade

11.1.2 Concentrated nitric acid, trace metal grade

11.2 ASTM Type II water

11.2.1 Deionized water is fed into an all-glass distillation unit. The resulting distillate is immediately placed into a plastic container. This is the only water acceptable for mercury analysis.

11.3 Argon gas supply

11.3.1 Welding grade or better. This is plumbed from a liquid argon tank located outside the building. Argon is used as the carrier gas during analysis.

11.4 Stannous chloride, 2% solution in 10% hydrochloric Acid.

11.4.1 Place 20 g stannous chloride into a clean 1 L polyethylene bottle. Add 100 mL concentrated HCl and swirl until the SnCl is dissolved. Add 880 mL ASTM Type II water then cap and mix.

11.4.2 Create a new standard number in Element™ when the stannous chloride lot changes.

12.0 STANDARDS PREPARATION

12.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140, for the preparation of mercury standards.

13.0 SAMPLE PREPARATION

13.1 Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of samples by matrix.

14.0 CALIBRATION PROCEDURES

14.1 A blank and five standards comprise the initial calibration curve. Refer to TriMatrix SOP GR-01-139 and SOP GR-01-140 for the preparation of calibration standards.

14.2 Prepare a digested calibration curve the same day as digestates and use for quantitation.

15.0 ANALYTICAL PROCEDURE

15.1 Analysts must be familiar with the software's operation by reading the software manual and through documented training from an experienced analyst before proceeding.

15.2 After running the calibration curve, analyze a second-source calibration verification (SCV) as a sample. The SCV is prepared from a second-source purchased standard and prepared at 3.0 ug/L. SCV recovery must be within 95 – 105% of the expected value for method reference 245.1 or within 90 – 110% for method references 7470A and 7471A.

15.2.1 If the SCV fails recovery, repeat the analysis.

15.2.2 If the second analysis fails, initiate corrective action:

15.2.2.1 Adjust the gas flow if needed.

15.2.2.2 Replace tubing if needed.

15.2.2.3 Check for other mechanical problems to verify the instrument is operating correctly.

15.2.3.4 Remake and redigest the calibration standards if necessary.

15.2.3.5 Remake the SCV standard if necessary.

15.2.3 After resolving the problem, recalibrate the instrument and run the SCV successfully.

15.2.4 Digestions may not be analyzed without an acceptable SCV.

15.3 Analyze a calibration blank (CCB) after the SCV. The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.

15.4.1 If the CCB is higher than the reporting limit, reanalyze one time.

15.4.2 If the second analysis fails, initiate corrective action.

15.4.2.1 Locate the source of the contamination.

15.4.2.2 Correct the contamination problem.

15.4.2.3 Repeat all associated sample digestions associated with the contamination problem and/or narrate the contamination.

15.5 Analyze a contract-required limit (CRL) standard at the beginning of each run.

15.5.1 A CRL is reanalysis of the digested 0.2 ug/L standard from the initial calibration curve as a sample.

15.5.2 CRL recovery must fall within 50 – 150% of the expected value. If the CRL result is above the calculated MDL value but outside the control limits, narrate in the analytical batch as follows:

15.5.2.1 The CRL recovery for this analyte was outside laboratory control limits. However, the only requirement is that the analyte be detected. No qualifications are necessary.

15.6 After analysis of the CRL, analyze a continuing calibration verification (CCV) standard. The CCV is the midpoint calibration standard analyzed as a sample. CCV recovery must be within 90 – 110% of the expected value.

15.6.1 If the CCV fails, reanalyze one time.

15.6.2 If the CCV still fails, stop the analysis and initiate corrective action.

15.6.2.1 Check to make sure the instrument is operating correctly.

15.6.2.2 Recalibrate.

15.6.2.3 Reanalyze all digestates since the last acceptable CCV.

15.7 Analyze a continuing calibration blank (CCB). The mercury concentration found in the CCB must not be greater than the absolute value of the reporting limit to be acceptable.

15.7.1 If the CCB fails, reanalyze one time.

15.7.2 If the second analysis fails, initiate corrective action:

15.7.2.1 If a high level sample is carrying over, analyze the CCB until the system is clean then reanalyze all digestates run since the last acceptable CCB.

15.7.2.2 If no apparent cause can be found, recalibrate then reanalyze all digestates run since the last acceptable CCB.

15.8 After the initial quality control has passed, analyze up to 10 samples (including the high and low concentration blank spikes) then repeat analysis of the CCV and CCB. Repeat, until all samples are analyzed.

Note: Shake all digestates before analysis. Solid matrix digestates must be filtered through a 0.45 µm filter before analysis.

15.9 Check all raw data to make sure each result is within the calibration range. If any result above the calibration range, mark the sample number and estimate a dilution factor. Make the dilution using a digestion CCB that matches the sample reagent matrix then analyze within the calibration range.

15.9 Verify the calibration after all samples have been analyzed as follows:

15.9.1 Analyze a final CCV successfully.

15.9.2 Analyze a final CCB successfully, which concludes the run.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Sample concentration is read directly from the computer printout in ug/L. Dilution factors must be taken into account in the ID/WT file and the sample information file.

16.1.1 Quantitate water, wastewater and potable water samples as follows:

$$C_{Final} = C_{Instr} \times DF_{Subseq}$$

where:

C_{Final} = The final reported concentration in ug/L
C_{Instr} = The concentration as read from the instrument printout in ug/L
DF_{Subseq} = Any dilution ratio subsequent to the digestion dilution

16.1.2 Quantitate TCLP and SPLP leachates as follows:

$$C_{Final} = \left(\frac{C_{Inst} \text{ X } DF_{subsequent}}{1000}\right)$$

Where:

C_{Final} = The final reported concentration in mg/L
C_{Inst} = The concentration as read from the instrument printout in ug/L
$DF_{subsequent}$ = Any dilution ratio subsequent to the digestion dilution
1000 = Conversion from ug/L to mg/L

16.1.3 Quantitate solid samples as follows:

$$C_{Final} = \left(\frac{C_{Inst} \text{ X } DF_{subsequent}}{1000}\right)$$

Where:

C_{Final} = The final reported concentration in mg/kg
C_{Inst} = The concentration as read from the instrument printout in ug/kg
$DF_{subsequent}$ = Any dilution ratio subsequent to the digestion dilution
1000 = Conversion from ug/kg to mg/kg

16.2 Duplicate measurements are taken and recorded for each sample. The average of these two readings is reported as the final result.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for correctly filling in, handing in and filing all associated paperwork. It is essential to perform these tasks for the laboratory to provide clients with defensible data.

17.2 LIMS Reporting

17.2.1 When finished running a sample set, all data must be uploaded to Element™, the Laboratory Information Management System (LIMS) in accordance with TriMatrix SOP GR-10-123.

17.2.1.1 To export a data set from the instrument for LIMS uploading with DataTool, click on the red arrow.

17.2.1.2 Choose "comma" and "browse" then select the "groups\lab.me\export data\216" folder and click "OK".

17.2.1.3 Add a backslash (\) and type the data set filename (include ".res" on the end). For example, to export dataset y17wta2, the path appears as "groups\lab_me\exportdata\216\y17wta2.res".

17.2.1.4 Click "OK" to export the data.

17.2.2 If an internal chain-of-custody is required, it is very important that COC forms be filled in correctly and returned to the COC file location. Refer to Attachment 23.5 for an example COC form.

17.3 Laboratory Required Paperwork

17.3.1 Data backup logbook and maintenance logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single lineout, which is dated and initialed. Writeovers are NOT acceptable.** Erroneous results must remain legible. A new result is placed near the incorrect result. Blank lines in logbooks must be Z'd out.

18.0 QUALITY ASSURANCE

18.1 All quality control data must be maintained and available for easy reference or inspection. In addition to quality control already discussed, the following requirements apply. Corrective action must be taken for unacceptable quality control results based on instructions given in this procedure, and in TriMatrix SOP GR-10-106.

18.2 Dilute and reanalyze all digestates having concentrations greater than the highest standard on the analytical curve.

18.3 For aqueous samples, include a minimum of one digestion blank (BLK), one high blank spike (BS) and one low blank spike (BS) with each batch of up to twenty samples. For solid samples, include a minimum of one digestion blank (BLK) and one BS with each batch of up to twenty samples. The BLK monitors contamination and carryover while the BS monitors digestion efficiency based on recovery. The BLK and BS must be carried through every step in the digestion and filtration procedure.

18.3.1 A BLK is an aliquot of ASTM Type II water digested as a sample. Any mercury found in the BLK must be at a concentration less than the reporting limit to be acceptable.

18.3.2 A BS is the BLK spiked at a high and low concentration.

18.3.2.1 Calculate BS recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike}}{\text{Spike Quantity}} \times 100$$

where:

C_{spike} = The analyzed concentration of the BS after spiking in ug/L or mg/kg

Spike Quantity = The concentration spiked into the BS in ug/L or mg/kg

18.3.2.2 Recovery of the high concentration BS must be within 85 – 115% of the expected value for aqueous samples. Statistical acceptance limits are used for solid samples. Recovery of the low concentration BS must be within 70 – 130% of the expected value. The low concentration BS is for internal use and is not reported.

18.4 Matrix spike and matrix spike duplicates are a sample spiked with a known mercury concentration. Prepare and analyze one matrix spike (MS) and one matrix spike duplicate (MSD) with each batch of up to twenty samples. The matrix spike monitors digestion efficiency in the specific sample matrix. A matrix spike duplicate is a replicate of the matrix spike using the same sample and monitors precision within the sample matrix.

18.4.1 Calculate matrix spike recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike} - C_{Orig}}{\text{Spike Quantity}} \times 100$$

where:
C_{spike} = The quantitated MS concentration in ug/L or mg/kg
C_{orig} = The unspiked sample concentration in ug/L or mg/kg
Spike Quantity = The spiked concentration in ug/L or mg/kg

18.4.2 Calculate precision between two spikes using the following equation:

$$\text{Relative Percent Difference} = \left| \frac{C_{MSD} - C_{MS}}{\frac{C_{MSD} + C_{MS}}{2}} \right| \times 100$$

where:
C_{MSD} = The MSD concentration as read from the instrument printout in ug/L or mg/kg
C_{MS} = The MS concentration as read from the instrument printout in ug/L or mg/kg

18.4.3 Matrix spike recovery must be within 70-130% of the expected recovery. However, statistical limits will be used when available but must not exceed 70 – 130%. MSD relative percent difference must be ≤20%.

18.4.4 If MS/MSD criteria fail, reanalyze once. If the second attempt fails, initiate corrective action:

18.4.4.1 Verify the instrument is operating correctly.

18.4.4.2 Verify all other quality control is acceptable.

18.4.4.3 Re-digest the sample and MS/MSD and/or narrate in accordance with TriMatrix SOP GR-10-106.

18.5 Calculate CRL recovery as follows:

$$\text{Percent Recovery} = \frac{C_{Spike}}{\text{Spike Quantity}} \times 100$$

where:

C_{spike} = The analyzed CRL concentration in ug/L
Spike Quantity = The CRL concentration (0.2 ug/L)

18.6 The Method of Standard Additions (MSA)

18.6.1 Use the method of standard additions for all TCLP extracts having 50% or less matrix spike recovery.

18.6.2 Use the method of standard additions on samples from at least one waste stream of an F-waste delisting petition project. If Section 18.6.1 is not met however, apply the method of standard additions to all samples in the delisting.

18.6.3 Standard addition involves adding known mercury concentrations to three sample aliquots. These aliquots are digested and analyzed. The unspiked sample concentration and three spiked aliquots are used to construct a linear regression curve. The curve is extrapolated to find the point where it crosses the x axis. The absolute value of this point is the sample mercury concentration.

18.6.4 Prepare three matrix spikes at 50%, 100%, and 150% of the mercury concentration found in the sample. Final volumes must be the same for each.

18.6.5 Digest and analyze to obtain mercury results for each (in ug/L). Using the MSA regression spreadsheet located on the library drive of the facility network, plot absorbance vs. concentration in ug/L. Construct a linear curve without forcing through the origin. The extrapolated x-intercept point is the sample mercury concentration. An example of an MSA plot is shown in Attachment 23.4. For results to be valid, the following limitations must be taken into consideration:

18.6.5.1 The regression curve must be linear, with a correlation coefficient of ≥0.995 and with a slope less than or equal to 20% of the external calibration curve slope.

18.6.5.2 The chemical form of the mercury added must respond the same way as the mercury in the sample.

18.6.5.3 The interference effect must be constant over the working range of concern.

18.6.5.4 The method of standard additions will not correct for additive interference.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

19.1.1 Initial Demonstration of Capability (IDC) Study

19.1.1.1 Prepare four second-source blank spikes at a concentration of 2.5 ug/L. Digest, filter and analyze following every step in the procedure.

19.1.1.2 Input the four results to the IDC spreadsheet located on the laboratory intranet library.

19.1.1.3 Average percent recovery must fall within 90 – 110% recovery. Standard deviation of the average must be ≤20% RSD.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the source of the problem, document changes to the procedure and/or techniques used then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed.

19.1.1.6 Give a copy of successful IDC spreadsheets and raw data to the quality assurance department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC) Study

19.1.2.1 The demonstration of capability study must be repeated annually.

19.1.2.2 The CDC may be accomplished in one of the following ways:

19.1.2.2.1 By repeating the IDC study

19.1.2.2.2 By using the last four results from an MDL study performed exclusively by the analyst during the course of routine sample analysis. Only the last four results may be used.

19.1.2.2.3 By using four consecutive blank spike results run during the course of routine sample analysis, exclusively by the analyst.

19.1.2.2.4 By successfully and exclusively analyzing a single-blind performance testing sample during the course of routine sample analysis.

19.2 A method detection limit (MDL) study must be completed annually in accordance with TriMatrix SOP GR-10-125 for each digestion procedure.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

22.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

22.3 *Test Method for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision I, September 1994, Method 7471A, *"Mercury in Solid and Semi Solid Waste* (Manual Cold-Vapor Technique)"

23.0 ATTACHMENTS

23.1 Instrument Maintenance Logbook Example

23.2 Sample Identification and Weighing Sheet Example – Aqueous

23.3 MSA Curve Example

23.4 Internal Chain-of-Custody Example

23.5 Sample Identification and Weighing Sheet Example – Solid

Attachment 23.1
Instrument Maintenance Logbook Example

TriMatrix Laboratories, Inc.

PSA Maintenance Logbook

Date	Analyst	Emission/Reference Readings		Changed Pump Tubes	Changed Lamp	Changed Probe	Replaced Sample Valve	Comments
5.06.03	DW							Replaced dryer tube
05/06/03	MSS	196.4	108.8	✓				
05/08/03	DW/MSS	200.1	110.8	✓				
05/09/03	MSS	185.9	114.1	✓				
05/09/03	MSS	196.1	112.1	✓				
05/13/03	MSS	195.1	114.1	✓				
05/14/03	MSS	190.4	112.2	✓				
05/15/03	MSS	193.1	113.1	✓				
05/16/03	MSS	193.9	111.3	✓				replaced GLS line & Dryer Tube
05/20/03	MSS	200.1	113.2	✓				
05/21/03	MSS	200.0	112.7	✓				
05/22/03	MSS	190.9	107.9	✓				
05/23/03	MSS	199.5	107.1	✓				
05/27/03	MSS	184.1	110.9	✓				replaced GLS line & Dryer Tube
05/05/03	MSS	180.3	107.9	✓				
06/06/03	MSS	197.1	114.0	✓				
06/09/03	MSS	200.1	115.0	✓				
06/11/03	MSS	200.2	114.8	✓				
06/23/03	MSS	200.5	115.1	✓				

file: MainMvalon.xls page: 3 of 25 revision: 1.0

Attachment 23.2
Sample Identification and Weighing Sheet Example – Aqueous

TriMatrix Laboratories, Inc.

Metals Laboratory Instrument Run Sequence Hg Aqueous

Instrument #: 216
Date:
Analyst:
SnCl #: 8110207

Data File:
Calibration BLK/ICB/CCB: 0.5% HNO3 LOT#1108080
Std/SCV prep date/analyst:

Standards/SCV prepped daily
Autosampler Position:

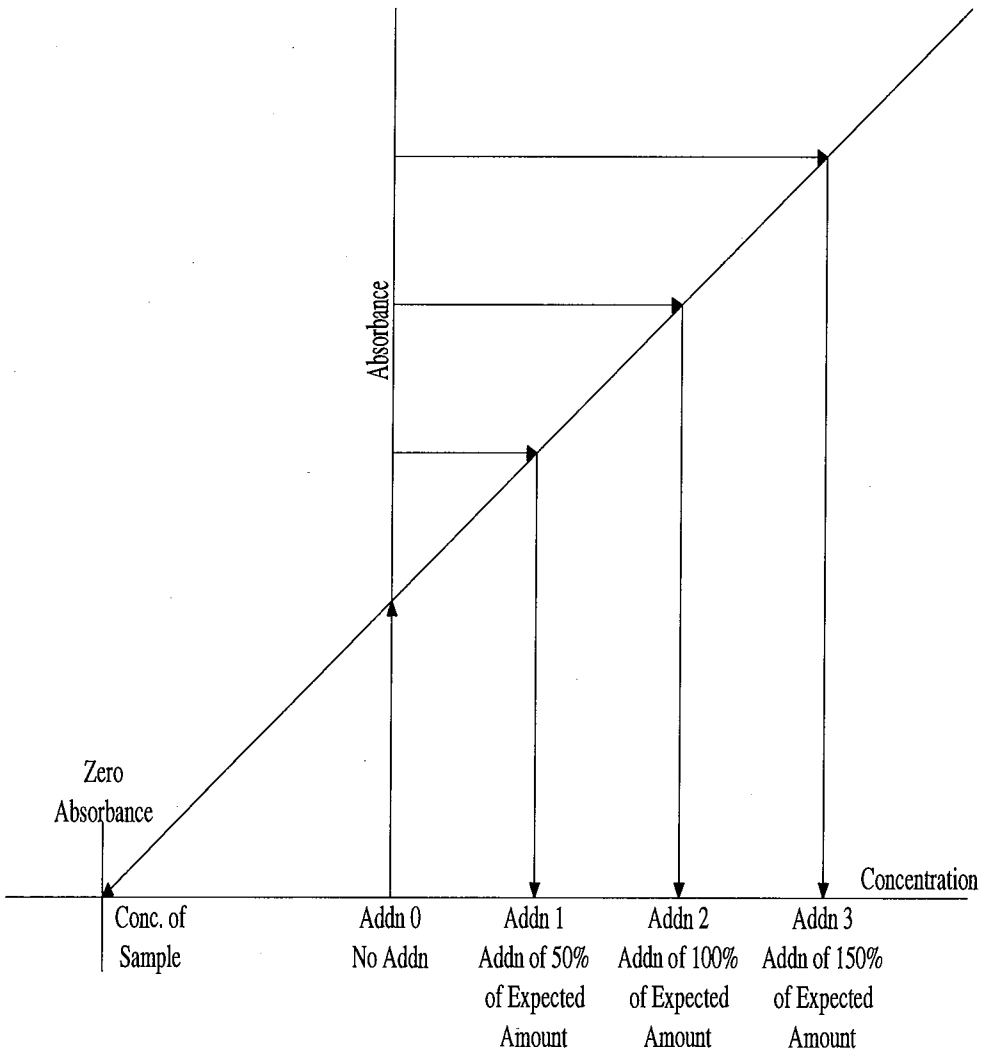
1. BLK/ICB/CCB/8050775
2. STD1/CRL/0.2 ug/L 8050776
3. STD2/0.5 ug/L 8050777
4. STD3/1.0 ug/L 8050778
5. STD4/CCV/2.0 ug/L 8050780
6. STD5/5.0 ug/L 8050781
7. SCV/3.0 ug/L 8100311

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
21					55				
22					56				
23					57				
24					58				
25					59				
26					60				
27					61				
28					62				
29					63				
30					64				
31					65				
32					66				
33					67				
34					68				
35					69				
36					70				
37					71				
38					72				
39					73				
40					74				
41					75				
42					76				
43					77				
44					78				

Hg_AQ.xls revision 1.0

Approved By: 1-15-09 **QA Officer** **Approved By:** 1-19-09 **Area Supervisor**

Attachment 23.3
MSA Curve Example



Attachment 23.4
Internal Chain-of-Custody Example

14-MAY-2003

TRIMATRIX LABORATORIES, INC.
CHAIN OF CUSTODY REPORT FOR AN ENTIRE SUBMITTAL

PAGE #6

CLIENT: MDEQ-RRD
SUBMITTAL: May 7 & 8, 2003 Soils
PROJECT: Former Osceola Refinery West Branch-Sag. Bay Dist.
PROJECT: 35923-7
Parameter: MERCURY, TOTAL
Method: CV/HG/7471/SOL
Ref Cit: USEPA-7471A
Matrix: SOIL

SAMPLE #	REMOVED BY: (SIGNATURE)	DATE & TIME REMOVED	RELINQUISHED BY: DATE & TIME	RECEIVED BY: DATE & TIME	DATE & TIME RETURNED
331322	M.A. Scott			05-5-03 1:30 PM	05-15-03 3:30 PM
331323	MSS				
331324	MSS				
331325	MSS				
331326	MSS				
331327	MSS				
331328	MSS				
331329	MSS				
331330	MSS				
331331	MSS				
331332	MSS				
331333	MSS				
331334	MSS				
331335	MSS				
331336	MSS				
331337	MSS			05-13-03 1:30 PM	05-15-03 3:30 PM

Attachment 23.5
Sample Identification and Weighing Sheet Example – Solid

TriMatrix Laboratories, Inc.

Metals Laboratory Instrument Run Sequence Hg Solids

Instrument #: 216
Date:
Analyst:
SnCl#: 8110207
Data File:
Calibration BLK/ICB/CCB: 0.5% HNO3 LOT#1108080
Std/SCV prep date/analyst:

Standards/SCV prepped daily
Autosampler Position:

1. BLK/ICB/CCB/8050782
2. STD1/CRL/0.2 ug/L 8050784
3. STD2/0.5 ug/L 8050786
4. STD3/1.0 ug/L 8050788
6. STD4/CCV/2.0 ug/L 8050789
6. STD5/5.0 ug/L 8050790
7. SCV/3.0 ug/L 8100310

Position #	ID	Dilution			Position #	ID	Dilution		
		Initial Volume (mL)	Final Volume (mL)	Dilution Factor			Initial Volume (mL)	Final Volume (mL)	Dilution Factor
11					45				
12					46				
13					47				
14					48				
15					49				
16					50				
17					51				
18					52				
19					53				
20					54				
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44					78				

Hg_solid.xls revision: 1.0

Approved By: 1-15-09 **QA Officer** **Approved By:** 1-19-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Block digestion of Solids for ICP and ICPMS

SW-846 Method 3050B

APPROVALS:

Area Supervisor: Marge A. Scott — Date: 01-15-09
Marge A. Scott

QA Officer: _____ — Date: 1/15-09
Tom C. Boocher

Operations Manager: _____ — Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-01-137
Revision Number: 1.5

Date Initiated: 2/06/01
Effective Date: 2/1/09

Date Revised: 1/15/09
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 18

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the block digestion of metals in sediment, sludge, soil and RCRA waste for ICP and ICP-MS analysis.

1.2 As the digestion does not attack silicate-bound metals, the procedure is not a total digestion technique for many samples.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 3050B, "Acid Digestion of Sediments, Sludge and Soils"

2.1.1 This procedure uses a 50 mL non-constricted block digestion cup as a deviation from method 3050B, which specifies a 250 mL block digestion vessel.

2.1.2 This procedure describes the use of 0.5 g dry weight as a deviation from method 3050B, which specifies the use of 1.0 g dry weight for digestion. However, acid ratios are maintained.

2.1.3 This procedure describes a routine use of the extra digestion step for antimony, tin, silicon and sometimes silver as a deviation from method 3050B, which specifies an optional digestion step for improved antimony, silver, barium and lead recovery.

3.0 SUMMARY OF PROCEDURE

3.1 Homogeneous and dried sediment, sludge and soil sample is weighed directly into a block digestion cup.

3.2 Samples are then digested with repeated additions of nitric acid then hydrogen peroxide.

3.3 If digesting for ICP, hydrochloric acid is used. If digesting for ICPMS, hydrochloric acid is not used.

3.4 All digestates are filtered then brought to a final volume of 50 mL.

3.5 Antimony, tin, silicon and sometimes silver are digested as follows:

3.5.1 Homogeneous and dried sediment, sludge and soil sample is weighed directly into a block digestion cup.

3.5.2 Samples are then digested with nitric acid and hydrochloric acid.

3.5.3 All digestates are filtered then the residue is washed with hot hydrochloric acid and hot laboratory reagent water to increase recovery.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure includes digestion for the following elements and instrumental techniques:

ICP	
Aluminum	Silicon
Calcium	Sodium
Iron	Strontium
Lithium	Titanium
Magnesium	
Potassium	

ICP or ICPMS			
Antimony	Chromium	Nickel	Zinc
Arsenic	Cobalt	Selenium	Gold
Barium	Copper	Silver	
Beryllium	Lead	Thallium	
Boron	Manganese	Tin	
Cadmium	Molybdenum	Vanadium	

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-100, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.3 TriMatrix SOP GR-01-129, *Inductively Coupled Plasma-Mass Spectrometry Perkin Elmer ELAN-6000/6100*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-01-115, *Mixing, Grinding and Drying Soils for Metals Analysis*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

5.7 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.8 TriMatrix SOP GR-16-103, *Glassware Cleaning and Preparation for Wet Chemistry and Metals Laboratories*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Refer to TriMatrix SOP GR-01-100 for a list of interferences and associated corrective actions when digesting for ICP analysis.

6.2 Refer to TriMatrix SOP GR-01-129 for a list of interferences and associated corrective actions when digesting for ICPMS analysis.

6.3 Some sludge matrices may not be applicable to this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Hot concentrated acids are used in standard and sample preparations. Wear disposable gloves, laboratory coats and approved safety glasses at all times when digesting samples and/or working in the laboratory.

7.3 Refer to the Material Safety Data Sheet (MSDS) library for specific information on any chemical used in the laboratory.

7.4 Check fume hoods to be sure each is functioning properly. All digestions must be done in a fume hood. If the hood is not working properly, immediately contact the area supervisor. Under no circumstances is any block digestion to be performed if the exhaust hood is not on and/or functioning properly.

7.5 Many chemicals and elements used are toxic. Minimize exposure by appropriate use of the personal protective equipment provided.

7.6 No food or drink is allowed in the digestion laboratory at any time, including gum.

7.7 Wash hands before starting work. Substances on the skin may interfere with metals analysis. Wash hands after finishing work. Chemical residuals can be injested and/or carried out of the laboratory if not removed.

7.8 Before leaving the digestion laboratory, remove disposable gloves and laboratory coat. Chemical and/or acid residue transport outside the digestions laboratory needs to be kept to a minimum.

7.9 Rinse all micropipette tips and beakers before disposal or cleaning.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are typically received by the laboratory in 500 mL sample jars.

8.2 Dry at least 30 g of solid sample for analysis and quality control in accordance with TriMatrix SOP GR-01-115. Dry a larger sample aliquot to obtain at least 30 g for low-solids sludge.

8.3 The maximum holding time for all preserved samples requiring this digestion is 180 days.

8.4 Digestates (solubilized samples) ready for analysis need not be refrigerated.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Micropipette, capable of delivering 250 µL

9.2 Block digester for metals digestion, Environmental Express, HotBlock™, model SC154

9.3 Block digestion cups, 50 mL, certified pre-cleaned and calibrated, Environmental Express, catalog SC475

9.4 Ribbed watchglass, disposable, Environmental Express, catalog SC505

9.5 Filtermate™, Environmental Express, catalog SC0501, for digestate filtration

9.6 Non-ribbed watchglass, disposable, SCP Science, catalog 010-500-081

9.7 Thermometers, NIST-traceable, digital, capable of reading to at least 100° C in 0.1° C increments.

9.8 Analytical balance, capable of weighing to 0.0001 g

9.9 Filter paper, Whatman no. 41 or equivalent

9.10 PTFE sieve, USS #10

9.11 Volumetric flask, 50 mL, type A

9.12 Filter funnels

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document micropipette calibration in accordance with TriMatrix SOP GR-10-111.

10.2 Clean the block digester and counters daily or if a spill occurs.

10.3 Calibrate the analytical balance in accordance with TriMatrix SOP GR-10-113.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water (ASTM Type II), MilliQ water system

11.2 Nitric acid (HNO_3), concentrated, trace metal grade

11.3 Prepare 1:1 (v/v) HNO_3 as follows:

11.3.1 Carefully measure 50 mL of HNO_3 to 50 mL of laboratory reagent water.

11.3.2 Prepare fresh daily.

11.4 Prepare 5% (v/v) HNO_3 as follows:

11.4.1 Carefully measure 5 mL of HNO_3 to 95 mL of laboratory reagent water.

11.4.2 Prepare fresh daily.

11.5 Hydrochloric acid (HCl), concentrated, trace metal grade

11.6 Hydrogen peroxide (H_2O_2), 30%, ACS grade

12.0 STANDARDS PREPARATION

12.1 Multi-element spiking solutions are purchased as commercially prepared solutions.

12.2 Refer to Attachments 20.1 and 20.2 for ICP spiking solution examples.

12.3 Refer to Attachments 20.3 and 20.4 for ICPMS spiking solution examples.

13.0 ANALYTICAL PROCEDURE

13.1 Sediment, soil and sludge digestion is as follows:

13.1.1 Preheat the block digester and adjust the temperature so 50 mL of 5% (v/v) nitric acid is maintained at 85° C. The addition of a watchglass at 85° C adds 10 degrees and maintains the digestion at 95° ±5° C while refluxing.

13.1.2 Mix and/or grind dried sample thoroughly, passing through a USS #10 sieve if necessary. For each sample, transfer 0.5 ±0.0005 g to a block digester cup. For best results, weigh sample directly into a tared digester cup at the balance. Record the actual mass used.

13.1.3 For all digestions, add 5.0 mL of 1:1 (v/v) HNO_3. Cover with a non-ribbed watchglass and reflux in the block digester at 95° ±5° C for 15 minutes. Do not boil.

13.1.4 After 15 minutes, remove from the digester to cool then measure 2.5 mL concentrated HNO_3 into each cup and cover with a non-ribbed watchglass. Reflux for 30 minutes. Repeat this step by adding 2.5 mL acid volumes and refluxing until brown fumes are no longer given off. Brown fumes indicate the digestion is not yet complete.

13.1.5 Once digestion is complete, change to a ribbed watchglass and evaporate to approximately 2.5 mL. Do not boil or let any part of the digestion cup bottom go dry. Repeat any sample digestion in which the digestion cup goes dry. Boiling should not occur if the block digester is properly adjusted.

13.1.6 When the volume is approximately 2.5 mL, remove and cool to room temperature. It is very important to have digestates at room temperature to avoid splattering when adding hydrogen peroxide. Add 1.0 mL of laboratory reagent water and 1.5 mL of 30% H_2O_2 slowly. Let any initial reaction occur without heat.

13.1.7 Reduce the block temperature by 10° C to control excess effervescence while still maintaining enough heat to maintain the peroxide reaction. Cover with a non-ribbed watchglass and return to the block digester, heating until any reaction stops. If effervescence becomes so vigorous that splattering is immanent, control by lifting from the heat until it subsides. Do not let digestate overflow the digestion cup as analyte will be lost. Repeat the digestion if overflow occurs.

13.1.8 For samples that continue to react, carefully add another 1.0 mL of 30% H_2O_2. Do not add more than a total of 5.0 mL. Repeat until no further reaction occurs or until the maximum 5.0 mL volume has been added.

13.1.9 After the H_2O_2 digestion is complete, cover with a ribbed watchglass and again evaporate to 2.5 mL. Do not boil or let the bottom of any digestion cup go dry.

13.1.10 After reducing the volume, remove from the heat and cool to room temperature again. For ICP sample analysis only, add 5.0 mL of concentrated HCl. Cover with a non-ribbed watchglass and reflux at 95° ±5° C for 15 minutes. Skip this step if the digestion is for ICPMS analysis.

13.1.11 Cool to room temperature, dilute to 50 mL with laboratory reagent water then use a Filtermate™ for removing particulates.

13.1.11.1 Perform the filtration slowly with little pressure placed to the plunger.

13.1.11.2 If excessive backpressure occurs, stop filtering and let the digestate settle out before continuing. Applying too much pressure to the Filtermate™ plunger can cause the filter to fail allowing particulates into the filtrate.

13.1.11.3 Do not rinse the filter since the final volume has already been measured.

13.1.12 Samples are ready for analysis after filtering.

13.2 Sediment, soil and sludge digestion for antimony, tin, silicon and sometimes silver is as follows:

13.2.1 Preheat the block digester and adjust the temperature so 50 mL of 5% (v/v) nitric acid is maintained at 85° C. The addition of a watchglass at 85° C adds 10 degrees and maintains the digestion at 95° ±5° C while refluxing.

13.2.2 Mix and/or grind dried sample thoroughly, passing through a USS #10 sieve if necessary. For each sample, transfer 0.5 ±0.0005 g to a block digester cup. For best results, weigh sample directly into a tared digester cup at the balance. Record the actual mass used.

13.2.3 Add 1.25 mL concentrated HNO_3 and 5.0 mL concentrated HCl. Cover with a non-ribbed watchglass and heat at 95 ±5° C for 15 minutes. Do not boil.

13.2.4 After 15 minutes, filter through Whatman no. 41 filter paper, collecting the filtrate in a 50 mL volumetric flask. Wash the filter paper while still in the filter funnel with no more than 2.5 mL of hot (~95° C) concentrated HCl then with 10 mL of hot ASTM Type II water. Collect all washings in the same 50 mL volumetric flask.

13.2.4 Return the filter and residue to the digestion cup. Add 2.5 mL of hot (~95° C) concentrated HCl. Return to the block digester with a non-ribbed watchglass and heat at 95 ±5° C until the filter paper dissolves. Remove from the digester. Rinse the watchglass and digestion cup sides with laboratory reagent water. Filter again and collect the filtrate in the same 50 mL volumetric flask. Let the filtrate cool to room temperature in the flask then dilute to volume with laboratory reagent water.

13.2.5 High concentrations of metal salts may precipitate upon cooling. If precipitation occurs, do not dilute to volume. Instead, add up to 5.0 mL of concentrated HCl to dissolve the precipitate. After the precipitate dissolves, dilute to volume.

13.2.6 After diluting, digestates are ready for analysis by ICP.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used.

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data.

14.5 If internal chain-of-custody (CoC) is required, it is very important that CoC forms be filled in completely and correctly. Organize, transfer and/or archive CoC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digester maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

14.8 Silicon digested by this procedure must be reported and/or narrated as being "recoverable silicon" since most soil silicates are not digested by the acids used.

15.0 QUALITY ASSURANCE

15.1 At least one digestion blank (BLK) must be carried through the entire digestion to monitor contamination with each digestion batch of up to 20 samples. Prepare the blank by digesting an empty digestion cup as a sample and using the maximum acid additions and digestion steps used in the digestion batch.

15.2 At least one blank spike (BS) must be carried through the entire digestion to document recovery without matrix interference in each digestion batch of up to 20 samples. Prepare blank spikes as follows:

15.2.1 For ICP digestions, measure 5.0 mL 1:1 (v/v) HNO_3 into a digestion cup and spike with 250 uL each of standards SSW-1A and SSW-SSS-2A. Perform the digestion using the maximum acid additions and digestion steps used in the sample batch.

15.2.2 For ICPMS digestions, measure 5.0 mL 1:1 (v/v) HNO_3 into a digestion cup and spike with 100 uL each of standards HP solutions A and B. Perform the digestion using the maximum acid additions and digestion steps used in the sample batch.

15.3 At least one matrix spike (MS)/matrix spike duplicate (MSD) must be prepared for each digestion batch of up to 20 samples to monitor matrix recovery and precision. Perform a MS/MSD for each matrix being digested (sediment, soil, sludge or unknown).

15.3.1 For ICP matrix spikes, measure 0.5 g of dried sample into a digestion cup and spike with 250 uL each of standards SSW-1A and SSW-SSS-2A. Perform the digestion using the same acid additions and digestion steps used in the un-spiked sample.

15.3.2 For ICPMS matrix spikes, measure 0.5 g of dried sample into a digestion cup and spike with 100 uL each of standards HP solutions A and B. Perform the digestion using the same acid additions and digestion steps used in the un-spiked sample.

15.4 Digestates must not boil during the evaporation step. If a digestate begins to boil during evaporation, immediately remove from the block and reduce the temperature. Some boiling will be evident during the reflux step. However, vigorous boiling during reflux will diminish the water-acid azeotrope and cause loss of analyte. If a digestate begin to boils vigorously during refluxing, immediately remove from the block and reduce the temperature. If boiling during evaporation or vigorous boiling during refluxing is not immediately addressed, discard and the re-prepare the sample and/or samples affected. Monitor the block temperature closely to minimize re-preparation.

15.5 Each commercially purchased spiking solution must be used within its expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal.

15.6 If a sample evaporates to dryness, some metals will be volatilized. If any portion of the digestion cup bottom goes dry, discard the digestate and re-prepare the sample in another batch.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). Analysts responsible for sample processing by this procedure must pass an IDC study first.

16.1.1 Initial Demonstration of Capability Study

16.1.1.1 Spike four digestion cups as in a blank spike preparation. Digest the four spikes following every step in this procedure and obtain analysis results for the four blank spikes. Analysis of the spikes must also include every step in the analytical procedure. Enter all four results obtained into the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must fall within the blank spike control limits found in Element™. Relative standard deviation must be less than or equal to 20%.

16.1.1.2 Alternatively, the last four of seven results used in an MDL study may be used as the IDC, if the MDL study was run exclusively by the analyst. ONLY the last four results may be used.

16.1.1.3 If either criterion is not met, locate and correct the source of any problem and repeat the study to confirm the corrective action was successful. Repeated failure however, will confirm a general problem with the procedure or techniques used. If multiple failures occur, correct the procedure or techniques used, and repeat the study.

16.1.1.4 Samples may not be processed by any analyst until an IDC study has been successfully completed. Give copies of successful IDC study spreadsheets and raw data to the quality assurance department for training documentation.

16.1.2 Continuing Demonstration of Capability Study

16.1.2.1 Annually, a Continuing Demonstration of Capability (CDC) study is required by each analyst.

16.1.2.2 A CDC may be accomplished by repeating the IDC study, by using the last four results from an MDL study, or by using four consecutive and exclusively run blank spike results obtained from routine sample analysis since the IDC study was completed. Alternatively, successful completion of an exclusively digested performance testing study result run since completing the IDC may be used.

16.1.2.3 Process CDC data using the same demonstration of capability spreadsheet and in the same manner as the IDC.

16.2 Method Detection Limit Studies

16.2.1 A Method Detection Limit (MDL) study must be performed annually for this procedure. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence the value is above zero.

16.2.2 The procedure used for an MDL study is based on the method given in the Code of Federal Regulations, Title 40, Part 136, Appendix B, latest revision.

16.2.3 Seven replicate digestions are performed using digestion cups spiked with all elements of interest at the estimated minimum reportable concentration.

16.2.4 The standard deviation of the average for each element, using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

16.2.5 If the concentration spiked is between the calculated MDL and ≤10 times the calculated MDL, and there are no zero percent recoveries in the seven results then the MDL passes.

16.2.6 If the MDL study does not pass acceptably, it must be re-digested. If a study needs re-digested at a different concentration, the entire set of seven needs re-digested. If a study does not pass due to poor reproducibility on one result, only that one result needs re-digested. However, only one result of the seven can be re-digested without re-digesting the entire study.

16.2.7 All seven MDL study results do not need digested in the same digestion batch.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 3050B, "Acid Digestion of Sediments, Sludge, and Soils"

20.0 ATTACHMENTS

20.1 ICP Spiking Solution SSW-1A Certificate of Analysis Example

20.2 ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

20.3 ICP/MS Spiking Solution A Certificate of Analysis Example

20.4 ICP/MS Spiking Solution B Certificate of Analysis Example

20.5 Work Order Report Example

20.6 Preparation Batch Report Example

Attachment 20.1
ICP Spiking Solution SSW-1A Certificate of Analysis Example

INORGANIC VENTURES

CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-1A

Lot Number: **Y-MEB188079**

Matrix: 2% HNO3(abs)

A607108
DA
7-06-06

2,500.00 µg/mL each:
Ca, K, Mg, Na,
250.00 µg/mL each:
Al, As, P, Se, Tl,
50.00 µg/mL each:
Ag, B, Ba, Cd, Co, Cr3, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn,
5.00 µg/mL each:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	249.8 ± 0.7 µg/mL	Arsenic, As	250.2 ± 0.8 µg/mL	Barium, Ba	50.16 ± 0.08 µg/mL
Beryllium, Be	5.004 ± 0.010 µg/mL	Boron, B	50.07 ± 0.10 µg/mL	Cadmium, Cd	50.16 ± 0.22 µg/mL
Calcium, Ca	2,500 ± 6 µg/mL	Chromium+3, Cr3	50.11 ± 0.16 µg/mL	Cobalt, Co	50.09 ± 0.12 µg/mL
Copper, Cu	50.17 ± 0.14 µg/mL	Iron, Fe	49.84 ± 0.32 µg/mL	Lead, Pb	50.17 ± 0.11 µg/mL
Lithium, Li	49.91 ± 0.08 µg/mL	Magnesium, Mg	2,501 ± 5 µg/mL	Manganese, Mn	49.97 ± 0.12 µg/mL
Nickel, Ni	50.10 ± 0.12 µg/mL	Phosphorus, P	249.5 ± 0.6 µg/mL	Potassium, K	2,500 ± 5 µg/mL
Selenium, Se	249.8 ± 0.8 µg/mL	Silver, Ag	50.01 ± 0.11 µg/mL	Sodium, Na	2,497 ± 1 µg/mL
Strontium, Sr	50.05 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.98 ± 0.14 µg/mL
Zinc, Zn	50.09 ± 0.12 µg/mL				

Certified Density: 1.049 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

Approved By: PD 1-15-09 QA Officer

Approved By: MAS 01-15-09 Area Supervisor

Attachment 20.2
ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

INORGANIC VENTURES™ CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-SSS-2A

Lot Number: **Y-MEB201134**

Matrix: tr. HF, 2% HNO3(abs)

A609280
DSL 9-11-06
exp. 10-01-07

250.00 µg/mL each:
Sb, Si, Sn,
50.00 µg/mL each:
Mo, Ti

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	249.1 ± 0.4 µg/mL	Molybdenum, Mo	50.02 ± 0.10 µg/mL	Silicon, Si	249.7 ± 0.8 µg/mL
Tin, Sn	250.6 ± 0.9 µg/mL	Titanium, Ti	50.14 ± 0.08 µg/mL		

Certified Density: 1.015 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS

· "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

· This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Attachment 20.3
ICP/MS Spiking Solution A Certificate of Analysis Example

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution A
Lot # 605319

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 3.5%	mg/L ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
Laboratory Director

A602735
exp 3-31-07
Rec'd 2-28-06

Exp Date: MAR '07
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

Approved By: 1-15-09 QA Officer Approved By: MAS 01-15-09 Area Supervisor

Attachment 20.4
ICP/MS Spiking Solution B Certificate of Analysis Example

ENVIRONMENTAL EXPRESS

Certificate of Analysis

HP2079-1-1L
Solution B
Lot # 605319

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.98+%	HNO_3, 2% + Tr HF	100 mg/L ± 0.5% Antimony, Silicon, Titanium, Molybdenum, Tin

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C Rains

Theodore C. Rains, Ph.D.
Laboratory Director

A602736
exp 3-31-07
rcvd 2-28-06

Exp Date: MAR '07
MSDS ATTACHED

490 Wando Park Blvd.
Mt. Pleasant, South Carolina 29464
Phone: 1.843.881.6560
Toll Free: 1.800.343.5319
FAX: 1.843.881.3964
www.environmentalexpress.com

Approved By: RD 1-15-09 QA Officer — Approved By: UAS 01-15-09 Area Supervisor

Attachment 20.5
Work Order Report Example

TriMatrix Laboratories, Inc. | WORK ORDER 0609330 | Printed: 7/11/2007 5:32:43PM

Page 1 of 6

Client: [redacted]
Project: [redacted]
Work Order: [redacted]

Project Manager: **Gary L. Wood**
Project Number: **[none]**
SDG:

Report To:
[redacted]

Invoice To:
[redacted]

Client Due Date: **Oct-04-06 16:00 (10 day TAT)**
Date Received: Sep-20-06 12:00 25.9°C
Date Logged In: Sep-21-06 16:37
W.O. Comments: *3FL*
Send cc of report with invoice to Richard Hodge.

Report Level: **3FL**
Received By: William D. Cole
Logged In By: Kristyn N. Courtnay

Analysis	Lab Due Date	TAT	Expires	Analysis Comments
0609330-01 06-35 [Waste] Sampled Sep-12-06 10:00 Eastern				**RCRA 8 Metals (Total & TCLP): Report pHs**
Ag TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ag Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cd TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cd Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cr TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Cr Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Hg TCLP 7470A	Oct-03-06 17:00	10	Oct-10-06 10:00	
Hg Total 7471A	Oct-03-06 17:00	10	Oct-10-06 10:00	
Pb TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Pb Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Se TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Se Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
TCLP Metals Extraction	Oct-03-06 17:00	10	Sep-26-06 10:00	
0609330-02 06-36 [Waste] Sampled Sep-12-06 10:00 Eastern				**RCRA 8 Metals (Total & TCLP): Report pHs**
Ag SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720
Ag TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ag Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720
As TCLP 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
As Total 6010B	Oct-03-06 17:00	10	Mar-11-07 10:00	
Ba SPLP 6010B	Jan-01-80 00:00		Sep-12-06 10:00	Added for SequenceQC in: 6092720

wko_TM_Basic.rpt

Approved By: GL 1-15-9 **QA Officer**
Approved By: MAS 01-15-09 **Area Supervisor**

Attachment 20.6
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0610939 Page 1 of 3

Printed: 7/11/2007 5:25:28PM

Metals, Waste, 3050B Digestion

(No Surrogate)

Batch Comments: MAS 09/25/06 &:00AM-3:30PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0609330	Se Total 6010B	0609330	Pb Total 6010B	0609330	Cr Total 6010B
0609330	Cd Total 6010B	0609330	Ba Total 6010B	0609330	As Total 6010B
0609330	Ag Total 6010B				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client/ QC Type*	*Extraction Comments*
0610939-BLK1		Sep-25-06 07:00	MAS	0.5	50					BLANK	
0610939-BS1		Sep-25-06 07:00	MAS	0.5	50			A607108	250	LCS	
0610939-MS1		Sep-25-06 07:00	MAS	0.5	50		0609330-02	A607108	250	MATRIX SPIKE	
0610939-MS2		Sep-25-06 07:00	MAS	0.5	50		0609330-02	A607109	250	MATRIX SPIKE	
0610939-MSD1		Sep-25-06 07:00	MAS	0.5005	50		0609330-02	A607108	250	MATRIX SPIKE DUP	
0610939-MSD2		Sep-25-06 07:00	MAS	0.5005	50		0609330-02	A607109	250	MATRIX SPIKE DUP	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-01	A	Sep-25-06 07:00	MAS	0.4998	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-02	A	Sep-25-06 07:00	MAS	0.5001	50					[redacted]	
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50					[redacted]	
0609330-03	A	Sep-25-06 07:00	MAS	0.4995	50					[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: DA 1-15-09 **QA Officer**

Approved By: MAS 01-15-09 **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Digestion of Mercury in Water, Wastewater and Aqueous Waste

EPA Method 245.1
SW-846 Method 7470A

APPROVALS:

Area Supervisor: Marge A. Scott — Date: 01-23-09
Marge A. Scott

QA Officer: _____ — Date: 1-22-9
Tom C. Boocher

Operations Manager: _____ — Date: 1/23/09
Jeff P. Glaser

Procedure Number: GR-01-140
Revision Number: 0.4

Date Initiated: 2/19/03
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Marge A. Scott
Total Number of Pages: 19

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the digestion of total mercury (inorganic and organic) in samples of groundwater, potable water, surface water, saline water, mobility leachate, and in aqueous domestic and industrial waste.

1.2 The minimum reporting limit is 0.2 ug/L.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

2.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

3.0 SUMMARY OF PROCEDURE

3.1 Prior to analysis, all client samples and quality control must be digested to convert organo-mercury complexes to inorganic mercury.

3.2 A measured sample aliquot, acids and potassium permanganate-potassium persulfate are transferred to a block digestion vessel and refluxed for 2 hours at 90-95° C.

3.3 The digestate is then prepared for analysis by semi-automated cold vapor atomic absorption spectrometry with the addition of hydroxylamine hydrochloride to reduce excess permanganate.

3.4 Inorganic mercury is converted to mercury in the Hg^{2+} state during the digestion, for detection and quantitation.

4.0 PARAMETER OR COMPOUND LIST

4.1 Mercury

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-111, *Micropipette/Macropipette Calibration and Verification*, latest revision

5.3 TriMatrix SOP GR-01-123, *Mercury in Aqueous and Solid Samples by Semi-Automated Cold Vapor Atomic Absorption Spectrometry*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Action*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Soil often contains mercury at low levels. When soil samples are mixed and ground, liquid samples may become contaminated. Therefore, keep the preparation area for aqueous mercury samples free of dust and dirt to avoid cross-contamination.

6.2 Also, areas where Kjeldahl nitrogen (TKN) and/or chemical oxygen demand (COD) samples are being prepared and analyzed can be sources of contamination if not properly coordinated.

6.3 If a mercury thermometer is broken anywhere in the laboratory, notify the metals laboratory.

6.4 Mercury may be lost if the block digestor temperature exceeds 95° C. Use a digital thermometer to monitor block digestion temperature, to prevent mercury loss from boiling. Record block digestor temperatures on the pre-treatment benchsheet.

6.5 Reagents may become contaminated over time. If contamination is suspected before a reagent's assigned expiration date, discard it and acquire or prepare fresh reagent.

6.6 Volatile materials such as chlorine which absorb at 253.7 nm are a positive interference. If volatile gases are suspect, notify the instrumental analyst to purge the dead air space in each digestion cup with an inert gas before adding stannous chloride solution.

6.7 Sulfide, chloride, copper (>10 mg/L) and tellurium are interferences.

6.8 Organic compounds which absorb around 253.7 nm are interferences. Such interferant concentration levels are difficult to isolate and quality control must be strictly followed to minimize.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids and oxidizers are used in this procedure. Wear disposable gloves, approved safety glasses and appropriate protective clothing at all times when handling these and other reagents. Always wear disposable gloves when handling digestates as well. Refer to the MSDS library on the laboratory intranet library for information on these or other chemicals utilized in the laboratory.

7.3 Mercury exists in many forms and is toxic in a variety of ways. Mercury vapor is toxic if inhaled. Do not operate any instrument if it is not properly vented. Mercury can also be absorbed through the skin. Wear

disposable gloves at all times when handling laboratory chemicals. Elemental Hg and Hg^{2+} are toxic if ingested. Always wash hands after handling mercury or any other chemical used in the laboratory.

7.4 No food or drink is allowed in the metals laboratory or any other laboratory within the facility. Food and drink may be consumed in laboratory offices if separated from the laboratory but may only be stored in the facility cafeteria.

7.5 Wash hands before starting work. Chemicals may be present on the skin that can interfere with mercury analysis. Wash hands before leaving the metals laboratory. Chemicals and acids may be on the skin that could eventually be ingested or passed to a third party through casual contact.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples must be acidified at the time of collection to a pH of <2. If the pH is not <2 when received by the laboratory, lower to <2 during the sample receipt process using 1:1 nitric acid/water. The maximum volumes of acid added to a sample (including initial volume) are:

Container Size	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10.0

NOTE: Sample preservation must occur in the original container to prevent the loss of any analytes adsorbed to the container wall. It is not permissible to remove and acidify a sample aliquot.

8.2 If a pH adjustment is necessary, a minimum of 24 hours must elapse before the sample digestion process may begin.

8.3 Consult the project chemist for instruction if the pH cannot be adjusted to <2 using the specified maximum amount of acid. Samples prepared and analyzed without proper pH preservation, results must be narrated as such.

8.4 Samples may be collected in glass or plastic. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.

8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 550 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.

8.6 Properly acid-preserved samples can be held for up to 28 days prior to digestion. Refrigeration is not required for samples or digestates.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Micropipette, capable of delivering 100 to 1000 µL

9.2 HotBlock™ block digestor, Environmental Express, model SC154

9.3 Polypropylene Digestion vessels certified pre-cleaned and pre-calibrated to 50 mL, Environmental Express, catalog SC-475

9.4 Block digestor cover

9.5 Filtermate, Environmental Express, catalog SC0501

9.6 Carboy, 50 L, high density polyethylene, with spigot

9.7 Beaker, 1 L, polyethylene

9.8 Volumetric flasks, type A, glass, 100 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document micropipette calibration daily. Refer to TriMatrix GR-10-111 for micropipette calibration instructions.

10.2 Clean the block digestor and counters daily, and when spills and/or splattering occur.

11.0 CHEMICALS AND REAGENTS

11.1 Concentrated nitric acid (HNO_3), trace-metal grade

11.2 Concentrated sulfuric acid (H_2SO_4), trace-metal grade

11.3 Metals laboratory reagent water

11.3.1 ASTM Type II Milli-Q system water is fed into an all-glass Barnstead distillation unit. The distillate is immediately stored in a 50 L high-density polyethylene carboy.

11.3.2 This is the only water acceptable for use in the metals laboratory and will be designated as metals laboratory reagent water.

11.4 Potassium persulfate ($K_2S_2O_8$), ACS grade or equivalent, prepare a 5% (w/v) solution as follows:

11.4.1 Weigh out 25 g ACS-grade $K_2S_2O_8$ into a 1 L polyethylene bottle. Add 500 ml distilled reagent water to the beaker. Place a stir bar in the solution, cover and stir on a magnetic stir plate until all crystals have dissolved.

11.4.2 This solution takes several hours to prepare. Keep the reagent in a tightly capped bottle when not in use to avoid contamination. Prepare fresh weekly or sooner if a precipitate forms.

11.5 Potassium permanganate ($KMnO_4$), low-mercury ACS grade, prepare a 5% solution (w/v) as follows:

11.5.1 Weigh out and transfer 25 g $KMnO_4$ crystals to a 1 L polyethylene bottle. Add 500 ml metals laboratory reagent water to the container. Place a stir bar in the beaker then cover and stir on a magnetic stir plate until all crystals are dissolved. Since this solution is nearly saturated it may take several hours to prepare. Prepare fresh daily.

11.5.2 Update the standard number in Element™ when a new lot number of $KMnO_4$ is used in the preparation.

11.5.3 If $KMnO_4$ solution is spilled, immediately soak up with absorbent pads or paper towels then clean with water. Place all contaminated pads or towels in a plastic bag to be appropriately disposed of. If a brownish stain remains from the spill, carefully remove by applying hydroxylamine hydrochloride. Prevent exposure to the skin and eyes at all times by wearing disposable gloves, safety glasses and a laboratory coat.

11.5.4 Potassium permanganate absorbs mercury when exposed to laboratory air. Keep the reagent bottle tightly sealed when not in use. Store in a dark cabinet to minimize autocatalytic reduction.

11.6 Sodium chloride (NaCl)/hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$), prepare a 12% solution (w/v) as follows:

11.6.1 Weigh out and transfer 30 g of NaCl and 30 g of $NH_2OH{\cdot}HCl$ to a 500 mL polyethylene bottle. Add 250 mL of distilled reagent water and a stir bar.

11.6.2 Place the container on a stir plate and stir until all crystals are dissolved. Prepare fresh daily.

11.6.3 Update the standard number in Element™ when a new lot number of either NaCl or $NH_2OH{\cdot}HCl$ is used in the preparation.

12.0 STANDARDS PREPARATION

12.1 Mercury stock standard is purchased commercially at 1000 mg/L from Inorganic Ventures. The second-source secondary calibration verification (SCV) standard is purchased at 1,000 mg/L from SCP Science.

12.1.1 Purchased mercury solutions have an expiration date of the date specified by the manufacturer.

12.1.2 Log each standard into Element™.

12.1.3 All stock standards are single-element solutions.

12.2 Intermediate standard (1000 ug/L mercury)

12.2 Intermediate standard (1000 ug/L mercury)

12.2.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.

12.2.2 Add about 20 mL of metals laboratory reagent water to the flask.

12.2.3 Add 0.5 mL concentrated trace-metal grade HNO_3 then swirl to mix.

12.2.4 Pipette 100 µL of 1000 mg/L mercury standard into the flask.

12.2.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.

12.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.3 Prepare calibration standards as follows:

12.3.1 Prepare 0 ug/L (a blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 2.0 ug/L, and 5.0 ug/L mercury standards. Begin the preparation by labeling six 100 mL volumetric flasks. Rinse the flasks several times with metals laboratory reagent water.

12.3.2 Add approximately 20 mL metals laboratory reagent water to each flask.

12.3.3 Add 0.5 mL concentrated trace-metal grade HNO_3 to each flask and swirl.

12.3.4 Pipette 500 µL of 1000ug/L Hg standard from Section 12.3 into the vessel labeled "5.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.5 Pipette 200 µL of 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "2.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.6 Pipette 100 µL of 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "1.0 ug/L Hg". Dilute to volume with metals laboratory reagent water then cap and mix.

12.3.7 Pipette 50 µL of the 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "0.5 ug/L Hg". Dilute to volume with metals laboratory water then cap and mix.

12.3.8 Pipette 20 µL of the 1000 ug/L Hg standard from Section 12.3 into the vessel labeled "0.2 ug/L Hg". Dilute to volume with metals laboratory water then cap and mix.

12.3.9 Dilute the volumetric labeled "Hg blank" to volume with metals laboratory water then cap and mix.

12.9.10 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation.

12.4 Prepare the Secondary Source Verification (SCV) as follows:

12.4.1 Intermediate SCV standard (1000 ug/L mercury)

12.4.1.1 Rinse a clean 100 mL volumetric flask with metals laboratory reagent water several times.

12.4.1.2 Add about 20 mL of metals laboratory reagent water to the flask.

12.4.1.3 Add 0.5 mL concentrated trace-metal grade HNO_3 then swirl to mix.

12.4.1.4 Pipette 100 µL of 1000 mg/L second-source mercury standard into the flask.

12.4.1.5 Dilute to the volumetric mark with metals laboratory reagent water, cap tightly and mix well.

12.4.1.6 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.4.2 Prepare the secondary calibration verification (SCV) standard as follows:

12.4.2.1 Label a 100 mL volumetric flask as "3.0 ug/L SCV Hg". Rinse the flask several times with metals laboratory reagent water.

12.4.2.2 Add approximately 20 mL metals laboratory reagent water.

12.4.2.3 Add 0.5 mL concentrated trace-metal grade HNO_3 to each flask and swirl.

12.4.2.4 Pipette 0.30 mL of 1000 ug/L Hg standard from Section 12.4.1 into the volumetric. Dilute to volume with metals laboratory reagent water then cap and mix.

12.4.2.5 Prepare daily. Update the standard number in Element™ when a new lot number of 1000 mg/L stock standard is used in the preparation

12.5 Complete preparation of the calibration and SCV standards as follows:

12.5.1 Measure 30 mL of each calibration standard and the SCV into digestion vessels.

12.5.2 Pipette 0.75 mL of concentrated HNO_3 into each vessel.

12.5.3 Pipette 1.5 mL of concentrated H_2SO_4 into each vessel.

12.5.4 Swirl to mix thoroughly.

12.5.5 Pipette 4.5 mL of 5% $KMnO_4$ into each vessel and let stand for 15 minutes.

12.5.6 Pipette 2.4 mL of 5% $K_2S_2O_8$ into each vessel.

12.5.8 Place in the block digestor and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C.

12.5.7 After digesting, cool to room temperature then pipette 1.80 mL of 12% $NaCl/NH_2OH{\cdot}HCl$ into each digestion vessel.

12.5.9 After adding, bring back to 50 mL with metals laboratory reagent water then cap tightly and mix thoroughly.

12.5.10 After a delay of at least 30 seconds, calibration standards and SCV are ready for the stannous chloride addition and immediate analysis.

12.6 Spike samples for quality control as follows:

12.6.1 Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 10 samples for each wastewater and/or potable water matrix run in accordance with method 245.1. Perform a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) at a frequency of at least every 20 samples for each water matrix run in accordance with method 7470A..

12.6.2 Prepare 2.0 ug/L matrix spikes (MS/MSD) as follows:

12.6.2.1 Measure 30 mL of the sample being spiked into each of two block digestion vessels.

12.6.2.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into each vessel.

12.6.2.3 Prepare these spiked sample aliquots in the same way the un-spiked sample is prepared.

12.6.3 Prepare a 0.5 ug/L blank spike (BS) as follows:

12.6.3.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.

12.6.3.2 Pipette 15 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.

12.6.3.3 Prepare in the same way as a sample, bringing the final volume to 50 mL.

12.6.4 Prepare a 2.0 ug/L blank spike (BS) as follows:

12.6.4.1 Measure 30 mL of metals laboratory reagent water into a block digestion vessel.

12.6.4.2 Pipette 60 µL of the 1000 ug/L spiking standard (Section 12.4.1) into the vessel.

12.6.4.3 Prepare in the same way as a sample bringing the final volume to 50 mL.

13.0 ANALYTICAL PROCEDURE

13.1 All samples and quality control must be digested before analysis. All digestates must be evaporated to below 50 mL within the digestion time and brought back to a final volume of 50 mL with metal laboratory reagent water at the end of the digestion.

13.2 Pre-heat the block digestor to 90-95° C.

13.3 Measure 30 mL of each well-mixed sample into a digestion vessel. Prepare the associated quality control samples and calibration standards.

13.4 To each digestion vessel add the following reagents:

13.4.1 Pipette in 0.75 mL of concentrated HNO_3

13.4.2 Pipette in 1.5 mL concentrated H_2SO_4

13.4.3 Pipette in 4.5 mL of $KMnO_4$

13.5 After adding, swirl to mix then let the samples sit for 15 minutes.

13.5.1 If a sample does not maintain the purple or brown permanganate color for at least 15 minutes, add up to an additional 3.0 mL of $KMnO_4$ solution but no more.

Note: Separate calibration standards and quality control must be prepared with the same permanganate volume to match the sample.

13.5.2 If a sample does not maintain the purple or brown permanganate color after the maximum $KMnO_4$ addition of 7.5 mL, discard the preparation, dilute the sample appropriately and prepare 30 mL of the dilution for digestion.

13.6 Pipette 2.40 mL of 5% $K_2S_2O_8$ into each digestion.

13.7 Place in the block digestor and position a cap on each digestion vessel without tightening then reflux for two hours at 90-95° C. Do not exceed 95° C.

13.8 After digestion, remove and cool to room temperature then pipette in 1.8 mL of 12% $NaCl/NH_2OH{\cdot}HCl$.

13.9 Finally, bring to a final volume of 50 mL with metals laboratory reagent water. Cap and mix thoroughly.

13.10 After a delay of at least 30 seconds, samples are ready for the stannous chloride addition and immediate analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch benchsheet report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used. (Attachment 20.1)

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data. (Attachment 20.3)

14.5 If internal chain-of-custody (COC) is required, it is very important that COC forms be filled in completely and correctly. Organize, transfer and/or archive COC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digestor maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

15.0 QUALITY ASSURANCE

15.1 Prepare and digest a method blank (BLK) for each batch of up to 20 samples digested, to monitor contamination. The BLK is 30 mL of metals laboratory reagent water measured into a block digestion vessel. The BLK vessel is then processed as a sample following every step in the procedure. Refer to TriMatrix SOP GR-01-123 for BLK acceptance criteria.

15.2 Prepare and digest the two blank spikes (BS) of Section 12.6.3 and 12.6.4 for each batch of up to 20 samples digested. Refer to TriMatrix SOP GR-01-123 for BS acceptance criteria.

15.3 Prepare and digest a matrix spike (MS)/matrix spike duplicate (MSD) as described in Section 12.6.2 and at the frequency described in Section 12.6.1, or prepare for each digestion session/matrix. Matrix spikes monitor matrix recovery and precision. Refer to TriMatrix SOP GR-01-123 for MS/MSD acceptance criteria.

15.4 Do not allow samples to boil during digestion. If a sample begins to boil, immediately remove it from the block digestor and turn the temperature down.

15.5 Do not use expired spiking solutions and/or standards. If a standard has expired, obtain or prepare another.

15.6 The method of standard additions must be performed on all leachates with 50% or less spike recovery. Refer to TriMatrix SOP GR-01-123 for preparing and digesting a standard addition series.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) study is also required annually.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare four blank spikes (BS) at a concentration of 2.0 ug/L in laboratory reagent water. ONLY the last four results may be used. Otherwise, digest and evaluate the four blank spikes following every step in the digestion and analysis procedures. Calculate average percent recovery and relative standard deviation by inputting the four result values to the IDC spreadsheet on the laboratory intranet library. Average percent recovery must fall within 85 – 115% recovery. Relative standard deviation must be ≤20%.

19.1.1.2 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the procedure or techniques used then repeat the study.

19.1.1.3 Samples may not be analyzed by any analyst until a demonstration of capability study has been successfully completed. Copies of successful study spreadsheets must be given to the quality assurance department.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability must be repeated annually by all analysts running samples.

19.1.2.2 The CDC may be accomplished by repeating the IDC study, by submitting four consecutive blanks spike values obtained during the course of routine sample

digestion, by submitting the last four MDL study values or by submitting an acceptable performance testing result. In each case, the work must be done exclusively by one analyst and submitted to quality control for documentation.

19.2 A Method Detection Limit (MDL) Study must be completed annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) on the laboratory intranet library when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 1, September 1994, Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)"

19.2 *Methods for the Determination of Metals in Environmental Samples,* Supplement I, May 1994, Revision 5.4, EMMC Version, "Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry", Method 245.1, Revision 3.0, May, 1994

20.0 ATTACHMENTS

20.1 Mercury Stock Standard Certificate of Analysis Example

20.2 Element™ Work Order Report Example

20.3 Element™ Preparation Batch Report Example

20.4 Stock Standards Log Example

20.5 Digestion Flowchart

Attachment 20.1
Mercury Stock Standard Certificate of Analysis Example

inorganic ventures / iv labs

195 lehigh avenue, suite 4, lakewood, nj 08701 usa
phone: 800-669-6799 • 732-901-1900 • fax: 732-901-1903
e-mail: ivsales@ivstandards.com • website: www.ivstandards.com

certificate of analysis

1.0 **Inorganic Ventures / IV Labs is an ISO Guide 34-2000 Certified Reference Material (CRM) Manufacturer: Certificate #883-02.** The certificate is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31-2000 (Reference Materials - Contents of certificates and label(s), ISO Guide 34-2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statisical Principles."

2.0 **DESCRIPTION OF CRM** Custom-Grade **1000 µg/mL Mercury in 3.5% (abs) HNO_3**

Catalog Number:	CGHG1-1, CGHG1-2, and CGHG1-5
Lot Number:	**Y-HG02038**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999555
Starting Material Lot No	05214TX
Matrix:	3.5% (abs) HNO_3

Me 14.98.8
A503277
OR
9-27-05

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 1007 ± 3 µg/mL

Certified Density: 1.026 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = 2[\frac{\Sigma s_i}{(n)^{1/2}}]^{1/2}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors.
(Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

. "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

. This IV product is Traceable to NIST via direct comparison to NIST SRMs. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors.

4.1 **Assay Method #1** **998 ± 4 µg/mL**
EDTA NIST SRM 928 Lot Number: 392110

Assay Method #2 **1007 ± 3 µg/mL**
ICP Assay NIST SRM 3133 Lot Number: 991304

Attachment 20.2
Element™ Work Order Report Example

TriMatrix Laboratories, Inc. **WORK ORDER 0610156** Printed: 10/12/2006 2:46:21PM

Page 1 of 2

Metals Sample Receipt Notice

Client: [redacted]
Project: [redacted]
Client Due Date: **Oct-20-06 16:00 (10 day TAT)**
W.O. Comments: *2RL*

Project Manager: **Gary L. Wood**
Project Number: **[none]**
Report Level: **2RL**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
[redacted]						
	Ag Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	As Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Ba Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cd Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cr Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Cu Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Hg Total 7470A		10	Nov-03-06 14:00	Oct-19-06 17:00	
	Ni Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Pb Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Se Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	
	Zn Total 6020		10	Apr-04-07 14:00	Oct-19-06 17:00	

Attachment 20.3
Element™ Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0901183 Page 1 of 1

Printed: 1/22/2009 1:38:35PM

Metals, Water, 7470A Digestion - Total

(No Surrogate)

Batch Comments: MAS 01/21/09 1:0.0-3:00PM

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0901199	Hg Total 7470A				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client/QC Type	Extraction Comments
0901183-BLK1		Jan-21-09 13:00	MAS	30	30					BLANK	
0901183-BS1		Jan-21-09 13:00	MAS	30	30			8100309	60	LCS	
0901183-MS1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE	
0901183-MSD1		Jan-21-09 13:00	MAS	30	30		0901199-12	8100309	60	MATRIX SPIKE DUP	
0901199-10	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-11	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-12	E	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-13	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-14	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-15	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-16	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr
0901199-17	C	Jan-21-09 13:00	MAS	30	30					[redacted]	wtr

Approved By: PD 1-22-09 — **QA Officer**

Approved By: MAS 01-23-09 — **Area Supervisor**

Attachment 20.4
Stock Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8060501

Description:	Mercury 1000 mg/L SCV Stock	Expires:	Feb-28-10
Standard Type:	Other	Prepared:	Jun-17-08
Solvent:	10% HNO3	Prepared By:	** Vendor **
Final Volume (mls):	100	Department:	Metals
Vials:	1	Last Edit:	Jun-17-08 12:56 by RGJ

SCP part# 140-051-801
Lot#SC8122815
Received 06/17/08

Analyte	CAS Number	Concentration	Units
Mercury, OWEP-TAR	7439-97-6	1000	mg/L
Mercury, OWEP-SAR	7439-97-6	1000	mg/L
Mercury	7439-97-6	1000	mg/L

Approved By: ___ 1-22-09 ___ **QA Officer** **Approved By:** MAS 01-23-09 **Area Supervisor**

Attachment 20.5
Digestion Flowchart

Pre-heat the block digestor to 90-95° C
↓
Measure 30 mL sample directly into the block digestion vessel and prepare quality control samples
↓
Pipette in 0.75 mL of concentrated HNO_3
↓
Pipette in 1.50 mL concentrated H_2SO_4
↓
Pipette in 4.50 mL of $KMnO_4$ ← ← ← ← ← ← ← ← ← ← ← ← ← ← ←
↓ ↑
Let sit for 15 minutes ↑
↓ ↑
Does sample maintain the purple or brown permanganate color for at least 15 minutes? → no → Pipette in up to 3.0 mL more of $KMnO_4$ ↑
↓ ↓ ↑
yes Let sit for 15 minutes ↑
↓ ↓ ↑
Pipette in 2.40 mL of 5% $K_2S_2O_8$ ← yes ← Does sample maintain the purple or brown permanganate color for at least 15 minutes? → no → Dilute sample appropriately
↓
Digest for 2 hours at 90 - 95° C
↓
Cool to room temperature
↓
Pipette in 1.80 mL of 12% NaCl/NH_2OH•HCl, bring to 50 mL volume with metals laboratory reagent water then cap and mix thoroughly
↓
After a delay of at least 30 seconds, samples are ready for the stannous chloride addition and immediate analysis.

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Block Digestion of Total Metals in Water for ICP

EPA Method 200.7
SW-846 Method 3010A

APPROVALS:

Area Supervisor: Marge A Scott — Date: 01/14/09
Marge A. Scott

QA Officer: [signature] — Date: 1-8-09
Tom C. Boocher

Operations Manager: Jeff Glaser — Date: 1/15/09
Jeff P. Glaser

Procedure Number: GR-01-147
Revision Number: 0.4

Date Initiated: 7/15/03
Effective Date: 1/20/09

Date Revised: 1/7/09
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 16

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Water, wastewater and leachate samples are prepared for total metals analysis by block digestion.

1.2 Silica-based matrices are not digested using this procedure.

1.3 Digestates are prepared for analysis by inductively coupled plasma spectrometry (ICP).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 1, July 1992, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"

2.1.1 This procedure describes the use of a 50 mL non-constricted block digestion cup as a deviation from method 3010A, which specifies the use of a Griffin beaker.

2.1.2 This procedure describes the use of 25 mL as a deviation from method 3010A, which specifies a 100 mL sample aliquot for digestion.

2.1.3 This procedure describes the use of 5 mL of nitric acid for the first reflux as a deviation from method 3010A which specifies 3 mL. Acid ratios are maintained and 1.25 mL is used for 25 mL instead of 5 mL for 100 mL.

2.2 *Code of Federal Regulations*, Title 40, Appendix C to Part 136, February 1, 2002, Method 200.7, "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes"

2.2.1 This procedure describes the use of a 50 mL block non-constricted digestion cup as a deviation from method 200.7, which specifies the use of a Griffin beaker.

3.0 SUMMARY OF PROCEDURE

3.1 Samples are prepared as follows:

3.1.1 A 25.0 mL aliquot of well-mixed, homogeneous aqueous sample is accurately measured into a digestion cup.

3.1.2 Analytes are solubilized by evaporating the sample to low volume after adding nitric acid while gently refluxing. Additional portions of nitric acid are added with further refluxing until the digestate is light in color or has color-stabilized.

3.1.3 The digestate is then gently refluxed with 1:1 (v/v) hydrochloric acid to dissolve any precipitate.

3.1.4 After digestion is complete and cool, the volume is adjusted with laboratory reagent water to 25.0 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 This procedure includes digestion for the following elements:

Aluminum	Chromium	Selenium	Calcium	Potassium
Antimony	Cobalt	Silver	Iron	Silicon
Arsenic	Copper	Strontium	Lithium	Sodium
Barium	Lead	Thallium	Magnesium	Sulfur
Beryllium	Manganese	Tin	Phosphorus	Titanium
Boron	Molybdenum	Vanadium		
Cadmium	Nickel	Zinc		

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-01-100, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.3 TriMatrix SOP GR-01-145, *Inductively Coupled Plasma Atomic Emission Spectrometry using Perkin Elmer Optima 3000/3300DV*, latest revision

5.4 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Refer to TriMatrix SOP GR-01-145 for a list of interferences and associated corrective actions when digesting for ICP analysis.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Concentrated acids are used in standard and sample preparations. Wear disposable gloves, laboratory coats and approved safety glasses at all times when digesting samples and/or working in the laboratory.

7.3 Refer to the Material Safety Data Sheet (MSDS) library for specific information on any chemical used in the laboratory.

7.4 Check fume hoods to be sure each is functioning properly. All digestions must be done in a fume hood. If the hood is not working properly, immediately contact the area supervisor. Under no circumstances is any block digestion to be performed if the exhaust hood is not function properly.

7.5 Many chemicals and elements used in this procedure are toxic. Minimize exposure by appropriate use of the personal protective equipment provided.

7.6 No food or drink is allowed in the digestion laboratory at any time, including gum.

7.7 Wash hands before starting work. Substances on the skin may interfere with metals analysis. Wash hands after finishing work. Chemical residuals can be injested and/or carried out of the laboratory if not removed.

7.8 Before leaving the digestion laboratory, remove disposable gloves and laboratory coat. Chemical and/or acid residue transport outside the digestions laboratory needs to be kept to a minimum.

7.9 Rinse all micropipette tips and beakers before disposal or cleaning.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND MATERIALS

8.1 Samples collected for digestion and analysis by ICP can be preserved in the field or upon receipt by the laboratory. If the pH is not <2 when received by the laboratory, lower to <2 during the sample receipt process using 1:1 nitric acid/water. The maximum volumes of acid added to a sample (including initial volume) are:

Container Size	Acid Volume (mL)
125	1.2
250	2.6
500	5.0
1000	10.0

8.2 If a pH adjustment is necessary, a minimum of 24 hours must elapse before the sample digestion process may begin.

NOTE: Sample preservation must occur in the original container to prevent the loss of any analytes adsorbed to the container wall. It is not permissible to remove and acidify a sample aliquot.

NOTE: If the sample must be analyzed within 24 hours of collection, the sample must be preserved immediately during collection.

NOTE: If boron is an analyte of interest the sample must be preserved immediately during collection.

NOTE: Samples collected for pre-treatment and analysis by method 6010 must be preserved immediately during collection.

8.3 Consult the project chemist for instruction if the pH cannot be adjusted to <2 using the specified maximum amount of acid. Samples prepared and analyzed without proper pH preservation, results must be narrated as such.

8.4 Samples may be collected in glass or plastic. If Boron is to be analyzed, use only plastic containers. Do not allow acidified sample to come into contact with metal to prevent contaminants dissolving into the sample.

8.5 To ensure sufficient sample volume for quality control and/or redigestion, collect at least 150 mL of unfiltered liquid. Smaller volumes may result in elevated reporting limits.

8.6 For some determinations of dissolved elements, the filtered and preserved sample may require digestion. Filtered samples that form a precipitate at preservation, during transit to the laboratory or in storage must be digested before the analysis.

8.7 Properly acid-preserved samples can be held for up to 180 days prior to digestion. Refrigeration is not required for samples or digestates.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Micropipette, capable of delivering 125 µL

9.2 Block digestor for metals digestion, Environmental Express, HotBlock™, model SC154

9.3 Block digestion cups, certified pre-cleaned, catalog SC475, available from Environmental Express

9.4 Ribbed watchglasses, disposable, Environmental Express, catalog SC505

9.5 Filtermate, Environmental Express, catalog SC0501, for digestate filtration

9.6 Non-ribbed watchglasses, disposable, Environmental Express, SCP Science, catalog 010-500-081

9.7 Thermometers, NIST-traceable, digital, capable of reading to at least 100° C in 0.1° C increments.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Verify and document the micropipette calibration daily.

10.2 Clean each block digestor and all counters daily. Clean up spills immediately.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water (ASTM Type II), milliQ water system

11.2 Nitric acid (HNO_3), concentrated, trace metal grade

11.3 Hydrochloric acid (HCl), concentrated, trace metal grade

11.3.1 Prepare a 1:1 (v/v) solution of HCl in water by measuring 50 mL of concentrated HCl into 50 mL of laboratory reagent water. Remember to perform the addition in a fume hood.

11.3.2 Expiration is six months from the date made.

12.0 STANDARDS PREPARATION

12.1 Multi-element ICP spiking solutions are purchased as commercially prepared solutions. Refer to Attachments 20.1 and 20.2 for ICP spiking solution examples.

13.0 ANALYTICAL PROCEDURE

13.1 Fill one block digestion cup with laboratory reagent water and place in a random hole of the block digestor. Cover with a watchglass that has a hole in the center and insert the digital thermometer through the hole. Using the thermometer and not the block digestor read-out, adjust the block temperature to 90 - 95° C.

13.2 Mix samples by vigorously shaking to achieve homogeneity. Immediately after shaking, measure 25.0 mL (or an appropriately smaller volume) into a block digestion cup. For samples requiring matrix spikes, shake vigorously before each 25.0 mL aliquot is removed.

13.3 Measure 0.75 mL of concentrated HNO_3 into each sample cup then cover with a ribbed watchglass.

13.4 After the block equilibrates to 90 - 95° C, place all samples in the block and cover with a ribbed watchglass. Evaporate to near dryness, which is approximately 1.25 mL. If necessary, measure 1.25 mL into an empty digestion cup for comparison. If any digestion goes to dryness, discard and re-prepare with another sample aliquot.

13.5 After evaporating to near dryness, cool by removing briefly from the block then add 1.25 mL of concentrated HNO_3. Cover with a non-ribbed watchglass then return to the block and gently reflux. Slight boiling should occur while refluxing but vigorous boiling must be avoided. Continue refluxing. If the digestate is not light in color after refluxing, cool and add another 0.75 mL HNO_3 then continue refluxing as before. Repeat 0.75 mL HNO_3 additions and refluxing until the digestate is light in color or does not change with continued refluxing.

13.6 When the digestion is complete, change to a ribbed watchglass and evaporate to approximately 0.75 mL. Be careful not to let any digestion go to dryness, else it will need discarded and re-prepared.

13.7 After evaporation to 0.75 mL, remove from the block to cool then measure 2.5 mL of 1:1 HCl into each digestate. Return to the block and gently reflux for an additional 15 minutes.

13.8 After refluxing with HCl, remove from the block and cool to room temperature.

13.9 When cool, dilute to 25.0 mL with laboratory reagent water then cap and shake vigorously. At this point, digestates are ready for analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Work orders are printed out by the Laboratory Information Management System (Element™) indicating which samples need digestion.

14.1.1 Initial and date any hand-written information on the work order report.

14.1.2 Organize and archive all work order forms in accordance with the TriMatrix quality assurance manual.

14.2 Completion of each sample with all quality control is entered back into Element™ and a preparation batch report is printed.

14.2.1 Enter all information associated with the digestion before printing.

14.2.2 The preparation batch report accompanies the sample digestion batch to the instrumental laboratory.

14.2.3 Initial and date any hand-written information on the preparation batch report.

14.2.4 Organize and archive all preparation batch report forms in accordance with the TriMatrix quality assurance manual.

14.3 Maintain the use of all standards and reagents in Element™ for traceability. Certificates of analysis are maintained on the laboratory intranet library for each standard and reagent used.

14.4 Analysts preparing digestion batches are responsible for correctly filling in, transferring and filing all associated paperwork. This is essential in providing the client with defensible data.

14.5 If internal chain-of-custody (COC) is required, it is very important that COC forms be filled in completely and correctly. Organize, transfer and/or archive COC forms in accordance with the TrMatrix quality control manual.

14.6 Fill in block digestor maintenance logbooks completely and correctly when maintenance is performed. Indicate blank areas in logbooks with a large "Z" that is dated and initialed.

14.7 Make corrections in any logbook or on any hardcopy with a single line over the mistake (not a writeover or scribble) then date and initial any new writing.

15.0 QUALITY ASSURANCE

15.1 At least one digestion blank (BLK) must be carried through the entire digestion to monitor contamination with each digestion batch of up to 20 samples. The blank is prepared by measuring 25.0 mL of laboratory reagent water or appropriate extraction fluid into a digestion cup and digesting as a sample.

15.2 At least one blank spike (BS) must be carried through the entire digestion to document recovery without matrix interference in each digestion batch of up to 20 samples. A blank spike is prepared by measuring 25.0 mL of laboratory reagent water or appropriate extraction fluid into a digestion cup, then spiking with 125 µL of solution SSW-1A and 125 µL of solution SSW/SSS-2A.

15.3 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for water samples, for each digestion batch of up to 20 samples.

15.4 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for leachates, for each digestion batch of up to 20 samples.

15.5 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for wastewater samples, for each digestion batch of up to 10 samples.

15.6 Prepare matrix spike and matrix spike duplicates by measuring a 25.0 mL sample aliquot into a digestion cup, then spiking with 125 µL of solution SSW-1A and 125 µL of solution SSW/SSS-2A.

15.7 Digestates must not boil during the evaporation step. If a digestate begins to boil during evaporation, immediately remove from the block and reduce the temperature. Some boiling will be evident during the reflux step. However, vigorous boiling during reflux will diminish the water-acid azeotrope and cause loss of analyte. If a digestate begin to boils vigorously during refluxing, immediately remove from the block and reduce the temperature. If boiling during evaporation or vigorous boiling during refluxing is not immediately addressed, discard and the re-prepare the sample and/or samples affected. Monitor the block temperature closely to minimize re-preparation.

15.8 Each commercially purchased spiking solution must be used within its expiration date. If a standard or solution has expired, remove it from the laboratory for proper disposal.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). Analysts responsible for sample processing by this procedure must pass an IDC study first.

16.1.1 Initial Demonstration of Capability Study

16.1.1.1 Spike four 25.0 mL aliquots of laboratory reagent water as in a blank spike preparation. Digest the four spikes following every step in this procedure and obtain analysis results for the four blank spikes. Analysis of the spikes must also include every step in the analytical procedure. Enter all four results obtained into the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation. Average recovery must fall

within the blank spike control limits found in Element™. Relative standard deviation must be less than or equal to 20%.

16.1.1.2 Alternatively, the last four of seven results used in an MDL study may be used as the IDC, if the MDL study was run exclusively by the analyst. ONLY the last four results may be used.

16.1.1.3 If either criterion is not met, locate and correct the source of any problem and repeat the study to confirm the corrective action was successful. Repeated failure however, will confirm a general problem with the procedure or techniques used. If multiple failures occur, correct the procedure or techniques used, and repeat the study.

16.1.1.4 Samples may not be processed by any analyst until an IDC study has been successfully completed. Give copies of successful IDC study spreadsheets and raw data to the quality assurance department for training documentation.

16.1.2 Continuing Demonstration of Capability Study

16.1.2.1 Annually, a Continuing Demonstration of Capability (CDC) study is required by each analyst.

16.1.2.2 A CDC may be accomplished by repeating the IDC study, by using the last four results from an MDL study, or by using four consecutive and exclusively run blank spike results obtained from routine sample analysis since the IDC study was completed. Alternatively, successful completion of an exclusively digested performance testing study result run since completing the IDC may be used.

16.1.2.3 Process CDC data using the same demonstration of capability spreadsheet and in the same manner as the IDC.

16.2 Method Detection Limit Studies

16.2.1 A Method Detection Limit (MDL) study must be performed annually for this procedure. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence the value is above zero.

16.2.2 The procedure used for an MDL study is based on the method given in the Code of Federal Regulations, Title 40, Part 136, Appendix B, latest revision.

16.2.3 Seven replicate digestions are performed using laboratory reagent water spiked with the elements(s) of interest at the estimated minimum reportable concentration.

16.2.4 The standard deviation of the average for each element, using all seven results is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

16.2.5 If the concentration spiked is between the calculated MDL and ≤10 times the calculated MDL, and there are no zero percent recoveries in the seven results then the MDL passes.

16.2.6 If the MDL study does not pass acceptably, it must be re-digested. If a study needs re-digested at a different concentration, the entire set of seven needs re-digested. If a study does not pass due to poor reproducibility on one result, only that one result needs re-digested. However, only one result of the seven can be re-digested without re-digesting the entire study.

16.2.7 All seven MDL study results do not need digested in the same digestion batch.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranent library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, Revision 1, July 1992, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy"

19.2 *Code of Federal Regulations*, Title 40, Appendix C to Part 136, February 1, 2002, Method 200.7, "Inductively Coupled Plasma-Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes"

20.0 ATTACHMENTS

20.1 ICP Spiking Solution SSW-1A Certificate of Analysis Example

20.2 ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

20.3 Work Order Report Example

20.4 Preparation Batch Report Example

20.5 Digestion Flowchart

Attachment 20.1
ICP Spiking Solution SSW-1A Certificate of Analysis Example

INORGANIC VENTURES CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com

tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-1A

Lot Number: **Y-MEB188079**

Matrix: 2% HNO3(abs)

A607108
DA
7-06-06

2,500.00 µg/mL each:
Ca, K, Mg, Na,
250.00 µg/mL each:
Al, As, P, Se, Tl,
50.00 µg/mL each:
Ag, B, Ba, Cd, Co, Cr3, Cu, Fe, Li, Mn, Ni, Pb, Sr, V, Zn,
5.00 µg/mL each:
Be

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Aluminum, Al	249.8 ± 0.7 µg/mL	Arsenic, As	250.2 ± 0.8 µg/mL	Barium, Ba	50.16 ± 0.08 µg/mL
Beryllium, Be	5.004 ± 0.010 µg/mL	Boron, B	50.07 ± 0.10 µg/mL	Cadmium, Cd	50.16 ± 0.22 µg/mL
Calcium, Ca	2,500 ± 6 µg/mL	Chromium+3, Cr3	50.11 ± 0.16 µg/mL	Cobalt, Co	50.09 ± 0.12 µg/mL
Copper, Cu	50.17 ± 0.14 µg/mL	Iron, Fe	49.84 ± 0.32 µg/mL	Lead, Pb	50.17 ± 0.11 µg/mL
Lithium, Li	49.91 ± 0.06 µg/mL	Magnesium, Mg	2,501 ± 5 µg/mL	Manganese, Mn	49.97 ± 0.12 µg/mL
Nickel, Ni	50.10 ± 0.12 µg/mL	Phosphorus, P	249.5 ± 0.6 µg/mL	Potassium, K	2,500 ± 5 µg/mL
Selenium, Se	249.8 ± 0.8 µg/mL	Silver, Ag	50.01 ± 0.11 µg/mL	Sodium, Na	2,497 ± 1 µg/mL
Strontium, Sr	50.05 ± 0.11 µg/mL	Thallium, Tl	249.9 ± 0.7 µg/mL	Vanadium, V	49.98 ± 0.14 µg/mL
Zinc, Zn	50.09 ± 0.12 µg/mL				

Certified Density: 1.049 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

Certified Value $(\bar{x}) = \frac{\Sigma x_i}{n}$

Uncertainty $(\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

Attachment 20.2
ICP Spiking Solution SSW-SSS-2A Certificate of Analysis Example

INORGANIC VENTURES™ CERTIFICATE OF ANALYSIS

195 Lehigh Avenue, Suite 4
Lakewood, New Jersey 08701 · USA
inorganicventures.com
tel: 800.669.6799 · 732.901.1900
fax: 732.901.1903
info@inorganicventures.com

1.0 **INORGANIC VENTURES** is an ISO Guide 34:2000 registered Certified Reference Material (CRM) Manufacturer (Certificate #883-02). The certificate is designed and the data is determined in accordance with ISO Guide 31:2000 (Reference Materials-Contents of Certificates and Labels), ISO Guide 34:2000 "Quality System Guidelines for the Production of Reference Materials," and ISO Guide 35-1989 "Certification of Reference Materials - General and Statistical Principals."

2.0 **DESCRIPTION OF CRM** Custom Solution

Catalog No.: TRIMATRIX-SSW-SSS-2A

Lot Number: **Y-MEB201134**

Matrix: tr. HF, 2% HNO3(abs)

A6092280
DSC 9-11-06
exp. 10-01-07

250.00 µg/mL each:
Sb, Si, Sn,
50.00 µg/mL each:
Mo, Ti

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
Antimony, Sb	249.1 ± 0.4 µg/mL	Molybdenum, Mo	50.02 ± 0.10 µg/mL	Silicon, Si	249.7 ± 0.8 µg/mL
Tin, Sn	250.6 ± 0.9 µg/mL	Titanium, Ti	50.14 ± 0.08 µg/mL		

Certified Density: 1.015 g/mL (measured at 22° C)

The Certified Value is based upon the most precise method used to analyze this CRM. The following equations are used in the calculation of the certified value and the uncertainty:

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

($\bar{x}$) = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEBILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

· "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
· This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

Attachment 20.3
Work Order Report Example

TriMatrix Laboratories, Inc. **WORK ORDER 0609285** Printed: 9/20/2006 2:30:22PM

Page 1 of 2

Metals Sample Receipt Notice

Client: [redacted]
Project: **Analytical Services**
Client Due Date: **Oct-04-06 16:00 (10 day TAT)**
W.O. Comments: *1RL*

Project Manager: **Lisa M. Harvey**
Project Number: **32207**
Report Level: **1RL**

Lab Number	Sample Name / Analysis	Matrix	Sampled Date / TAT	Expire Date	Sample Comments / Lab Due Date	Comments
[redacted]						
	Ag Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cd Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cr Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Cu Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Ni Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Pb Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	
	Zn Total 200.7		10	Mar-18-07 00:00	Oct-03-06 17:00	

Approved By: MB 1-8-09 **QA Officer**
Approved By: MAS 01/14/09 **Area Supervisor**

Attachment 20.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0610868 Page 1 of 2 Printed: 9/22/2006 9:36:45AM

Metals, Waste Water, 3010A Digestion

Batch comments:
09/22/06 7:00am-3:30pm
Inst 219 #179 @ 95C
Hotblock Vessel Lot #A511LS106

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Client Spike ID	Client uL Spike	Comments		
0609168-01	A	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					
0609168-03	A	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					
0609285-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					
0609295-01	D	Sep-22-06 07:00	MAS	25	25	[redacted]					
Na Total 200.7											
0609300-04	A	Sep-22-06 07:00	MAS	25	25	[redacted]			report at 10 ug/L		report at 10 ug/L
Cu Total 200.7											
0609303-01	B	Sep-22-06 07:00	MAS	25	25	[redacted]			Added for BatchQC in: 0610868		BatchQC
Ag Total 200.7	Al Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Mo Total 200.7	Na Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7		
0609304-01	B	Sep-22-06 07:00	MAS	25	25	[redacted]			wkly		wkly
Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Zn Total 200.7								
0609305-01	A	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Al Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7				
0609316-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					
0609327-02	F	Sep-22-06 07:00	MAS	25	25	[redacted]					
Mo Total 200.7											
0609327-04	F	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					
0609337-01	C	Sep-22-06 07:00	MAS	25	25	[redacted]					
Ag Total 200.7	Cd Total 200.7	Cr Total 200.7	Cu Total 200.7	Ni Total 200.7	Pb Total 200.7	Zn Total 200.7					

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: [illegible] 1-8-09 **QA Officer** **Approved By:** MAS 01/14/09 **Area Supervisor**

Attachment 20.5
Digestion Flowchart

Measure 25.0 mL of homogeneous sample into a digestion vessel
↓
add 0.75 mL of concentrated nitric acid
↓
cover with a ribbed watchglass
↓
evaporate to about 1.25 mL at 90-95 deg. C (do not boil)
↓
remove from the block digestor to cool then add 1.25 mL concentrated nitric acid
↓
change to a non-ribbed watchglass and return to the block digestor
↓
reflux (slight boiling will be observed but vigorous boiling must be avoided)
↓
if the digestate is not light in color after refluxing, cool and add another 0.75 mL nitric acidthen continue refluxing as before
↓
repeat 0.75 mL nitric acid additions and refluxing until the digestion is complete
↓
remove from the block digestor and change to a ribbed watchglass
↓
evaporate to about 0.75 mL at 90-95 deg. C (do not boil)
↓
remove from the block digestor to cool then add 2.5 mL 1:1 hydrochloric acid
↓
change to a non-ribbed watchglass and return to the block digestor
↓
reflux gently for 15 minutes
↓
remove from the block digestor and cool to room temperature
↓
dilute to 25.0 mL with laboratory reagent water then cap and shake vigorously
↓
digestates are ready for analysis

Approved By: 1-8-09
QA Officer

Approved By: 01/14/09
Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Polychlorinated Biphenyls (PCB) by Gas Chromatography

EPA Method 608
SW-846 Method 8082

APPROVALS:

Area Supervisor: Daniel J. Mierendorf Date: 8/21/09
Daniel J. Mierendorf

QA Officer: Date: 8-21-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 8/21/09
Jeff P. Glaser

Procedure Number: GR-03-128
Revision Number: 2.5

Date Initiated: 4/2/98
Effective Date: 9/5/09

Date Revised: 8/21/09
Pages Revised: All

By: Daniel J. Mierendorf
Total Number of Pages: 43

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure is used to quantitate polychlorinated biphenyls (PCBs) as extracted Aroclors from water, wastewater, soil, wipes, oil, and waste samples. Aroclors are multi-component analytes. When samples contain more than one Aroclor, a higher level of analyst expertise is required to attain acceptable qualitative and quantitative analysis. The same is true of Aroclors subjected to environmental degradation (weathering), or degradation by treatment technologies. Such degraded mixtures may have significantly different peak patterns compared to Aroclor standards.

1.2 A second analytical technique (different column or GC/MS) must be utilized to confirm positive results.

1.3 Minimum reporting limits (RL) are as follows:

1.3.1 0.100 ug/L for water and wastewater samples

1.3.2 0.017 mg/kg for soil samples

1.3.3 0.500 µg for wipe samples

1.3.4 0.500 mg/kg for diluted waste samples

Reporting limits are highly matrix dependent and the minimum reporting limit might not be achievable with all matrices. Refer to Attachment 21.1 for a default reporting limit list by analyte.

1.4 The linear calibration range begins at the reporting limit and increases to approximately 32 times the reporting limit. The range of analysis may be extended upward by diluting extracts into the linear calibration range.

1.5 Analysis of PCB analytes is restricted for use by or under the supervision of analysts experienced in gas chromatography and chromatographic interpretation.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 8082, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Revision 0, December, 1996

2.2 *Code of Federal Regulations*, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, Method 608, Organochlorine Pesticides and PCBs

3.0 SUMMARY OF PROCEDURE

3.1 For the analysis, extracts are injected into a gas chromatograph (GC) with two simultaneously loaded columns. The volume injected is 1 or 2 µL.

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3.2 After separation by the column, analyte response is obtained with an electron capture detector (ECD).

3.3 Sample concentration is determined by comparing sample response to a previously run and verified calibration obtained from certified standards of known concentration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Aroclors available for analysis are listed below. Other Aroclors may be analyzed provided an acceptable demonstration of capability is performed which includes all steps of the extraction, cleanup and analysis.

Compound	CAS Registry No.
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
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-04-101, *Semi-Volatile Organic Laboratory Corrective Actions*, latest revision

5.2 TriMatrix Laboratories SOP GR-09-105, *Waste Dilution Preparation Method for PCBs and Pesticides*, latest revision

5.3 TriMatrix Laboratories SOP GR-09-107, *Extraction of Organochlorine Pesticides and PCBs in Water*, latest revision

5.4 TriMatrix Laboratories SOP GR-09-108, *Extraction of PCBs/Pesticides in Soil, Sludge, and Wastes*, latest revision

5.5 TriMatrix Laboratories SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.6 TriMatrix Laboratories SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.7 TriMatrix Laboratories SOP GR-09-111, *Florisil Column Cleanup*, latest revision

5.8 TriMatrix Laboratories SOP GR-09-120, *Florisil/Silica Gel cleanup of PCB's/Toxaphene/Chlordane*, latest revision

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5.9 TriMatrix Laboratories SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.10 TriMatrix Laboratories SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Samples often have components that interfere with electron capture detection and/or capillary column chromatography. To minimize interference, extracts are cleaned up before analysis.

6.2 Solvents can introduce oxygenates and sulfur compounds when concentrated after sample extraction giving an undesirable ECD response. Such interferences often coelute with PCB congeners. All extraction solvents must be demonstrated interference-free by the analysis of blanks. Use only pesticide grade or better solvents. Each solvent lot from a vendor must be tested for acceptance prior to use. Once the lot is documented to be contaminant-free by the analysis of at least one extraction blank that indicates no peaks, a large quantity of the test lot is sequestered from the vendor to ensure consistent and verified purity.

6.3 Other sources of contamination are extraction equipment. Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use. Use only high reagent grade or better reagents.

6.3 Carryover can occur whenever highly concentrated samples precede the analysis of low or non-detect samples. To reduce carryover, autosampler syringes are rinsed between each injection. When necessary, analyze a solvent blank immediately after a high level sample. If carryover is suspected and/or observed in the solvent blank, all affected samples must be re-analyzed.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 PCBs can cause irritation upon contact and are carcinogenic. Open, prepare and dispense standards and solutions only in a fume hood. Wear a laboratory coat, disposable gloves and safety glasses when working in the laboratory.

7.3 Extraction solvents are toxic. Avoid contact with the skin, avoid inhalation of solvent vapors and do not ingest (no chewing gum in the laboratory). Open solvent containers under a fume hood.

7.4 All analyzed sample extracts and expired standards must be disposed of properly. Consolidate all samples and extracts testing positive for any concentration of PCBs as PCB-containing waste.

7.5 If a PCB or solvent spill occurs, notify the area supervisor and safety officer. Evacuate the area and begin cleaning up the spill as outlined in the safety manual.

7.6 Every six months, an ECD wipe test must be performed on each detector as dictated by federal law. The test is used to determine whether an ECD is leaking radioactive material. Three areas must be checked as follows:

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7.6.1 The detector housing

7.6.2 The detector entrance

7.6.3 The detector exhaust

7.6.4 Wipe tests are sent to the National Leak Test Center for testing. Leak test reports are kept on file in the safety office.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water, wastewater and leachate samples are collected in unpreserved 1000 mL amber glass jars with PTFE septum-lined lids then stored at 0 – 6° C until extraction. At least two 1000 mL aliquots of aqueous sample must be collected to meet reporting limit requirements or to re-extract when necessary. If matrix spikes are needed, collect two additional 1000 mL sample volumes.

8.2 Waste, sludge and soil samples are collected in glass jars with no preservative added. At least 30 g of solid is needed to meet reporting limit requirements. Additional sample must be collected to re-extract when necessary, for matrix spikes and for a percent solids determination.

8.3 Clean wipes are sent to the client in 125 mL glass jars with PTFE septum-lined lids, with 30 mL of hexane in the jar.

8.4 The water sample extraction must be performed within 7 days of the date of collection for method 8082 and within 365 days for method 608. Soils must be extracted within 14 days of the date of collection. Extract analysis must be completed within 40 days of sample extraction for method 8082 and within 365 days for method 608.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Gas chromatograph equipped with dual $_{63}$Ni electron capture detectors and flow valves capable of accurately delivering very low flow rates (~10 mL/min). Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.1.1 Instrument Set-up:

9.1.1.1 Injector Temperature: 205° C isothermal

9.1.1.2 Detector Temperature: 350° C

9.1.1.3 Oven Program: Initial temperature at 120° C (no hold), ramp to 160° C at 20° C/min (no hold), ramp to 300° C at 9° C/min, hold 3.67 minutes

9.1.1.4 Carrier Flow: 6.0 mL/min

9.1.2 Autosampler: Leap A200SE or Combi-Pal™ programmable injectors

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9.2 Software operated on a personal computer workstation possessing the capability to collect and archive analysis data. Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer specifications associated with the analytical instrument.

9.3 Columns:

9.3.1 Column A: RTX CLP, 30 m length, 0.53 mm ID, 0.50 µm film thickness.

9.3.2 Column B: RTX CLP2, 30 m length, 0.53 mm ID, 0.42 µm film thickness.

9.4 Microsyringes, glass, various sizes, gas-tight

9.5 Autosampler vials with PTFE-faced silicone septa, 2.0 mL

9.6 Amber vials, with PTFE-lined screw caps, 40 mL

9.7 Volumetric flasks, 10, 100, and 1000 mL class A

9.8 Analytical balance, capable of accurately weighing to 0.0001 g

9.9 Pasteur Pipets, 2 mL, disposable, glass

9.10 Vials, borosilicate glass with PTFE-lined screw caps, 10 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Direct inject gas chromatographs are equipped with silicone septa (Supelco Thermogreen or equivalent) which eventually core and leak. These must be replaced to avoid contaminating the injection port liner with rubber particles and to eliminate column bleed. Replace septa frequently enough to prevent leaking and bleed problems and/or after approximately 100 injections.

10.2 Injection port liners can become a source of contamination and flow restriction by accumulating sample debris and septum particles. Periodically replace the injection port liners to minimize the loss of peak resolution.

10.3 When conditions merit, clip about 6" off the front of the analytical column. When column resolution or chromatography deteriorates and cannot be solved by column clipping, the column must be replaced.

10.4 Electron capture detectors may begin to bleed radioactive particles as they age. Wipe tests must be conducted every six months to ensure there is no bleed. The cause is that sample components and constant use at high temperature oxidize the $_{63}$Ni foil inside a detector. Foil degradation also raises baseline noise and decreases the linear calibration range. When this happens, send the detector out for cleaning and/or refoiling. NEVER tamper with any internal ECD part as this could lead to radiation exposure. All ECD repair must be done by an outside licensed professional.

10.5 Replace gas filters annually.

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11.0 CHEMICALS AND REAGENTS

11.1 PCB standards are purchased as certified solutions in heat-sealed ampules.

11.2 Hexane, pesticide grade or better

11.3 Helium carrier gas, ultra high purity

11.4 Nitrogen makeup gas, filtered for oxygen, hydrocarbons and moisture

11.5 Surrogate standards – tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB), purchased as certified solutions.

12.0 STANDARDS PREPARATION

12.1 Stock Standards

12.1.1 Aroclor stock standards are purchased as certified solutions in hexane or methanol at 1000 ug/mL which are serially diluted in hexane. Stock standards are stored in 100 mL amber screw-cap bottles at 0 – 6° C and away from light. Being multicomponent analytes, PCB standards are prepared independently except for 1221/1254 and 1016/1260. These analyte pairs are combined due to dissimilar elution times.

12.1.2 Surrogate stock standards are purchased as certified solutions in acetone, typically at 200 ug/mL.

12.1.2 All stock standards must be replaced after one year or sooner if comparison with calibration standards indicates a problem.

12.2 Intermediate standards are made by diluting stock standards in hexane (1:100). Intermediate standards are stored in 40 mL amber screw-cap vials at 0 – 6° C and away from light.

12.3 Calibration Standards

12.3.1 A standard of Aroclors 1016/1260 includes the majority of peaks represented in the other five Aroclors. Consequently, a multi-point initial calibration of the 1016/1260 mixture at six concentrations demonstrates detector response linearity without performing independent calibrations for each of the seven Aroclors.

12.3.2 Prepare six calibration standards containing equal concentrations of Aroclors 1016 and 1260, and equal concentrations of surrogates TCMX and DCB. Dilute with hexane. The following concentrations correspond to the expected sample concentration range and bracket the linear detector response: 0.04, 0.08, 0.16, 0.32, 0.64 and 1.28 ug/mL for 1016/1260, and 0.005, 0.01, 0.02, 0.04, 0.08, 0.16 ug/mL for TCMX/DCB.

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12.3.3 The lowest calibration standard is the minimum reporting limit.

12.3.4 The same calibration concentrations are used if a multi-point calibration is used for Aroclors other than 1016/1260. However, an acceptable linear multi-point 1016/1260 calibration justifies the use of a single-point calibration for quantitation of other Aroclors except when otherwise specified.

12.3.5 Standards of each Aroclor are required for pattern recognition. Assuming linearity has been demonstrated by the 1016/1260 calibration, a single-point calibration may be used for pattern recognition. Prepare a standard at 0.32 ug/mL for each Aroclor and 0.04 ug/mL for TCMX/DCB.

12.4 All standards must be labeled with the following information:

12.4.1 Initials of preparer

12.4.2 Preparation date

12.4.3 Concentration, with units

12.4.4 Standard name

12.4.5 Standard identification number as it appears in the standards log

12.4.6 Expiration dates must be no later than six months from the date of preparation for intermediate and working standards and no later than one year for purchased stock standards that have been opened and stored without headspace. The expiration date of an intermediate or working standard cannot exceed the purchased standard(s) expiration date from which it was made.

12.4.7 Solvent used

12.5 All standards preparation must be recorded in the standards log and uniquely identified. Refer to Attachment 21.2 for an example standards log.

13.0 SAMPLE PREPARATION

13.1 Water samples are extracted in accordance with TriMatrix SOP GR-09-107.

13.2 Soil and wipe samples are extracted in accordance with TriMatrix SOP GR-09-108

13.3 Waste dilution samples are extracted in accordance with TriMatrix SOP GR-09-105.

13.4 Extracts are cleaned up for sulfur in accordance with TriMatrix GR-09-109.

13.5 Extracts are cleaned up with sulfuric acid in accordance TriMatrix GR-09-110.

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13.6 Extracts are cleaned up with Florisil and silica gel in accordance with TriMatrix SOP GR-09-111 or TriMatrix SOP GR-09-120.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 Following a clean hexane blank, run a 6-point curve for Aroclors 1016/1260 (refer to Section 12.3.2 for concentrations). The low standard, when extracted, is equivalent to the minimum reporting limit (500 ug/L for waste dilution, 0.1 ug/L for water, ~17 ug/kg for soil and 500 ug/wipe for wipes). A single-point 0.32 ug/mL calibration standard is used for pattern recognition of other Aroclors.

14.1.1.1 Inject 1.0 µL of each calibration standard. Choose five peaks for quantitation, which are characteristic of the pattern. Record the height of each Aroclor peak chosen. Try to choose peaks that are at least 25% of the height of the largest peak in the Aroclor pattern. Later-eluting peaks are generally more stable for quantitation.

14.1.2 Determine the average calibration factor (CF_{avg}) for the sum of the chosen peaks using the following equations:

$$\text{Calibration Factor (CF)} = \frac{\text{sum of the chosen peak responses}}{\text{Aroclor concentration, ug/mL}}$$

$$\text{Average Calibration Factor } (CF_{avg}) = \frac{\text{sum of calibration factors } (CF_{sum})}{\text{number of calibration points}}$$

14.1.3 Calculate the relative standard deviation (%RSD) of CF_{avg} using the following equation:

$$\text{percent relative standard deviation (\%RSD)} = \frac{\text{standard deviation of } CF_{avg}}{CF_{avg}} \Big| 100$$

Note: The RSD value must be ≤20% to assume the calibration passes through the origin to use average calibration factor when reporting by method 8082 (or ≤10% for method 608). If the calibration does not in fact pass through the origin, do not use CF_{avg} even if %RSD is acceptable.

14.1.4 If %RSD is greater than the method specified criterion (10% for 608 and 20% for 8082), perform instrument maintenance and run the calibration again. Polynomial regression must not be used if the calibration factor has previously been acceptable. Instrument maintenance must be performed to obtain an acceptable average calibration factor or linear regression. When linear regression is used, the correlation coefficient (r) must be ≥0.99.

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Note: If %RSD is >20%, the calibration does not actually pass through the origin, or if linear regression is used, a multi-point regression must be used for quantitation where a single-point calibration CF_{avg} of Aroclor 1221, 1232, 1242, 1248, 1254, 1262 or 1268 might otherwise be allowed.

Note: Single-point calibration may be used to report by method 8082 only if the project and/or data quality objective does not require a multi-point calibration.

Note: For samples analyzed by method 608 or if project and/or data quality objectives dictate, detected Aroclors other than 1016/1260 must be re-analyzed with a multi-point calibration of the Aroclor.

14.1.5 Construct linear regression curves by plotting peak response (height) against concentration. The regression must not be forced through the origin:

14.1.5.1 Linear regression requires five calibration points and is calculated as follows:

$C_s = (H_s - b)/a$

Where:
H_s = Height of the target analyte peak, in sample "s"
C_s = Concentration of the target analyte in calibration standard "s"
a = Slope "s" of the curve (also called the coefficient of C)
b = The intercept

14.1.5.2 Do not select regression as a calibration option unless it has been proven that after instrument and column maintenance, calibration does not characteristically go through the origin and/or has not previously generated a consistently acceptable average calibration factor.

14.1.6 Sample analysis may not begin without an acceptable 1016/1260 calibration. For an Aroclor 1260 calibration curve example, refer to Attachment 21.3.

14.1.7 Initial calibration if analyzing by method 608 is as follows:

14.1.7.1 Initial calibration consists of a minimum of three concentration levels for all Aroclors.

14.1.7.2 The %RSD must be <10% to use an average calibration factor for quantitation.

14.1.7.3 If %RSD is >10%, a minimum of a three-point linear calibration curve must be constructed for ALL Aroclor quantitation, not just 1016/1260. The correlation coefficient (r) must be ≥0.99 for an acceptable regression.

14.2 Second-Source Calibration Verification (SCV)

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14.2.1 Prepare a second-source calibration verification standard (SCV) at 0.40 ug/mL to validate the initial calibration Aroclors 1016/1260 solutions. Acceptance limits for the SCV are listed in the laboratory information management system (Element™)

14.2.2 If recovery is unacceptable, re-run the SCV once. If still unacceptable, re-prepare the SCV and re-analyze successfully. If the second SCV fails, re-calibrate and re-analyze the SCV successfully. Sample analysis may not begin without an acceptable SCV being run.

14.3 Continuing Calibration Verification (CCV)

14.3.1 On non-curve days, analyze a mid-point CCV containing 0.32 ug/mL of Aroclors 1016/1260 and surrogates (0.05 ug/mL). Calculate percent difference or percent drift. Regardless of calibration technique, percent difference or percent drift must be within ±15% of the expected concentration to be acceptable.

14.3.2 When using average calibration factor, verification is performed using percent difference:

$$\textit{Percent Difference} = \frac{CF_{ccv} - CF_{avg}}{CF_{avg}} \times 100$$

where:
CF_{ccv} = Response factor from verification standard
CF_{avg} = Average calibration factor from initial calibration

14.3.3 When using linear regression, verification is performed using percent drift:

$$\text{Percent Drift} = \frac{R_2 - R_1}{R_1} \times 100$$

where:
R_1 = Expected concentration, in ug/mL
R_2 = Observed concentration, in ug/mL

14.4.3 A CCV is not required on the remaining Aroclors unless a multi-point regression is used.

14.4.4 Quantitate based on the following criteria:

14.4.4.1 If an Aroclor other than 1016/1260 is identified using method 8082, quantitate using the single-point standard run with the initial 1016/1260 calibration.

Note: Single-point calibration may be used for method 8082 samples only if the 1016/1260 calibration model is CF_{avg} but may not be used if project and/or data quality objectives specify otherwise.

14.4.4.2 For Aroclors 1016 or 1260, use the initial 1016/1260 calibration.

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15.0 ANALYTICAL PROCEDURE

15.1 The analysis sequence begins with a hexane blank (BLK), followed by an initial calibration or on non-curve days by the 0.32 ug/mL Aroclor 1016/1260 CCV. Analyze the other Aroclor CCVs, at 0.32 ug/mL, at this time for confirmation analysis.

15.2 After every 10 injections, the 1016/1260 CCV must be analyzed to verify the calibration is in control. Both Aroclors in the CCV must have less than ±15 percent difference or drift from the initial calibration. If any CCV Aroclor fails the ±15 percent criteria on the low side (less than the expected value), all extracts run before and after the failing CCV must be re-injected on a calibration that is in control. If any CCV Aroclor fails the ±15 percent criteria on the high side (greater than the expected value), only those extracts with detectable results need re-injected.

Note: At least once per month, vary the CCV concentration to demonstrate detector and calibration accuracy at concentrations other than the mid-point.

15.3 Identify individual PCB congeners by comparing the congener retention time against the initial calibration peak. For positive identification, the peak retention time must elute within the calculated retention time window on both columns. However, PCB Aroclors must be identified by pattern recognition

15.3.1 Many Aroclors contain identical congeners. However the relative peak height is unique for each, with each Aroclor having a distinct pattern. To identify and report an Aroclor, the sample pattern of peaks must match the standard. Often, a pattern may be difficult to isolate because of a complex matrix or weathering. Consequently, experience and analytical judgment must be used in the identification process. Aroclor 1221 is unique and presents a challenge. It elutes early and has a simple pattern. All 1221 congeners are part of other Aroclors and are most affected by matrix. Exhibit caution in the identification of Aroclor 1221.

15.3.2 Aroclors can be grouped into two groups. Early eluting Aroclors (Attachment 21.9 – 21.18) are as follows:

15.3.2.1 Aroclors 1221, 1232, 1242, 1248 and 1016 are early eluters. The initial and most significant criterion for identification is the proportional relationship between peaks 1 through 7 in Attachment 21.8 (key identification peaks). Secondly, the absence or presence of peaks 8 through 14.

15.3.2.2 Quantitation

Use at least three peaks, typically five from 1 through 7 (Attachment 21.8) except for 1221 (Attachment 21.12). Do not use disproportionate peaks to avoid matrix interference. As a rule of thumb, relative peak ratios used for quantitation in samples must be within 25% of the ratios in the identified Aroclor standard.

Note: If there is an obvious overlapping peak or other matrix interference precluding the use of any chosen calibration peak, recalibrate and report using the sum of the unaffected peaks and discuss in the report narrative that matrix interference precluded the use of all chosen calibration peaks.

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15.3.3 Late eluting Aroclors (Attachments 21.9 – 21.12, 21.19 – 22.22)

15.3.3.1 Late eluting Aroclors are usually easier to identify. The following criteria apply:

15.3.3.1.1 Aroclors 1254, 1260, 1262, and 1268 are late eluters

15.3.3.1.2 The second indication is the absence or presence of peaks 8 through 14 (Attachment 21.8).

15.3.3.1.3 The third indication is the relationship of peaks 8 through 13 to each other.

15.3.3.1.4 The fourth indication is the proportion of later eluting peaks.

15.3.3.2 Quantitation. Use at least three congener peaks, preferably five. Use peaks later than peak 7 (Attachment 21.8) if early Aroclors are present. Do not use disproportionate peaks, to avoid matrix interference.

15.3.4 Samples containing more than one Aroclor.

15.3.4.1 Once an Aroclor is identified, the same peaks cannot be used to identify a different Aroclor since it is impossible to identify 1248 and 1242 in the same sample. If both are present it will appear to be one or the other. Early and late eluting Aroclors are not clearly divided. If an Aroclor is present from each elution group, patterns may be distorted by overlapping peaks. This is important when attempting to identify multiple Aroclors. High levels of an early eluting Aroclor may appear to be later eluting peaks of a separate Aroclor and care must be taken to look at relative peak intensities when comparing with standard patterns.

15.3.4.2 Peaks 1 through 7 must be used for quantitating early eluting Aroclors (Attachment 21.8). Peaks 8 through 14 must be used for late eluting Aroclors. A minimum of three peaks (preferably five) from each elution group are required for quantitation. These peaks have been shown to be at least 25% of the highest peak. A calibration must be performed if using other congeners than those chosen for the initial calibration, with samples and all quality control quantitated against the new calibration.

15.4 Retention Time Windows

15.4.1 Perform a retention time study by making three injections of each Aroclor (not just the 1016/1260 mixture) over a 72-hour period and average the results. Calculate the mean and standard deviation for each peak used in quantitation. Calculate the retention time window by multiplying the standard deviation by ±3.

15.4.2 For a positive identification based on retention time, all congener quantitation peaks must elute within the absolute retention time.

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15.4.3 Since an initial CCV is run before sample analysis each day/shift, establish the center of the retention time window for the day/shift using the initial CCV quantitation peak retention times. Establish the retention time window using the initial CCV retention times plus or minus three times the retention time study standard deviation.

15.4.4 All Aroclor peaks chosen for quantitation in samples, quality control and standards run after the ICV must elute within the retention time window. Samples run in association with any subsequent CCV eluting outside the retention time window must be re-analyzed.

15.4.5 Update retention time windows whenever a new column is installed.

15.5 Example chromatograms showing Aroclor patterns on the RTX-CLP and RTX-CLP2 columns are provided in Attachments 21.9 – 21.22.

15.6 All targeted analyte identifications must be confirmed by running on the secondary column or by gas chromatography/mass spectrometry (GCMS).

15.6.1 When confirmation is made using a second column, the secondary analysis must meet all QC criteria described for the primary column, including calibration and retention times. Evaluate the agreement between results after identification has been confirmed by relative percent difference.

15.6.2 Calculate relative percent difference (RPD) using the formula below where R_1 and R_2 are the two results. The vertical bars in the numerator indicate the absolute value of the difference. Therefore, RPD is always a positive value.

$$RPD = \frac{|R_1 - R_2|\,200}{R_1 + R_2}$$

15.6.3 If one result is significantly higher (if RPD is greater than 40%), check to see if an obviously overlapping peak is causing one erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration. If no anomalies are noted, review the chromatographic conditions.

15.6.4 If there is no evidence of chromatographic problems such as overlapping peaks, report the lower result (unless otherwise specified) with narration as to the disparity between column results.

15.6.5 If RPD is less than 40%, report from the primary column.

15.7 If an extract concentration exceeds the calibration range, dilute and re-analyze. All extract dilutions must keep average peak response in the upper half of the calibration range. Re-dilute if concentrations are outside this range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Aroclors identified in samples must be quantitated by comparison with corresponding standards.

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16.2 Once an Aroclor has been identified, the sum of the height response of congener peaks are compared to the sum of height response of corresponding peaks in the calibration in accordance with Section 14.0.

16.3 If any chosen quantitation peak needs omitted due to interference, a minimum of three peaks must remain for quantitation. The calibration must be adjusted by removing any rejected congener peak from the summed peak response. Quality control must also be re-quantitated using the new summed peak response and acceptance re-verified.

16.4 Sample concentration is calculated using the extract Aroclor concentration in one of the following equations:

Water Samples:

$$\text{Sample Concentration (in ug/L)} = \frac{(\text{ug/mL})(V_e)(DF)}{(V_w)}$$

Wipes:

$$\textit{Wipe Concentration (in total ug)} = (ug/mL)(Ve)(DF)$$

Soil Samples:

$$\text{Sample Concentration (in mg/kg)} = \frac{(\text{ug/mL})(Ve)(DF)}{(Vs)(PS)} \times 1000$$

where:

V_e = Total volume of extract, in µL
DF = Post extraction dilution factor as a ratio
V_w = Initial water sample volume, in mL
V_s = Initial soil sample mass, in g
PS = Soil percent solids in decimal form (0.98 not 98%)

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are responsible for data quality and for filling in all documentation and paperwork correctly. It is important to document analysis by correctly filling in, handing in and archiving all paperwork correctly.

17.2 Laboratory Information Management System (Element™) data transfer must be in accordance with TriMatrix SOP GR-10-123. If internal chain-of-custody is required, it is very important that a CoC form be filled in correctly and completely.

17.3 All run, maintenance, CD archival and standard logs must be filled in completely and correctly. Corrections on hardcopy must be made with a lineout (not a writeover) and must be initialed and dated. Blank lines in run logbooks must be Z'd out. Refer to Attachment 21.6 for a run logbook example.

17.4 All calibration run hardcopy must be archived in the correct storage box.

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17.5 All other analysis documentation must be archived appropriately for ease in retrieval.

18.0 QUALITY ASSURANCE

18.1 Blanks, surrogates and matrix spikes must be included as part of quality control.

18.2 Before calibration, initial CCV or samples are run, a solvent blank must be analyzed to demonstrate the analytical system free from interferences and contamination at the reporting limit. Solvent blanks must be run more frequently when showing evidence of carryover. The extraction blank (BLK) may be used as a solvent blank to minimize runs.

18.2.1 The extraction blank (BLK) is laboratory reagent water extracted with hexane as a sample and containing surrogates.

18.2.2 The BLK must be non-detect at the minimum reporting limit.

18.2.3 At a minimum, the BLK is run at the beginning of every analytical shift and/or with each batch of up to 20 samples. Whichever is more frequent.

18.2.4 The BLK must be extracted more frequently if showing contamination to isolate the problem.

18.2.5 Extraction blanks must be prepared as samples, in accordance with all extraction procedure requirements, including cleanup.

18.2.6 Refer to TriMatrix SOP GR-04-101 for corrective action during blank contamination.

18.3 Surrogate criteria are as follows:

18.3.1 All samples must be spiked with tetrachloro-m-xylene and decachlorobiphenyl surrogates. Until twenty samples of a given matrix have been analyzed, acceptance limits will be 50-150% recovery. Once twenty samples of a given matrix have been analyzed, statistical acceptance limits must be generated in Element™ and used. At a minimum, surrogate recovery limits will be updated annually on a matrix-by-matrix basis. Calculate surrogate recovery for each extract analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-04-101 to determine when and how data is qualified.

18.3.1.1 High surrogate recovery may be due to co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.3.1.2 Low recovery may be due to poor extraction efficiency in the matrix. Verify by re-extracting the sample if sufficient sample volume and hold time permits.

18.3.1.3 If surrogate recovery in a BLK is below the lower control limit (LCL), only samples with failing surrogate recovery require re-extraction if sufficient sample volume and hold time permits. If BLK surrogate recovery is above the upper

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control limit (UCL), no corrective action is required if sample surrogate recovery is acceptable.

18.3.1.4 If surrogate recovery is outside control limits in the matrix spike and/or matrix spike duplicate, re-analysis is only required if blank spike (BS) recovery is also outside control limits. If the BS is out-of-control after re-analysis, all associated samples must be re-extracted if sufficient sample volume and hold time permits.

18.3.2 Control limits are calculated as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where:
p = average percent recovery
s = standard deviation of the percent recovery

18.3.3 If surrogate recovery is not within control limits, take corrective action based on TriMatrix SOP GR-04-101, which outlines sample re-extraction if sufficient sample volume and hold time permits and/or data qualification. If sample volumes and holding times permit, samples with low recoveries must be re-extracted, unless matrix interference indicates otherwise. Samples with high recoveries must be re-extracted only if there are detectable results. If many samples are out-of-control for no apparent reason, the gas chromatograph must be inspected for mechanical failure and appropriate repairs made. Once repairs have been made, extracts run in association with the failure must be re-analyzed.

18.3.4 Continuing calibration verification (CCV) surrogate recovery is handled as follows:

18.3.4.1 If a CCV has a surrogate recovery outside control limits but surrogate recovery in all samples is in control, instrument recalibration and extract re-analysis is not required.

18.3.4.2 If the CCV <u>and</u> sample extracts have high surrogate recovery, sample extracts must be rerun after instrument recalibration.

18.3.4.3 If the CCV <u>and</u> sample extracts have low recovery, sample extracts must be rerun after instrument recalibration.

18.4 Matrix Spikes (MS) and Blank Spikes (BS)

18.4.1 Extract a matrix spike and matrix spike duplicate at a frequency of once for each batch of up to 20 samples for each matrix.

18.4.1.1 If one to ten samples are extracted in a month, at least one matrix spike and one matrix spike duplicate must be extracted.

18.4.1.2 When reporting by method 608, matrix spike at a frequency of once for each batch of up to 10 samples.

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18.4.1.3 When reporting by method 608, extract the blank spike once for each batch of up to 10 samples. Otherwise, a blank spike must be extracted every shift samples are extracted, regardless of the number of samples extracted.

18.4.1.4 If there is insufficient sample volume to perform matrix spikes, extract a duplicate blank spike.

18.4.2 Until 15-20 matrix spikes, matrix spike duplicates and blank spikes have been analyzed, evaluate recovery against default limits of 50-150% and a duplication limit of 40%. Once generated, statistical control limits are listed in Element™.

18.4.3 Control limits must be updated at least annually.

18.4.4 Calculate matrix spike recovery is calculated as follows:

$$\text{Percent Recovery} = \frac{(\text{Aspk} - \text{Asmp})}{\text{SPK}} \times 100$$

where:
Aspk = Concentration in the spiked sample (ug/L)
Asmp = Concentration in the unspiked sample (ug/L)
SPK = Concentration spiked (ug/L) [(concentration of spike standard in ug/mL x mL spiked) / initial sample volume]

18.4.5 Calculate relative percent difference (RPD) as follows:

$$\text{RPD} = \frac{|\text{SPK}_1 - \text{SPK}_2|\,200}{\text{SPK}_1 + \text{SPK}_2}$$

where:
SPK_1 = Matrix Spike
SPK_2 = Matrix Spike Duplicate

18.4.6 If recovery or duplication is not within acceptable limits, perform corrective action in accordance with TriMatrix SOP GR-04-101.

18.4.7 If a blank spike is out-of-control, the problem must immediately be identified and corrected before further samples are extracted. The purpose of an LFB is to verify that out-of-control analytes in a matrix spike are the result of matrix interference rather than extraction or instrumental error. Failure of any part of a blank spike requires corrective action up to and including re-extraction of all associated samples. The only exception is when blank spike recovery is high and all associated samples are below the reporting limit.

18.4.8 Every effort must be made to determine the reason for a blank spike failure (whether mis-spiked, mis-extracted or something else) and appropriate actions taken. A non-conformance report must be written that outlines the investigation and findings. Corrective action must be in accordance with TriMatrix SOP GR-04-101.

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18.4.9 The Aroclor used for matrix and blank spiking must be changed periodically throughout the year so all analytes are spiked.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an initial demonstration of capability (IDC) study. While a demonstration of capability study is not instrument-dependent, a study is required on every instrument and every column to demonstrate an acceptable level of accuracy and precision.

19.2 Spike 1.0 mL of 1016/1260 blank spiking solution into each of four, 1 L aliquots of water and extract as samples. Cleanup is not necessary for the study unless the extraction analyst is performing a demonstration of capability study also. The blank spiking solution is not to be a standard used for calibration.

19.3 Analyze the four extracted spikes following every step in this procedure.

19.4 Input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and standard deviation for each Aroclor.

19.4.1 Average recovery must be within blank spike control limits and standard deviation must be ≤20% RSD. If both criteria pass, the analyst and the instrument/column used are authorized to run samples.

19.4.2 If any criterion fails, the demonstration of capability study must be repeated.

19.5 For a failed study, locate and correct the source of the problem and repeat the study successfully. Repeated failure will indicate a general problem with the procedure and/or techniques used. If this occurs, locate the problem, correct the procedure and/or techniques used and repeat the study successfully.

19.6 Samples may not be analyzed by any analyst or on any instrument until a successful demonstration of capability study has been completed and submitted to the quality control department for approval and documentation.

19.7 A Method Detection Limit (MDL) study must be performed for each Aroclor (not just for 1016/1260). MDL studies must be performed on all instruments involved in PCB analysis, on both columns and for each preparation procedure. Refer to TriMatrix SOP GR-10-125.

20.0 REFERENCES

20.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846,* 3rd Edition, Final Update III, December 1996, Method 8082, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Revision 0, December, 1996

20.2 *Code of Federal Regulations*, Title 40 Protection of Environment, Volume 19, July 1, 2001 Edition, Chapter I Environmental Protection Agency, Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A, Method 608, Organochlorine Pesticides and PCBs

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21.0 ATTACHMENTS/APPENDICES

21.1 Default Reporting Limits

21.2 Standards Log Example

21.3 Sample of Calibration Curves for PCB 1016/1260

21.4 Preparation Batch Report Example

21.5 Analysis Sequence Report Example

21.6 Sample Page of Instrument Run Logbook

21.7 Method Detection Limit Study Example

21.8 Key Peaks for Aroclor Identification

21.9 1016/1260 on RTX CLP Column

21.10 1016/1260 on RTX CLP2 MS Column

21.11 1221/1254 on RTX CLP Column

21.12 1221/1254 on RTX CLP2 Column

21.13 1232 on RTX CLP Column

21.14 1232 on RTX CLP2 Column

21.15 1242 on RTX CLP Column

21.16 1242 on RTX CLP2 Column

21.17 1248 on RTX CLP Column

21.18 1248 on RTX CLP2 Column

21.19 1262 on RTX CLP Column

21.20 1262 on RTX CLP2 Column

21.21 1268 on RTX CLP Column

21.22 1268 on RTX CLP2 Column

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Attachment 21.1
Default Reporting Limits

Attachment 21.1
Default Reporting Limits

Compound	Water (ug/L)	Soil (mg/kg)
PCB-1016	0.10	0.33
PCB-1221	0.10	0.33
PCB-1232	0.10	0.33
PCB-1242	0.10	0.33
PCB-1248	0.10	0.33
PCB-1254	0.10	0.33
PCB-1260	0.10	0.33
PCB-1262	0.10	0.33
PCB-1268	0.10	0.33

Compound	Wipes (ug)	Waste (mg/kg)
PCB-1016	0.5	0.50
PCB-1221	0.5	0.50
PCB-1232	0.5	0.50
PCB-1242	0.5	0.50
PCB-1248	0.5	0.50
PCB-1254	0.5	0.50
PCB-1260	0.5	0.50
PCB-1262	0.5	0.50
PCB-1268	0.5	0.50

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Attachment 21.2
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
A604465

Description:	(AMP) Aroclor 1016	Expires:	Sep-30-08
Standard Type:	Other	Prepared:	May-12-06
Solvent:	Solvent Lot #W-125M-07-02	Prepared By:	** Vendor **
Final Volume (mls):	1	Department:	Semivolatiles GC
Vials:	2	Last Edit:	Apr-12-07 13:29 by JLW

NSI part# 125M
LOT#125M-07-02
Received 10/31/06

Analyte	CAS Number	Concentration	Units
PCB-1016	12674-11-2	1000	ppm
PCB-1016 [2C]	12674-11-2	1000	ppm

Reviewed By Date

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Approved By: M 8-21-09 **QA Officer**

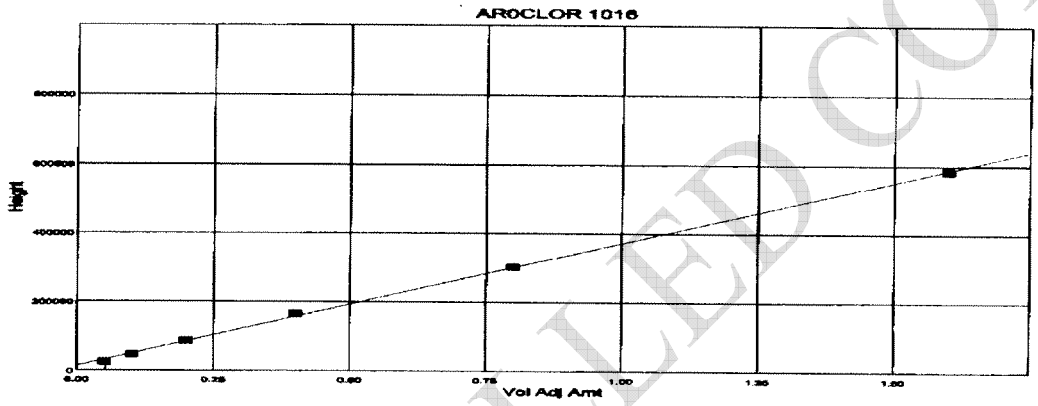
Approved By: 8/21/09 **Area Supervisor**

Attachment 21.3
Sample of a Calibration Curves for PCB 1016/1260

Fit Analysis Output For Method File: "C:\TC4\GC144\AB183\PCBAA.MTH"
Component Name: "AROCLOR 1016"
Date: 11/2/07 Time: 14:17

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) r^2 = 0.999340
Calibration Curve = (14080.706690) + (358052.114081)X

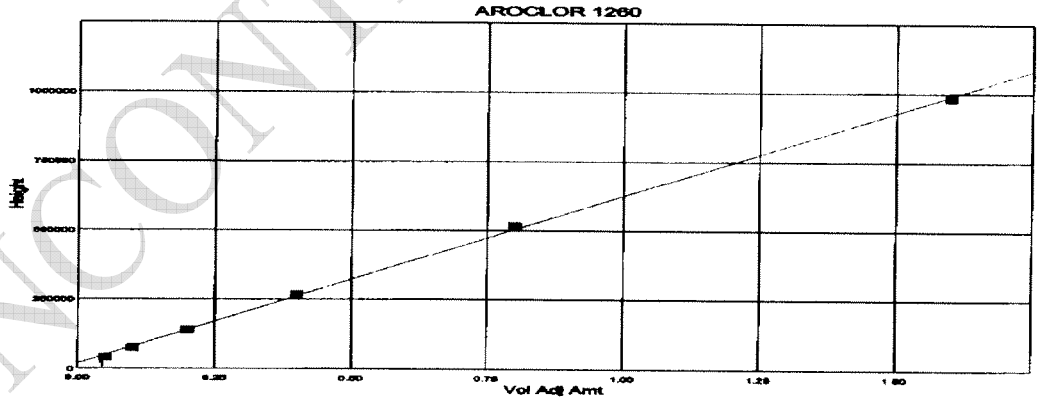


Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	0.050000	0.030468	0.019532	64.108	24989.846	31883.401	-6893.555	-21.626
2	0.100000	0.092697	0.007303	7.879	47271.013	49986.007	-2714.994	-5.242
3	0.200000	0.205577	-0.005577	-2.713	87688.228	85691.218	1997.019	2.330
4	0.400000	0.424569	-0.024569	-5.787	166098.724	157301.641	8797.083	5.592
5	0.800000	0.804923	-0.004923	-0.612	302885.168	300522.497	1762.681	0.587
6	1.600000	1.581766	0.018234	0.517	584015.925	586964.178	-2948.253	-0.502

Fit Analysis Output For Method File: "C:\TC4\GC144\AB183\PCBAA.MTH"
Component Name: "AROCLOR 1260"
Date: 11/2/07 Time: 14:18

Curve Parameters:

Curve #1 : 1st Order
Weighting Factor = 1.0 (No Weighting) r^2 = 0.998451
Calibration Curve = (18219.918867) + (607243.679103)X



Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
1	0.050000	0.036571	0.013429	36.719	40427.606	48582.095	-8154.488	-16.785
2	0.100000	0.092478	0.007522	8.134	76376.448	78944.279	-4567.831	-5.788
3	0.200000	0.200880	-0.000880	-0.438	140263.845	139648.646	591.199	0.424
4	0.400000	0.431739	-0.031739	-7.351	268245.919	261117.382	7128.537	2.730
5	0.800000	0.820798	-0.020798	-2.534	516644.454	504014.854	12629.600	2.506
6	1.600000	1.587433	0.012567	0.792	982178.780	989889.797	-7631.017	-0.771

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Attachment 21.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0705430 Page 1 of 1

Printed: 9/14/2007 4:20:19PM

Semivolatiles GC, Water, 3510C Liquid-Liquid Extraction

Surrogate #1 = 7040593 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0705288	8082 PCBs (std 7 aroclors)	0705322	8082 PCBs (std 7 aroclors)		

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0705430-BLK1		May-18-07 09:34	ASC	1000	2	1000				BLANK	
0705430-BS1		May-18-07 09:34	ASC	1000	2	1000		6120832	1000	LCS	
0705288-01	D	May-18-07 09:34	ASC	1000	2	1000				[redacted]	
0705322-01	H	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-02	I	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-03	E	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-04	G	May-18-07 09:34	ASC	1060	2	1000				[redacted]	
0705322-05	E	May-18-07 09:34	ASC	1060	2	1000				[redacted]	

Comments	Analyst Initials

bch_TriMatrix.rpt

Approved By: 8-21-09 **QA Officer**

Approved By: 8/21/09 **Area Supervisor**

Attachment 21.5
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **7052932** Page 1 of 3 Printed: 9/14/2007 4:24:07PM

Semivolatiles GC, Water, May-29-07

Instrument = 144, Calibration = 7E04002

Sequence Analyses:
608 PCBs (std 7 aroclors)
608 PCBs (Tonawanda)
8082 PCBs (master list)
8082 PCBs (std 7 aroclors)

Lab Number	Analysis	Contain	STD ID	ISTD ID	Client QC Type	Extraction Comments
7052932-ICV1	QC		7030266		INITIAL CAL CHECK	
7052932-ICV2	QC		7030946		INITIAL CAL CHECK	
7052932-ICV3	QC		7040212		INITIAL CAL CHECK	
7052932-ICV4	QC		7030269		INITIAL CAL CHECK	
7052932-ICV5	QC		7030271		INITIAL CAL CHECK	
0705286-04	8082 PCBs (master list)	A 02			[redacted]	[redacted]
0705392-01	8082 PCBs (std 7 aroclors)	C 02			[redacted]	[redacted]
0705433-BLK2	QC				BLANK	
0705433-BS2	QC				LCS	
0705430-BLK1	QC				BLANK	
0705430-BS1	QC				LCS	
0705288-01	8082 PCBs (std 7 aroclors)	D 01			[redacted]	
0705322-01	8082 PCBs (std 7 aroclors)	H 01			[redacted]	
0705322-02	8082 PCBs (std 7 aroclors)	I 01			[redacted]	
0705322-03	8082 PCBs (std 7 aroclors)	E 01			[redacted]	
7052932-CCV1	QC		7030271		CALIBRATION CHECK	
0705322-04	8082 PCBs (std 7 aroclors)	G 01			[redacted]	
0705322-05	8082 PCBs (std 7 aroclors)	E 01			[redacted]	
7052932-CCV2	QC		7030271		CALIBRATION CHECK	
0705651-BLK1	QC				BLANK	
0705651-BS1	QC				LCS	

Comments:

Analyst Initials:

seq_TriMatrix.rpt

Approved By: PD 8-21-09 **QA Officer**

Approved By: DJM 8/21/09 **Area Supervisor**

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Attachment 21.6
Sample Page of Instrument Run Logbook

TriMatrix Laboratories, Inc.

Instrument ID: HP5890 SERIES II 144	**Date:** 4/14/04	**Sequence #:** AB90	**Date Archived, Disk ID**
Instrument Settings/Injection Volume	**Breakdown**	**Quantitation Information**	**Routine Maintenance Items**
Column Type: RTXCLP/RTXCLP2	**DDT % Breakdown:**	**Curve Date:**	**New Column Date:**
Injection Volume: 1 uL	**Endrin % Breakdown:**	**Curve Type: Regression or Average CF**	**Column Clipped:** (Yes) / No
Program: 1 / 2 / 3 / 4	**Total % Breakdown <20%: Pass / Fail**	**Quantitated By: Curve**	**New Injection Port Liner** (Yes) / No
			New Septa (Yes) / No
			New Syringe: Yes (No)

Analyst	Run ID	File ID	Injection Time	Method	Client	Matrix	Dilution	ICV/CCV Check (85-115)	Sample Notes, Standard Numbers, Analytical Batch Information, etc.
JTAM	INST BLK	98	19:48	PC04144A/B		QC		PASS	
	1232	99	20:06						GC3.6-1
	1242	100	20:24						GC3.6-3
	1248	101	20:42						GC3.6-5
	1221/1254	102	21:00						GC3.6-7
	L1	103	21:18						GC3.6-10
	L2	104	21:36						
	L3	105	21:54						
	L4	106	22:12						
	L5	107	22:30						
	L6	108	22:48						
	L7	109	23:06						
	LCS	110	23:24						GC3.7-14
	MPB102 24094	111	23:42			Soil			
	LFB102	112	0:00						
	LFB102	113	0:18						

Approved By: DD 8-21-09
QA Officer

Approved By: [signature] 8/21/09
Area Supervisor

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Attachment 21.7
Method Detection Limit Study Example

TriMatrix Laboratories, Inc.

SEMI-VOLATILE LABORATORY
2007 SOIL
METHOD DETECTION LIMIT VERIFICATION STUDY

Parameter / Compound	Instrument Number	Reference Citation	MDL Result	Units	Date	Analyst	Verification Concentration	MDL/MDL Verification Concentration Difference	MPB Result	MDL Verification Result	MPB/MDL Verification Response Increase	Acceptable (≥3x MPB Response)?
Aroclor 1016	144B	8082	0.0042	mg/kg	4/12/07	JLW	0.01666	3.97	<.01666	0.01301	>3	Yes
Aroclor 1221	144B	8082	0.0060	mg/kg	4/25/07	JLW	0.01666	2.78	<.01666	0.02534	>3	Yes
Aroclor 1232	144B	8082	0.0084	mg/kg	4/12/07	JLW	0.01666	1.98	<.01666	0.02401	>3	Yes
Aroclor 1242	144B	8082	0.0163	mg/kg	4/12/07	JLW	0.01666	1.02	<.01666	0.02405	>3	Yes
Aroclor 1248	144B	8082	0.0038	mg/kg	4/12/07	JLW	0.01666	4.38	<.01666	0.02169	>3	Yes
Aroclor 1254	144B	8082	0.0040	mg/kg	4/12/07	JLW	0.01666	4.17	<.01666	0.02225	>3	Yes
Aroclor 1260	144B	8082	0.0054	mg/kg	4/12/07	JLW	0.01666	3.09	<.01666	0.00921	>3	Yes
Aroclor 1262	144B	8082	0.0052	mg/kg	4/12/07	JLW	0.01333	2.56	<.01333	0.01908	>3	Yes
Aroclor 1268	144B	8082	0.0044	mg/kg	4/12/07	JLW	0.01333	3.03	<.01333	0.02278	>3	Yes

file 8082 SOIL PCB_MDL2007.XLS

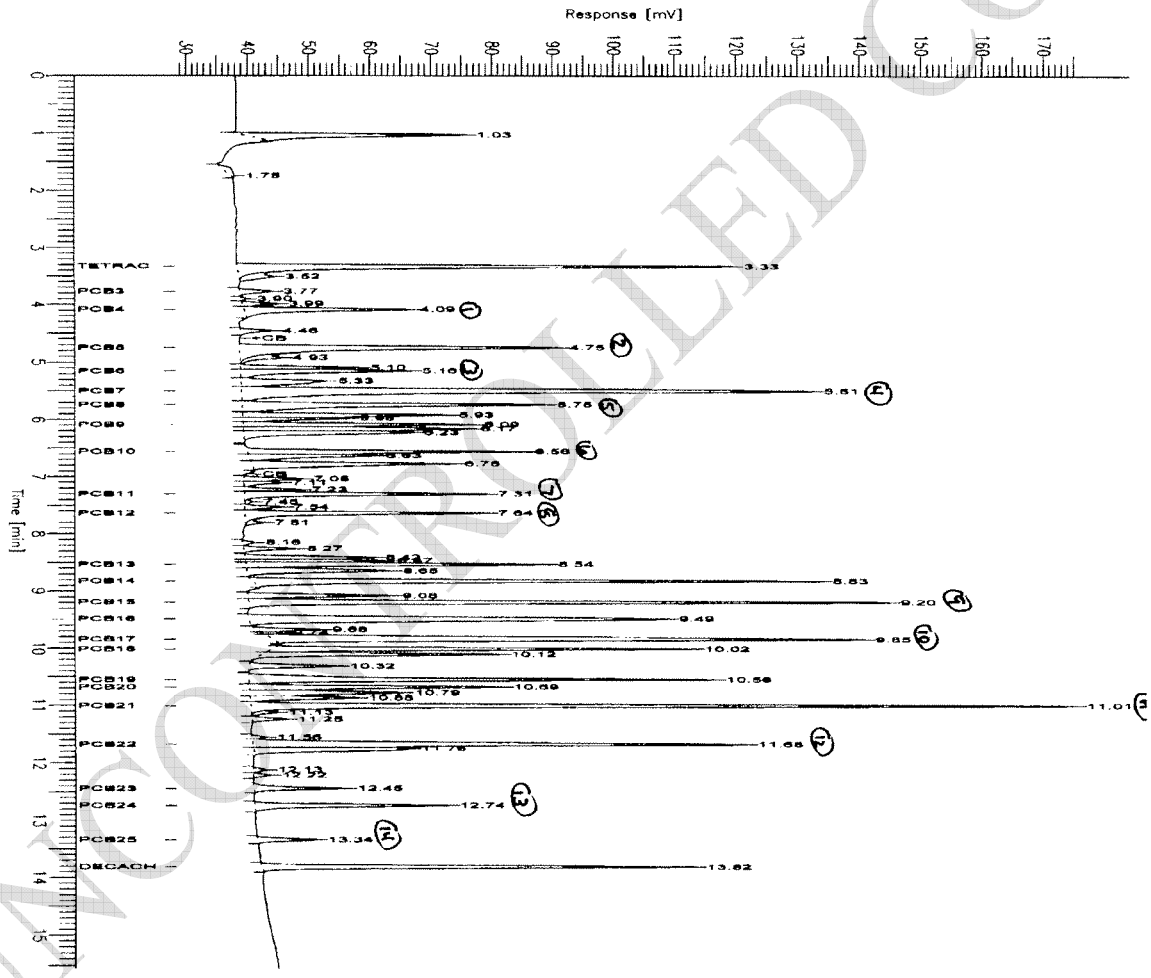
revision 2006.14

Attachment 21.8
Key Peaks for Aroclor Identification

Chromatogram

Sample Name : L4
FileName : C:\TC4\GC144\B90_229.RAW
Method : PC041948.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 28 mV

Sample #: 229 Page 1 of 1
Date : 4/29/04 14:56
Time of Injection: 4/19/04 14:29
Low Point : 28.24 mV High Point : 175.40 mV
Plot Scale: 147.2 mV



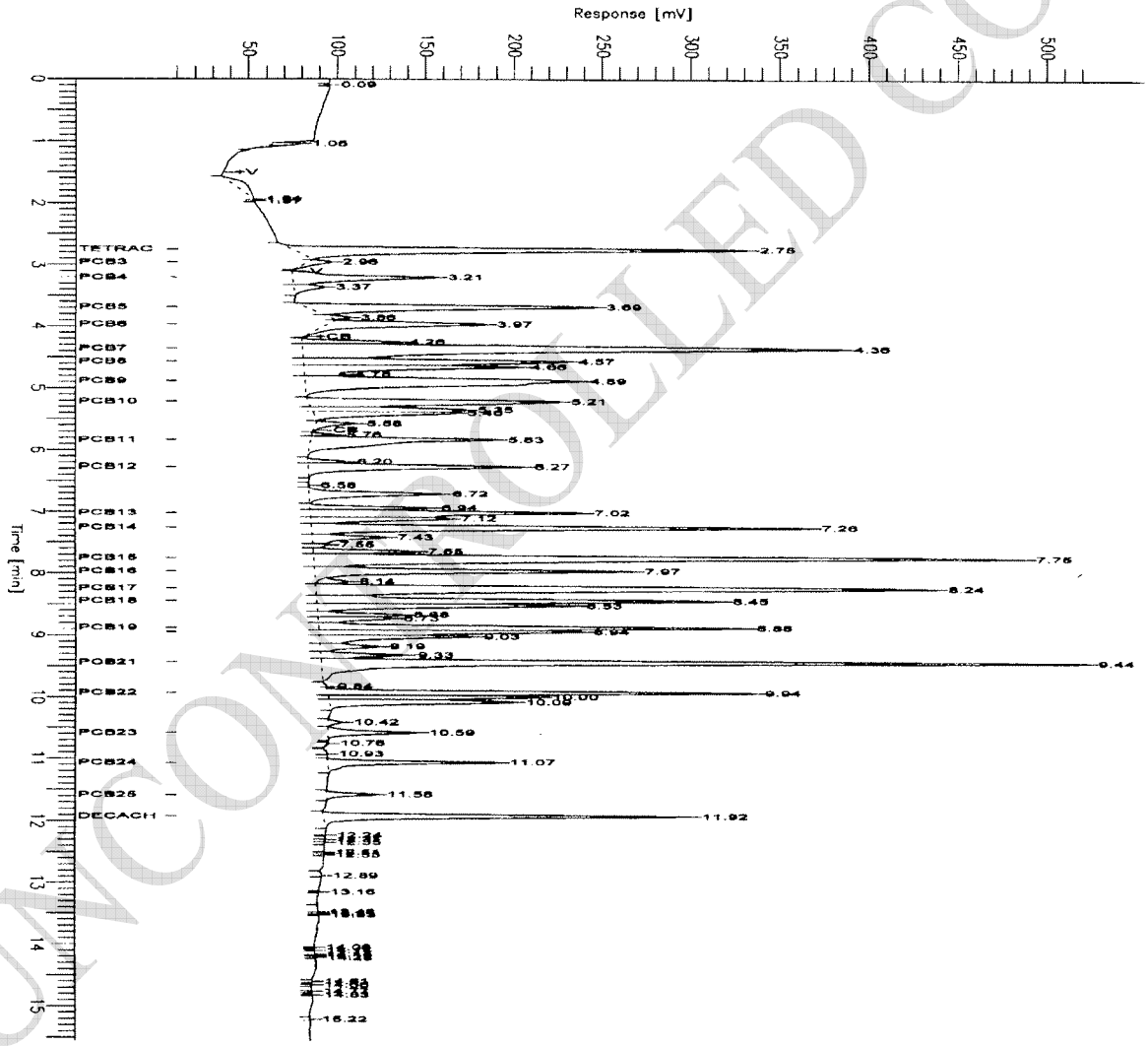
UNCONTROLLED COPY

Attachment 21.9
1016/1260 on RTX CLP Column

Chromatogram

Sample Name : L4 1016/1260
FileName : C:\TC4\GC144\A90_229.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 10 mV

Sample #: 229 Page 1 of 1
Date : 4/29/04 14:57
Time of Injection: 4/19/04 14:29
Low Point : 9.98 mV High Point : 523.95 mV
Plot Scale: 514.0 mV



UNCONTROLLED COPY

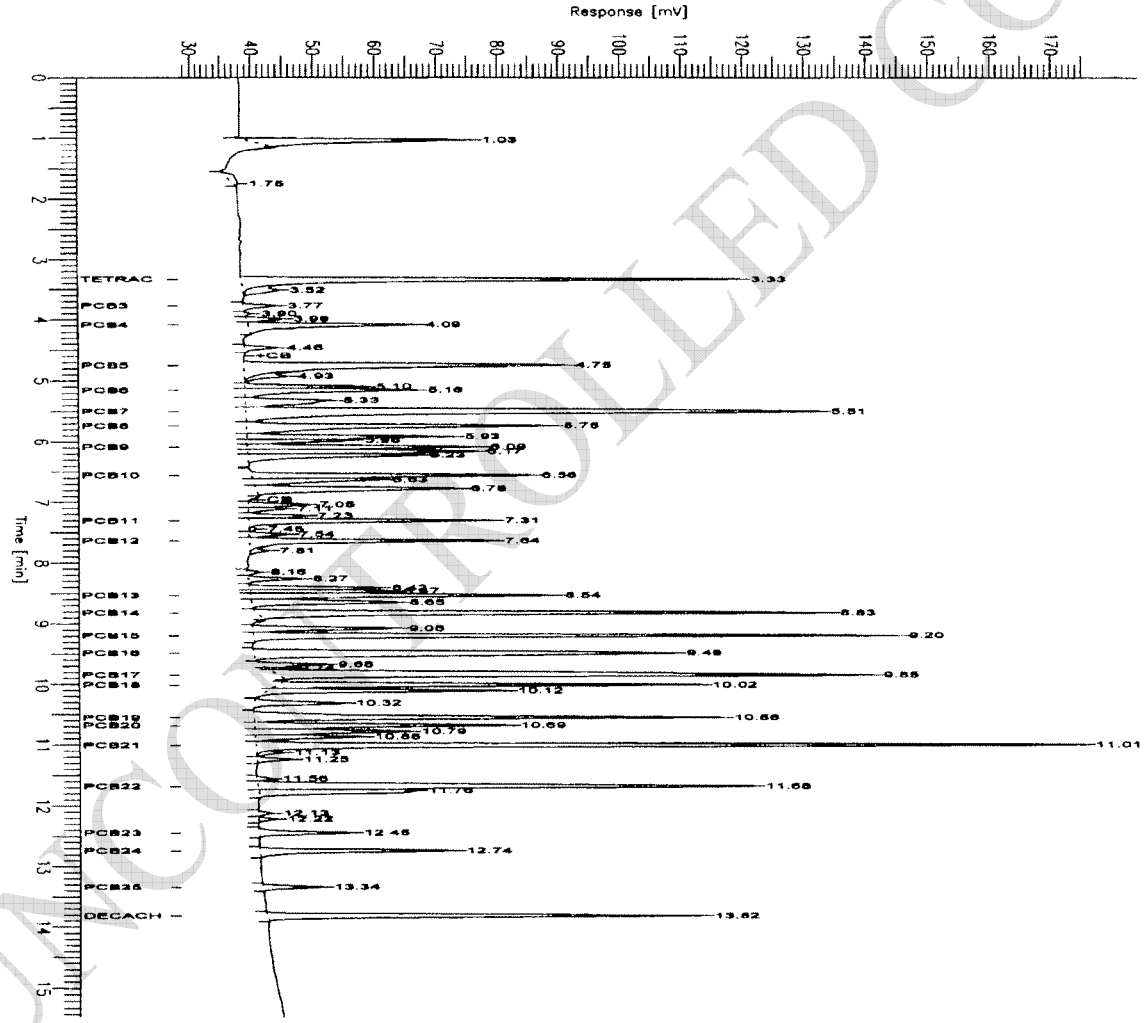
Approved By: 8-21-09 **QA Officer** **Approved By:** 8/21/09 **Area Supervisor**

Attachment 21.10
1016/1260 on RTX CLP2 MS Column

Chromatogram

Sample Name : L4 1016/1260
FileName : C:\TC4\GC144\B90_229.RAW
Method : PC041948.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 28 mV

Sample #: 229 Page 1 of 1
Date : 4/29/04 14:56
Time of Injection: 4/19/04 14:29
Low Point : 28.24 mV High Point : 175.40 mV
Plot Scale: 147.2 mV



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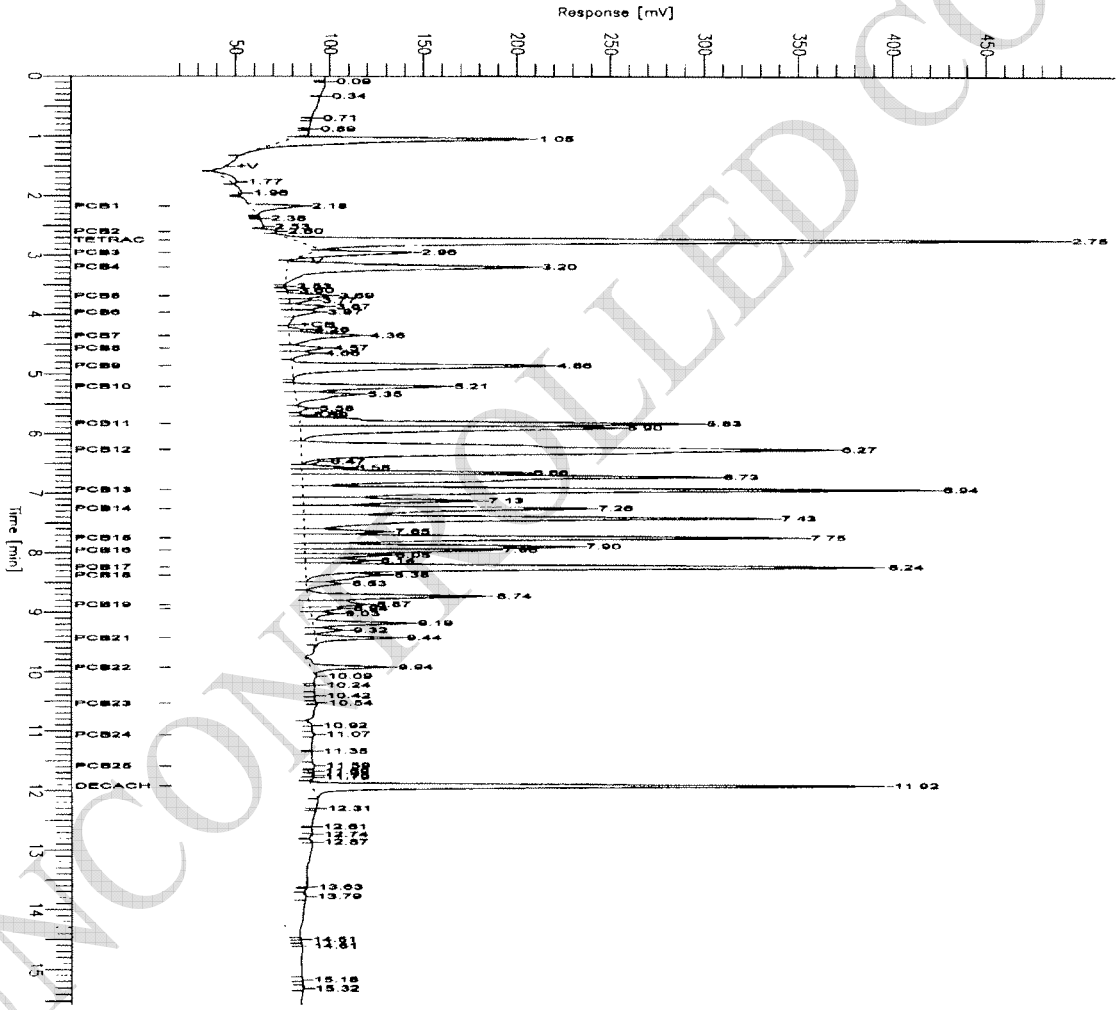
Attachment 21.11
1221/1254 on RTX CLP Column

Chromatogram

Sample Name : 1221/1254
FileName : C:\TC4\GC144\A90_223.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 15 mV

Sample #: 223 Page 1 of 1
Date : 4/29/04 14:57
Time of Injection: 4/19/04 12:41
Low Point : 15.22 mV High Point : 490.73 mV
Plot Scale: 475.5 mV



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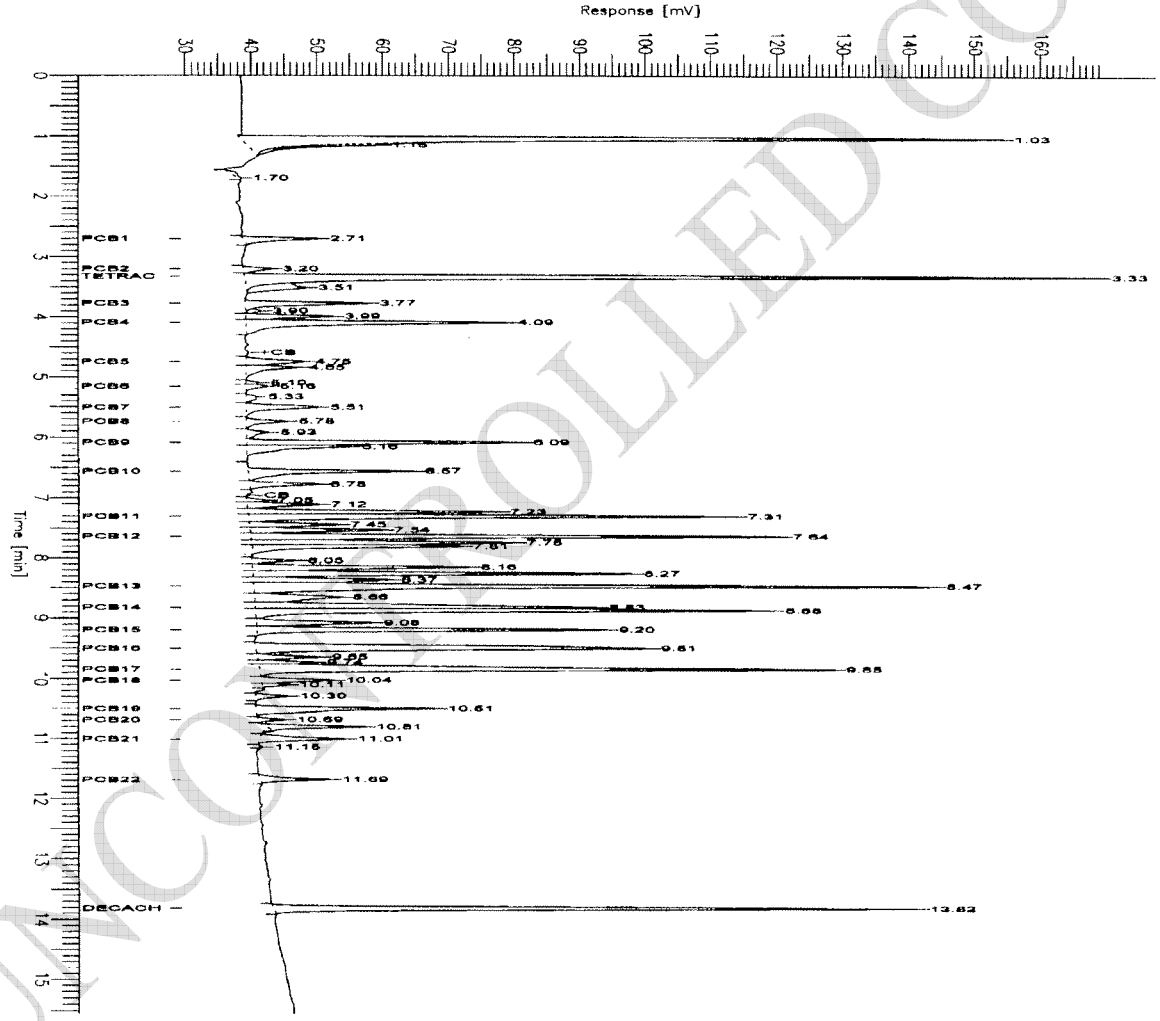
Approved By: 8-21-09 **Approved By:** 8/21/09
QA Officer **Area Supervisor**

Attachment 21.12
1221/1254 on RTX CLP2 Column

Chromatogram

Sample Name : 1221/1254
FileName : C:\TC4\GC144\B90_223.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 29 mV

Sample #: 223 Page 1 of 1
Date : 4/29/04 14:55
Time of Injection: 4/19/04 12:41
Low Point : 29.39 mV High Point : 169.23 mV
Plot Scale: 139.8 mV



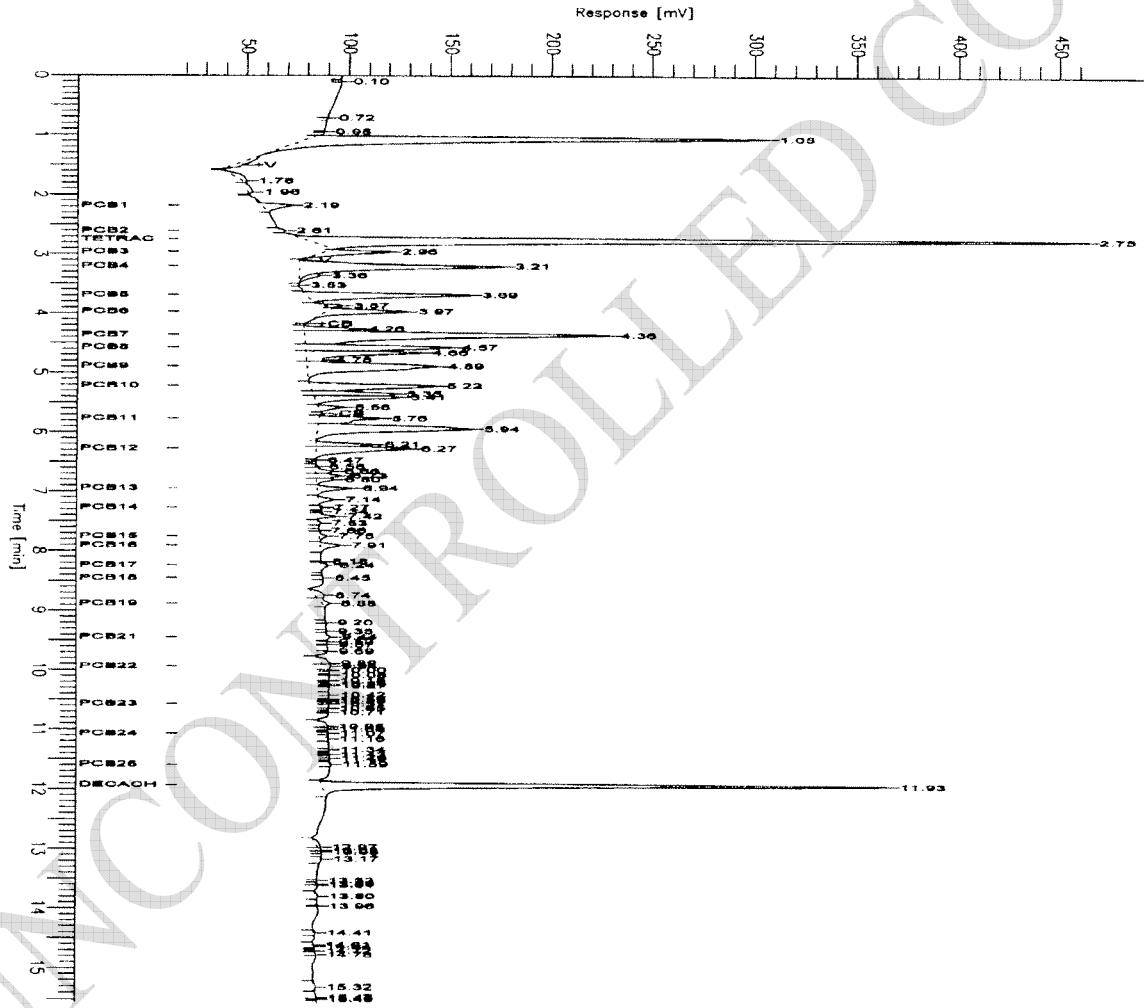
UNCONTROLLED COPY

Attachment 21.13
1232 on RTX CLP Column

Chromatogram

Sample Name : 1232
FileName : C:\TC4\GC144\A90_220.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 17 mV

Sample #: 220 Page 1 of 1
Date : 4/29/04 14:56
Time of Injection: 4/19/04 11:47
Low Point : 16.51 mV High Point : 464.38 mV
Plot Scale: 447.9 mV



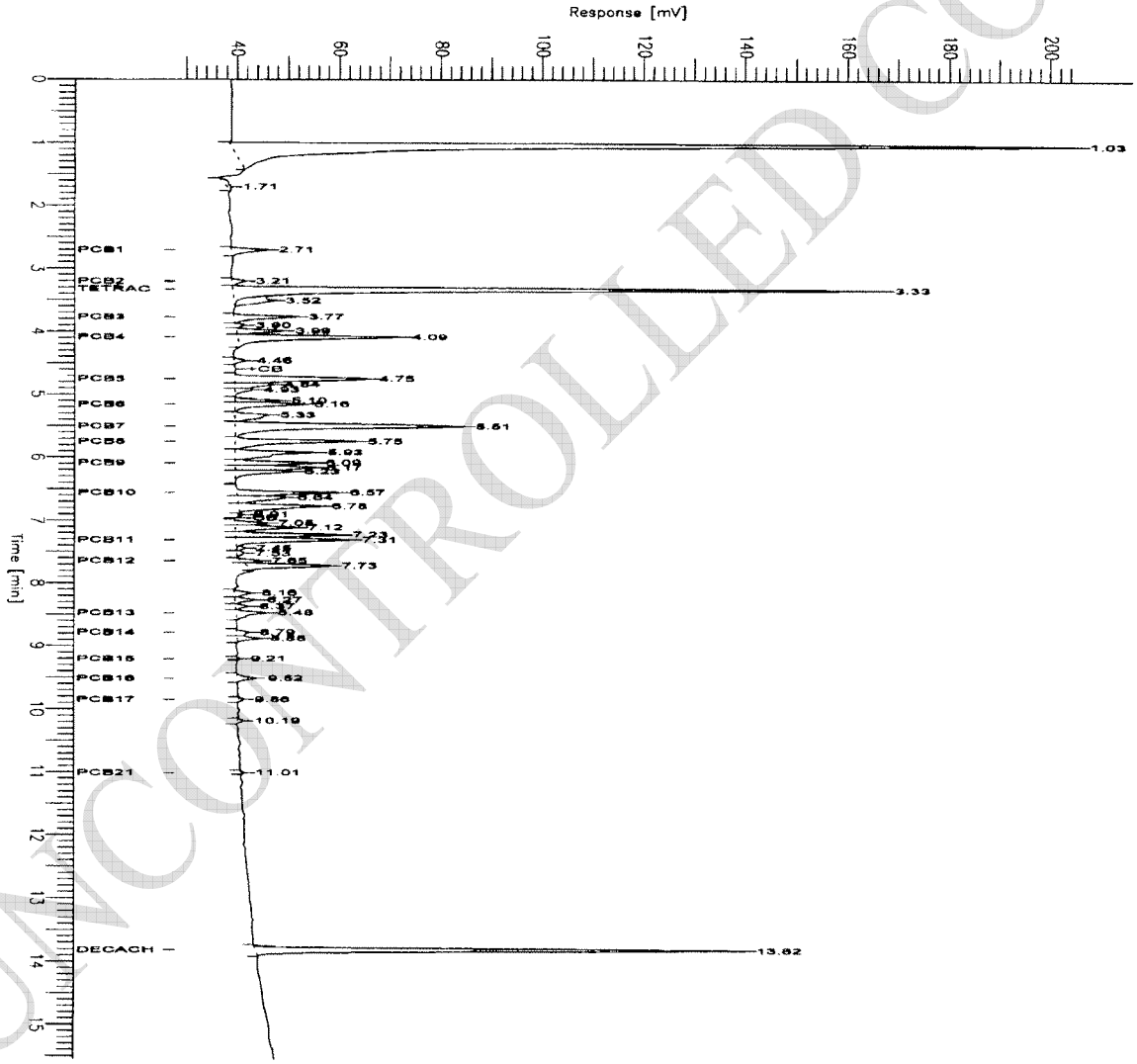
UNCONTROLLED COPY

Attachment 21.14
1232 on RTX CLP2 Column

Chromatogram

Sample Name : 1232
FileName : C:\TC4\GC144\B90_220.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 28 mV

Sample #: 220 Page 1 of 1
Date : 4/29/04 14:55
Time of Injection: 4/19/04 11:47
Low Point : 28.04 mV High Point : 205.58 mV
Plot Scale: 177.5 mV



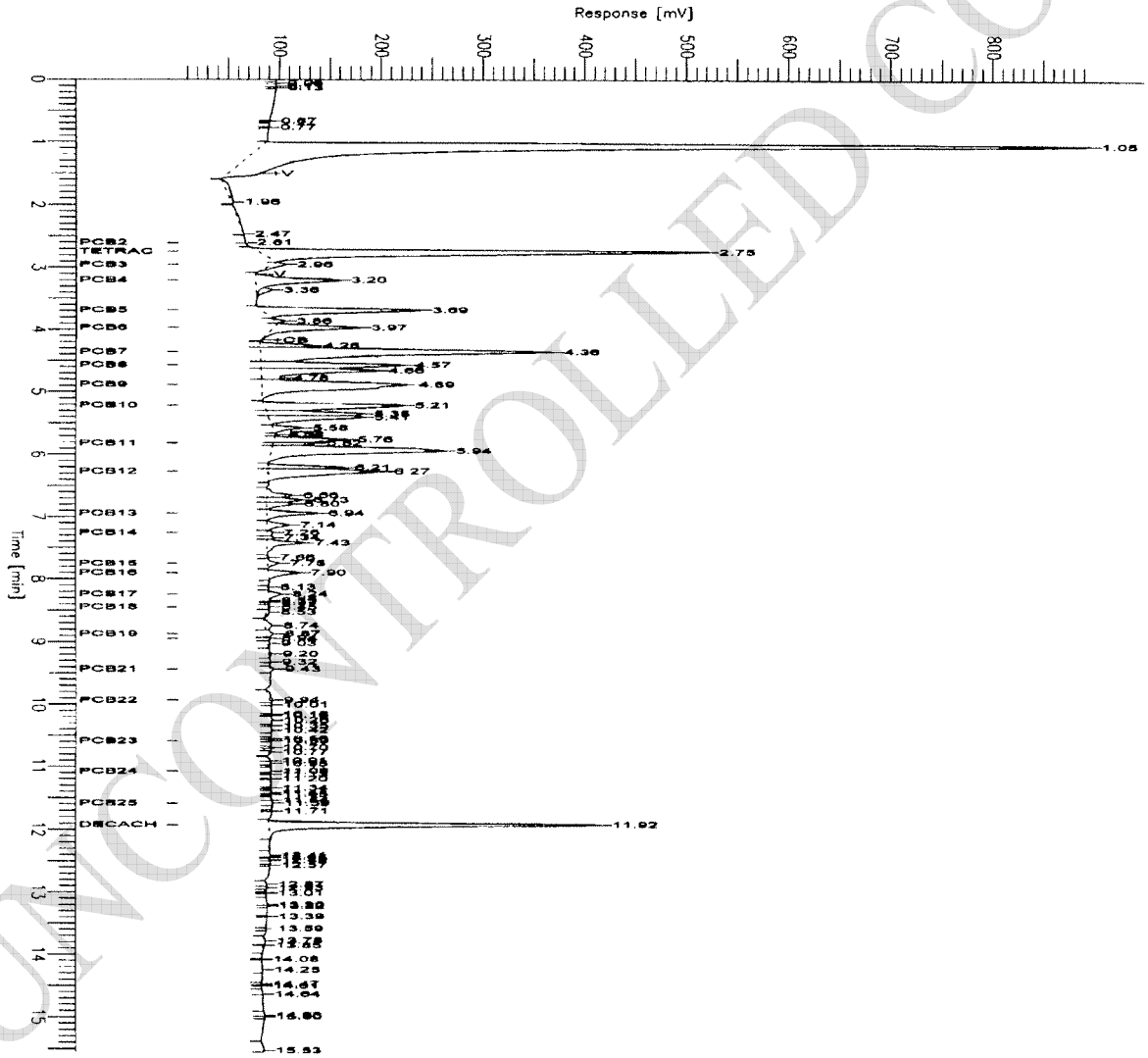
UNCONTROLLED COPY

Attachment 21.15
1242 on RTX CLP Column

Chromatogram

Sample Name : 1242
FileName : C:\TC4\GC144\A90_221.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 0 mV

Sample #: 221 Page 1 of 1
Date : 4/29/04 14:56
Time of Injection: 4/19/04 12:05
Low Point : 0.42 mV High Point : 895.90 mV
Plot Scale: 895.5 mV



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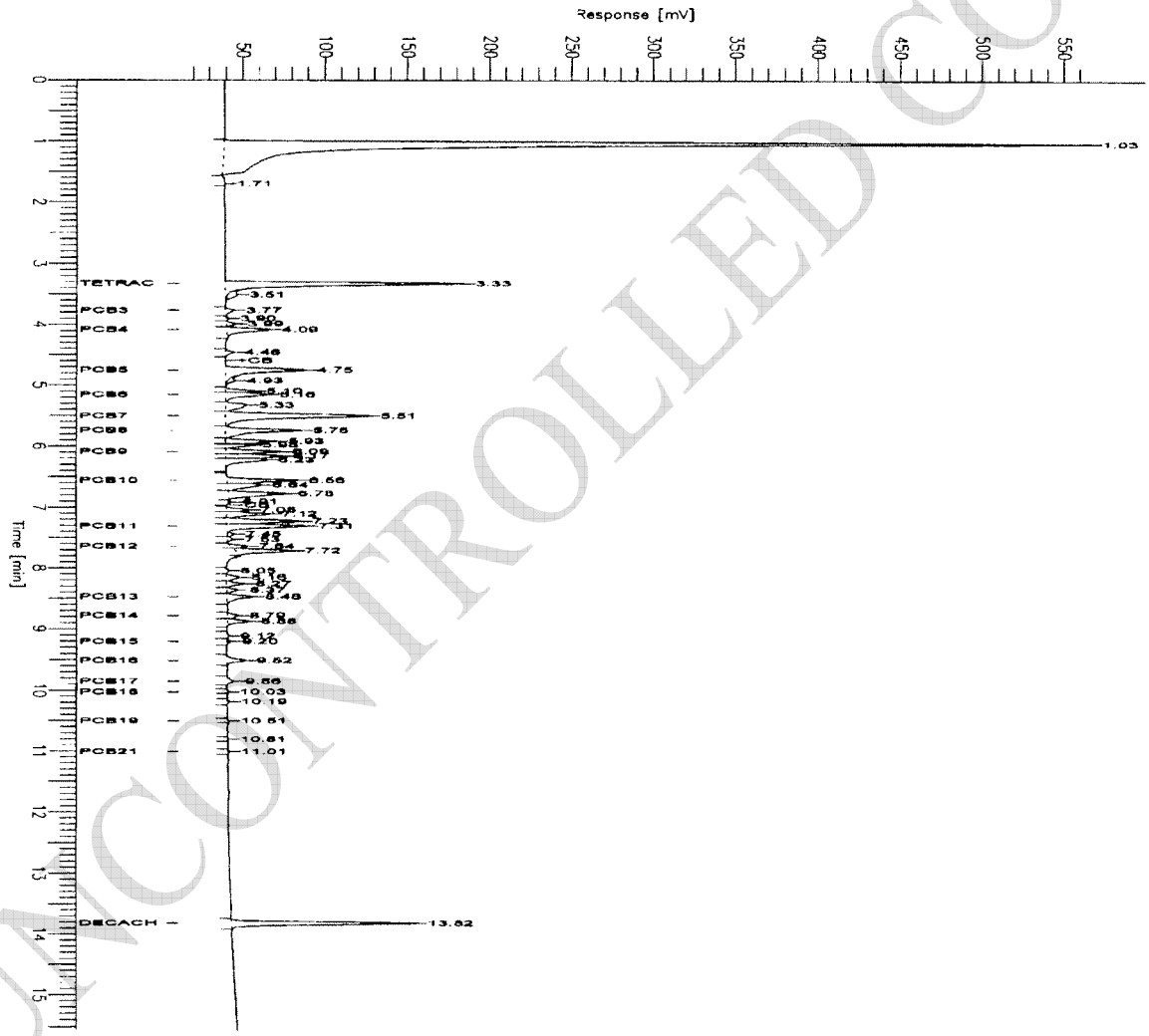
Approved By: 8-21-09 **QA Officer** **Approved By:** 8/21/09 **Area Supervisor**

Attachment 21.16
1242 on RTX CLP2 Column

Chromatogram

Sample Name : 1242
FileName : C:\TC4\GC144\B90_221.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 11 mV

Sample #: 221 Page 1 of 1
Date : 4/29/04 14:55
Time of Injection: 4/19/04 12:05
Low Point : 11.04 mV High Point : 566.75 mV
Plot Scale: 555.7 mV



UNCONTROLLED COPY

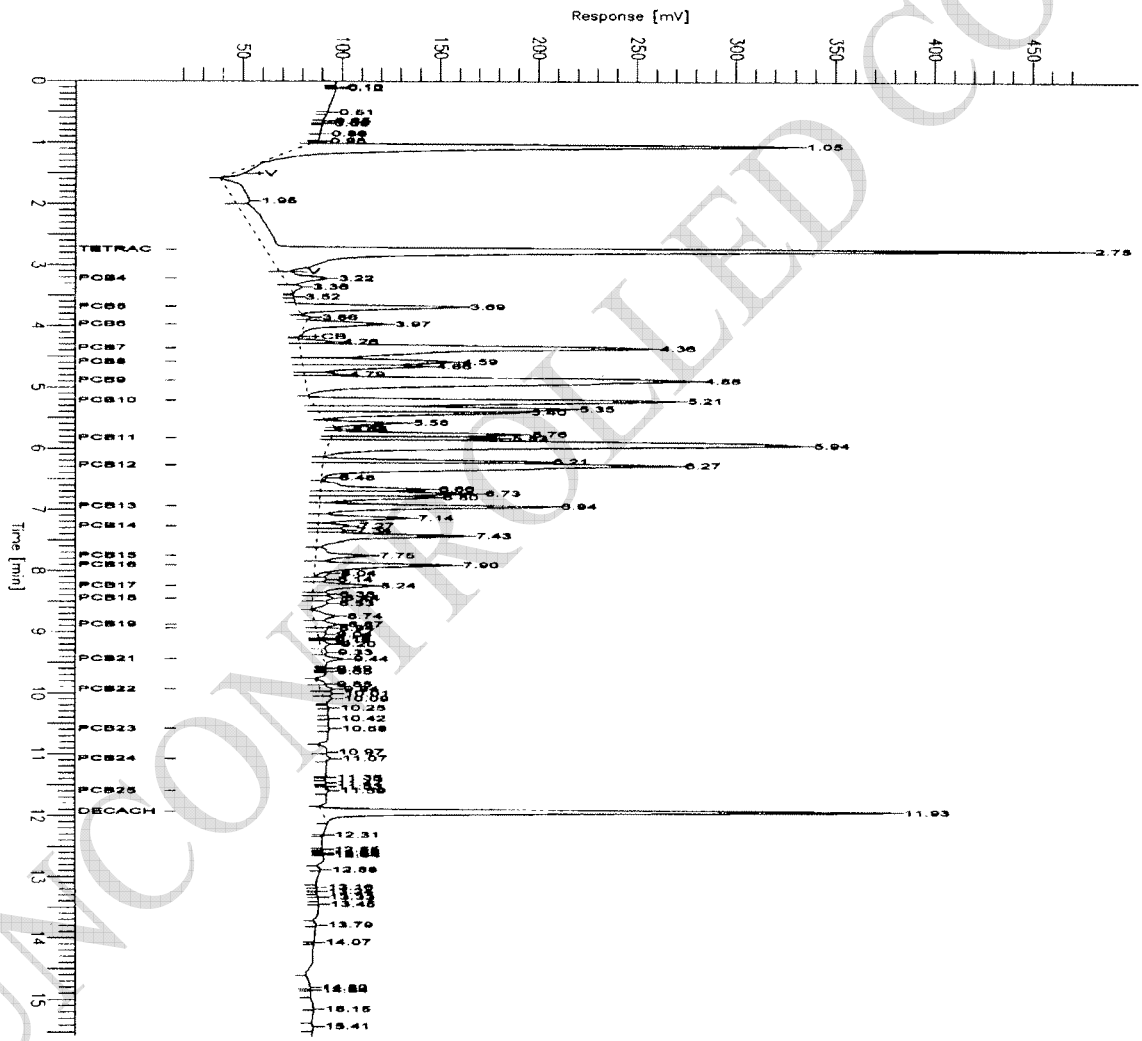
Approved By: 8-21-09 **Approved By:** 8/21/09
QA Officer **Area Supervisor**

Attachment 21.17
1248 on RTX CLP Column

Chromatogram

Sample Name : 1248
FileName : C:\TC4\GC144\A90_222.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 17 mV

Sample #: 222 Page 1 of 1
Date : 4/29/04 14:56
Time of Injection: 4/19/04 12:23
Low Point : 16.61 mV High Point : 476.49 mV
Plot Scale: 459.9 mV

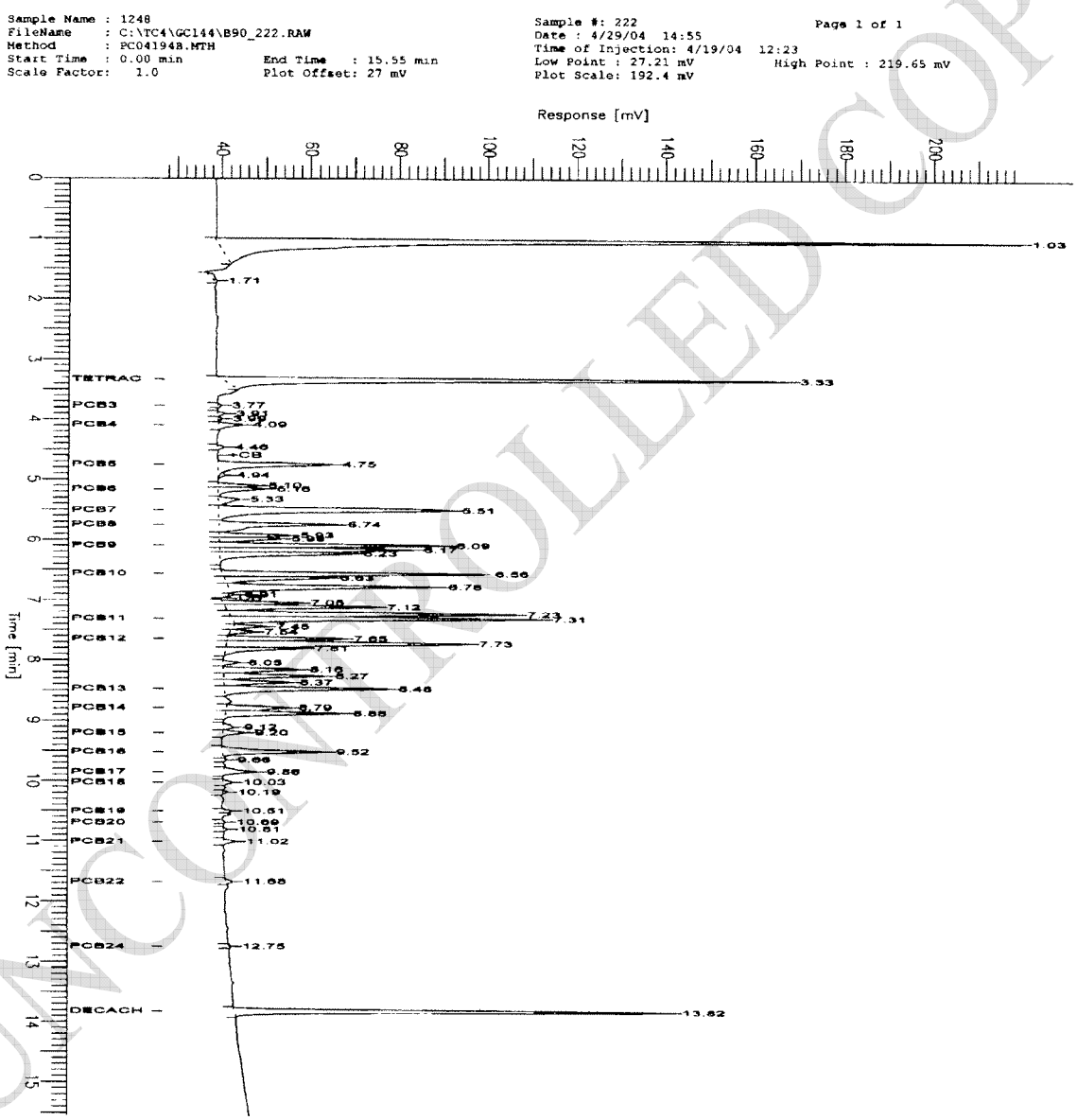


UNCONTROLLED COPY

Approved By: 8-21-09 **QA Officer** **Approved By:** 8/21/09 **Area Supervisor**

Attachment 21.18
1248 on RTX CLP2 Column

Chromatogram



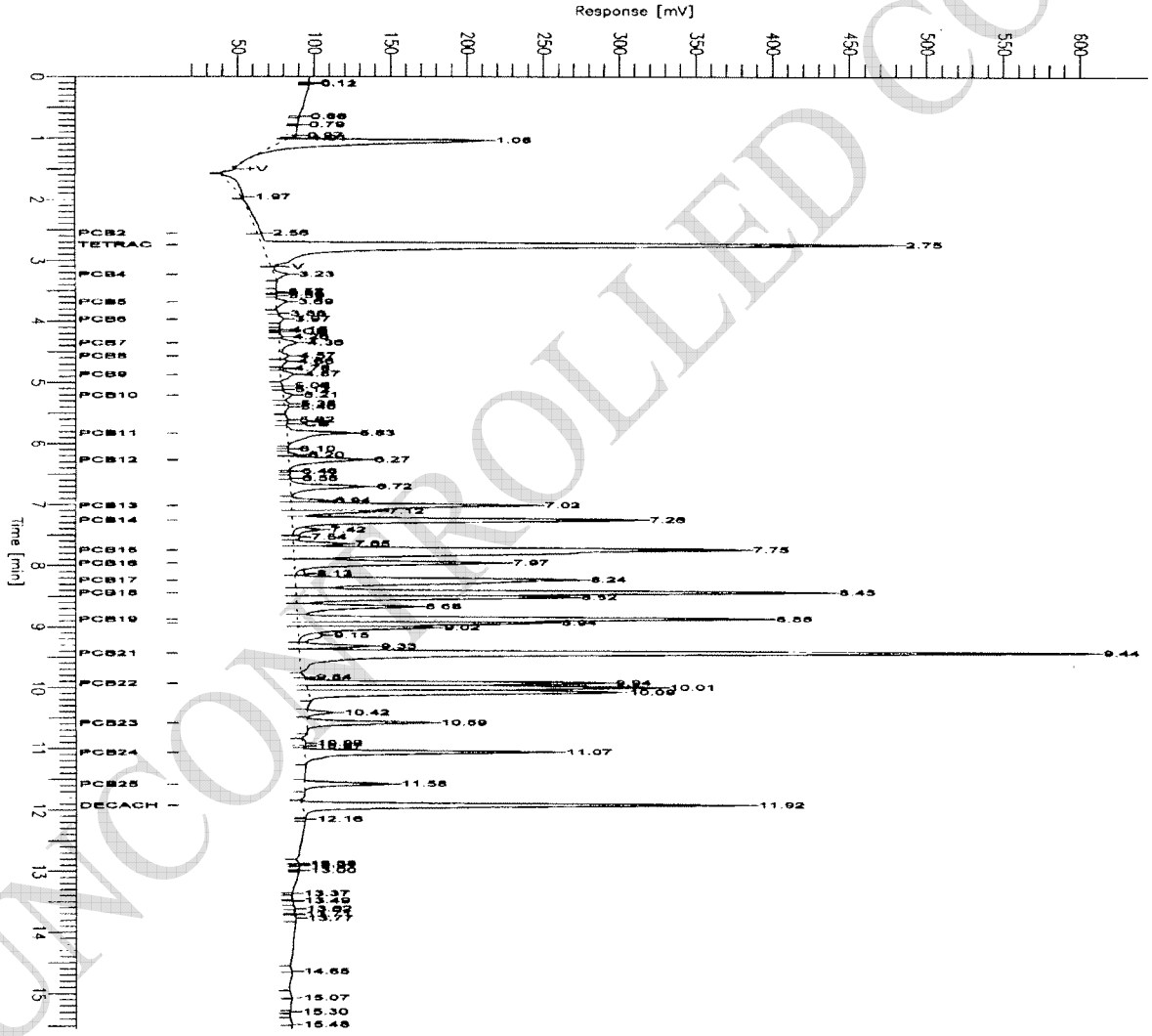
UNCONTROLLED COPY

Attachment 21.19
1262 on RTX CLP Column

Chromatogram

Sample Name : 1262
FileName : C:\TC4\GC144\A90_224.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 11 mV

Sample #: 224 Page 1 of 1
Date : 4/29/04 14:57
Time of Injection: 4/19/04 12:59
Low Point : 10.71 mV High Point : 608.82 mV
Plot Scale: 598.1 mV



UNCONTROLLED COPY

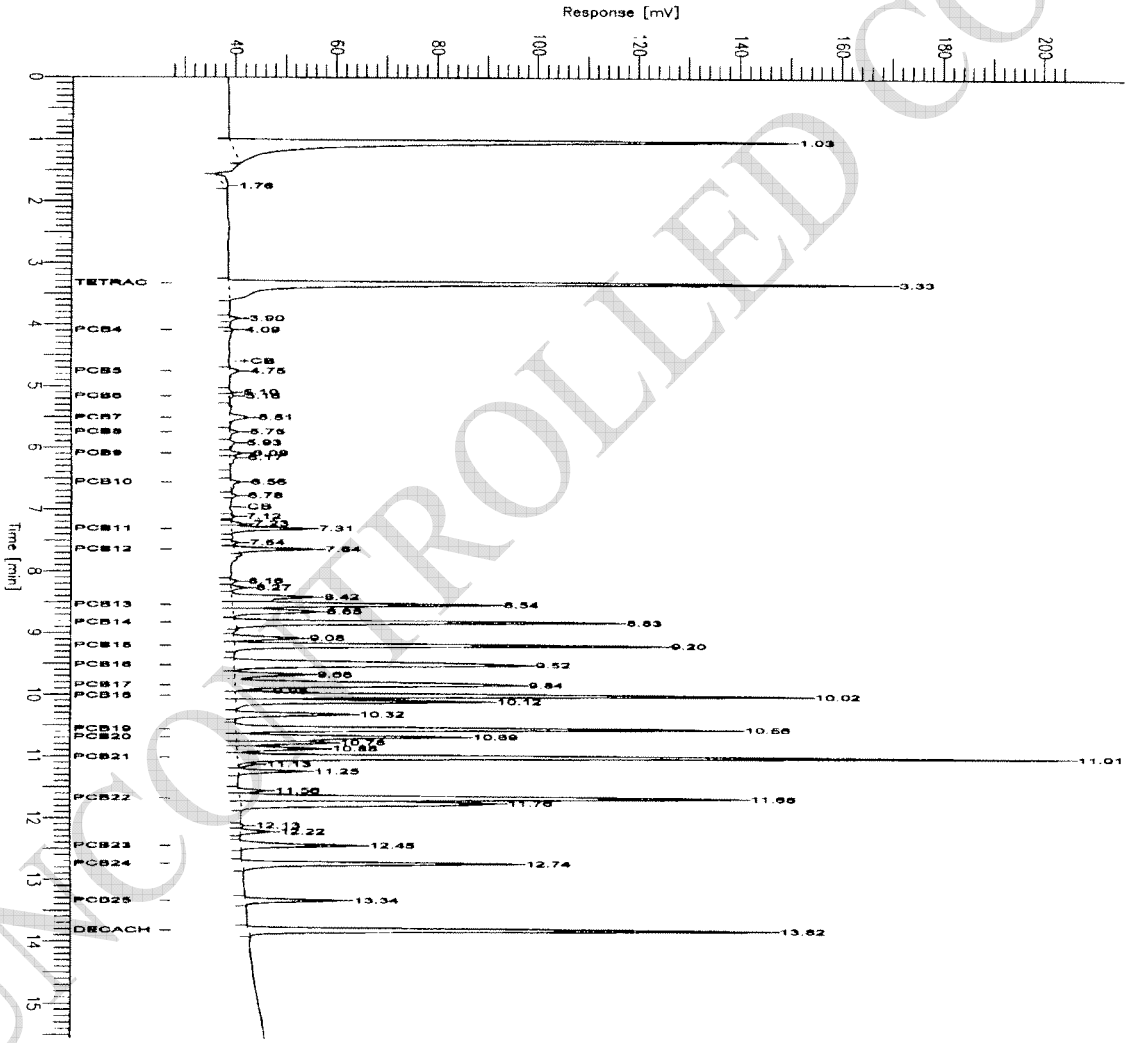
Approved By: 8-21-09 **Approved By:** 8/21/09
QA Officer **Area Supervisor**

Attachment 21.20
1262 on RTX CLP2 Column

Chromatogram

Sample Name : 1262
FileName : C:\TC4\GC144\B90_224.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 28 mV

Sample #: 224 Page 1 of 1
Date : 4/29/04 14:55
Time of Injection: 4/19/04 12:59
Low Point : 27.59 mV High Point : 205.23 mV
Plot Scale: 177.6 mV



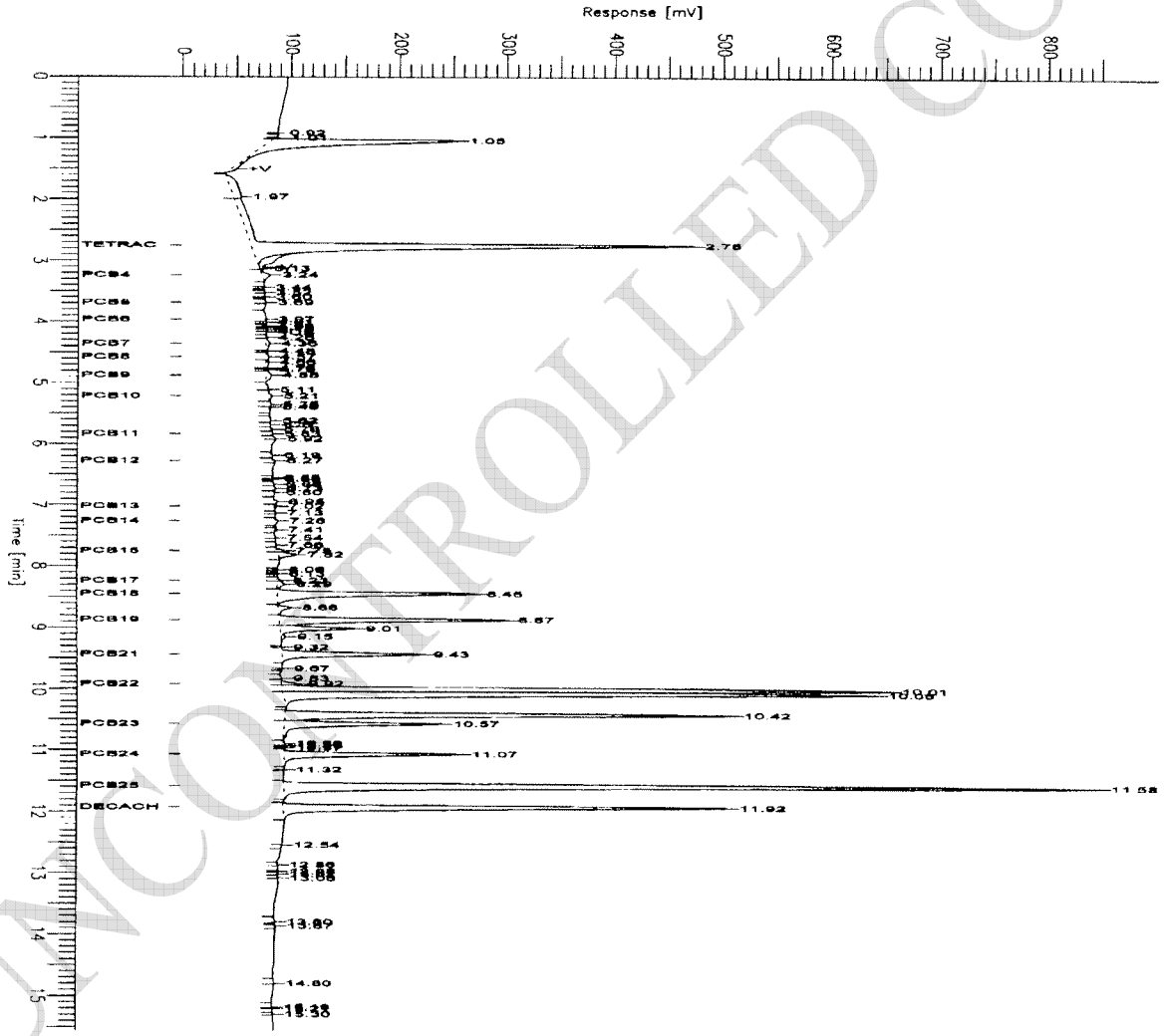
UNCONTROLLED COPY

Attachment 21.21
1268 on RTX CLP Column

Chromatogram

Sample Name : 1268
FileName : C:\TC4\GC144\A90_225.RAW
Method : PC04194A.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: -1 mV

Sample #: 225 Page 1 of 1
Date : 4/29/04 14:57
Time of Injection: 4/19/04 13:17
Low Point : -1.35 mV High Point : 850.53 mV
Plot Scale: 851.9 mV



UNCONTROLLED COPY

Approved By: 8-21-09 **QA Officer** **Approved By:** 8/21/09 **Area Supervisor**

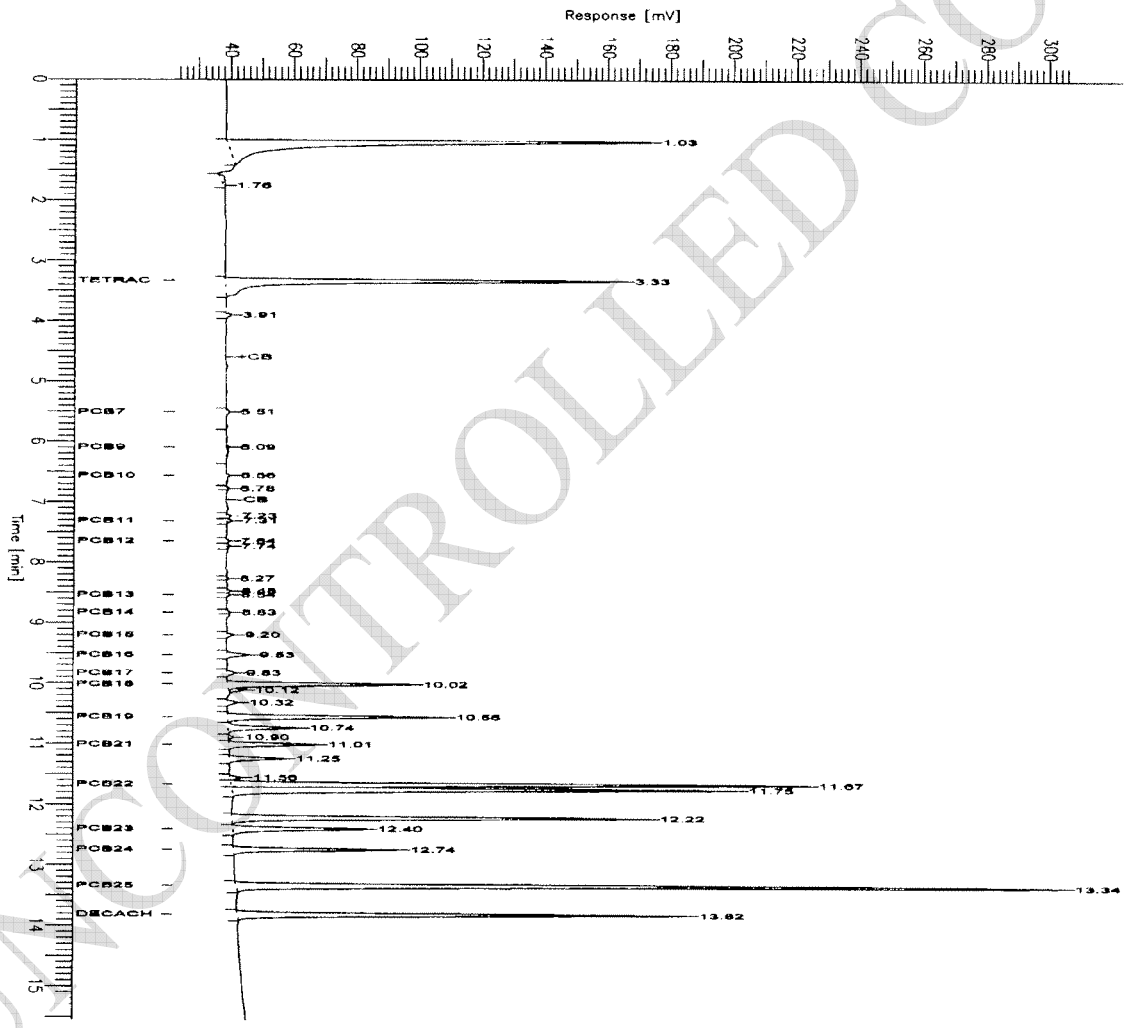
Attachment 21.22
1268 on RTX CLP2 Column

Chromatogram

Sample Name : 1268
FileName : C:\TC4\GC144\B90_225.RAW
Method : PC04194B.MTH
Start Time : 0.00 min End Time : 15.55 min
Scale Factor: 1.0 Plot Offset: 23 mV

Sample #: 225
Date : 4/29/04 14:55
Time of Injection: 4/19/04 13:17
Low Point : 22.56 mV High Point : 306.40 mV
Plot Scale: 283.8 mV

Page 1 of 1



UNCONTROLLED COPY

Approved By: AD 8-21-09 **Approved By:** AM 8/21/09
QA Officer **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Base/Neutral/Acid Compounds by Gas Chromatography/Mass Spectrometry

EPA Method 625
SW-846 Method 8270C

APPROVALS:

Operations Manager: _____ Date: 1/23/09
Jeff P. Glaser

QA Officer: _____ Date: 1-22-09
Tom C. Boocher

Area Supervisor: _____ Date: 1/23/09
Janet M. Kudirka

Procedure Number: GR-04-103
Revision Number: 5.5

Date Initiated: 12/9/98
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Janet M. Kudirka
Total Number of Pages: 46

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is used to determine the concentration of semi-volatile organic compounds in extracts of various solid waste matrices, soils, groundwaters, and wastewaters.

1.2 This procedure is used to quantify most basic, neutral, and acid extractable organics that are soluble in methylene chloride, and capable of elution without derivatization as sharp peaks from a gas chromatograph, fused-silica capillary column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. Refer to Table 1 for routine compounds, retention times, primary and secondary quantitation ions, internal standards, and surrogates. Table 1A lists additional analytes that are amenable to this procedure.

1.3 The following compounds may require special treatment. Benzidine can be subject to oxidative loss during solvent concentration. The chromatography must be monitored for peak broadening and tailing. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction with acetone, and photochemical decomposition. N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. 1,2-diphenylhydrazine also decomposes in the injection port into azobenzene. Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 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 materials.

1.4 Default reporting limits for clean extracted samples using 1000 mL or 30 g concentrated to 10 mL and analyzed by this procedure are listed in Tables 2 and 2A. Reporting limits will be proportionately higher depending upon the sample matrix, the preparation technique and extract dilutions. If a client requires lower reporting limits than those in Tables 2 and 2A, the final extract volume may be concentrated to 1.0 mL

1.5 This procedure is restricted to use by or under the supervision of analysts experienced in gas chromatography/mass spectrometry, and skilled in mass spectra interpretation. Each analyst must demonstrate the ability to generate acceptable results.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996

2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

2.3 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit

3.0 SUMMARY OF PROCEDURE

3.1 Before analysis, samples must be extracted using appropriate sample preparation and cleanup techniques. Waters will be extracted using TriMatrix SOP GR-09-101. Soils will be extracted using TriMatrix SOP GR-09-103. Extracts are analyzed by injection into a gas chromatograph, with mass spectrometric identification and detection of compounds. Quantitation is made by comparison with standards.

3.2 Wastewater sample analysis for NPDES permits under the Clean Water Act follows USEPA Method 625, requiring the following modifications to this procedure.

3.2.1 DFTPP tunes and continuing calibration standards are analyzed once every 24 hours instead of every 12.

3.2.2 A 3-point calibration may be used in place of a 6-point calibration. Standard practice, however will generally be to quantify using the 6-point calibration. System Performance Check Compounds (SPCCs) and Calibration Check Compounds (CCCs) are not used. Instead the response factors (RF) for every compound of interest must have <35% RSD to use the average RF for quantitation. If these criteria are not met, a new calibration curve must be constructed.

3.2.3 The continuing calibration standard does not use SPCCs and CCCs. The RF for compounds of interest in the continuing calibration standard that is on the 625 list must be ≤20% difference when compared against the calibration. If not, a new continuing calibration standard will be run with acceptable percent differences, or a new 3-point or 6-point calibration will be run.

3.2.4 There are no criteria for internal standard areas or retention times in Method 625. Method 8270C criteria will be followed.

3.2.5 Every compound of interest must be in matrix spikes and Laboratory Fortified Blanks, not just the limited list specified in Method 8270C. Laboratory generated windows for surrogate and spike recoveries are calculated as specified in this procedure.

4.0 PARAMETER OR COMPOUND LIST

4.1 Analytes for analysis by Method 8270C or Method 625 are listed in Table 1 and 1A. Other analytes may be determined providing a demonstration of capability study is successfully completed. In addition, QC criteria described for Table 1 and 1A compounds must be considered the minimum acceptable for additional analytes.

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-09-101, *The Extraction of BNAs in Water*, latest revision

5.2 TriMatrix Laboratories SOP GR-09-103, *Extraction of Semi-Volatile BNAs in Soil, Sediment and Sludge*, latest revision

5.3 TriMatrix Laboratories SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision

5.4 TriMatrix Laboratories SOP GR-09-104, *Waste Dilution of BNAs,* latest revision

5.5 TriMatrix Laboratories SOP GR-15-102, *Waste Disposal,* latest revision

5.6 TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision

5.7 TriMatrix Laboratories SOP GR-10-123, *ELEMENT Data Transfer and Revies,* latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware, leading to discrete artifacts and/or elevated baselines in the total ion current profiles. Do not use plastic materials, including polypropylene squirt bottles. Phthalate esters leaching from plastics can result in false positives. All materials used in extraction and analysis must routinely be demonstrated to be interference-free by running method performance blanks. Rinse all washed glassware with pesticide grade or better methylene chloride immediately before use. Use only high purity reagents (reagent grate or better) and solvents (pesticide grade or better). Solvents used for extraction and analysis must be tested prior to use. Once they are proven to be contaminant-free, large lots are sequestered from the vendor to ensure purity.

6.2 Carryover can occur whenever highly concentrated samples precede the analysis of low or non-detect samples. To reduce carryover, autosampler syringes are rinsed between extract injections. Whenever possible, the analyst should analyze a reagent blank immediately after a high level sample. If carryover is suspected, all affected samples must be re-analyzed.

6.3 Corrective actions for interference and contamination are outlined in TriMatrix SOP GR-04-101, *Semivolatile Laboratory Mass Spectrometry Corrective Actions*, latest revision.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the Laboratory Safety Manual and Chemical Hygiene Plan. Safety glasses are mandatory in all laboratory areas, except when entering data at computer terminals.

7.2 Methylene chloride and most target analytes, surrogates, and spikes are highly toxic and suspected carcinogens. Every precaution must be taken to avoid dermal contact, ingestion and inhalation. Wear appropriate protective clothing and disposable gloves when handling any of the above compounds. Prepare standards and extracts for analysis under a fume hood.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve the reporting limits in Tables 2 and 2A, a minimum 30 g of soil or 1000 mL of aqueous sample must be collected and extracted. Additional sample volume is necessary if matrix spikes are required.

8.2 Samples should be collected in amber glass jars, with PTFE-lined screw-cap lids. These jars must meet or exceed current USEPA cleanliness standards. Plastic containers and/or plastic-lined lids must NOT be used due to potential phthalate ester contamination.

8.3 Samples must be stored away from light, at 4° C ± 2° C. Store extracts away from light and ≤ -10° C.

8.4 Analysis must be performed within 40 days of sample extraction.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Gas chromatograph/mass spectrometer system – this procedure is applicable to either of the systems described in 9.1.1 or 9.1.2.

9.1.1 Varian Saturn II ion trap mass spectrometer is set up to scan from 40-450 mass units twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The spectrometer produces a mass spectrum that meets all criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

9.1.1.1 Ion Trap operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-450 amu
- Scan time: 820 amu/second = 2 scan/second
- Source temperature: 190° C
- Transfer line temperature: 250° C

9.1.1.2 Varian 3400 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C until benzo[g,h,i]perylene has eluted.
- SPI Injector Temperature Program: 45° C for 0.5 minute, then to 300° C at 100°C/minute.
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 6 psi (~ 1 to 2 mL/minute)

9.1.2 The Agilent 5973 Mass Selective Detector (MSD) is set up to scan from 40-550 mass units at least twice per second, using 70 volts (nominal) electron energy in the electron impact mode. The detector produces a mass spectrum that meets all the criteria in Table 3, when 5.0 ng of decafluorotriphenylphosphine (DFTPP) is injected onto the analytical column.

9.1.2.1 MS operating conditions:

- Electron energy: 70 volts (nominal)
- Mass range: 40-550 amu
- Scan time: sampling rate of 3 = 2.89 scan/second
- Source temperature: 230° C
- Transfer line temperature: 280° C

9.1.2.2 Agilent 6890 GC operating conditions:

- Column Temperature Program: 45° C for 1.5 minutes, then to 200° C at 10°C/minute, then to 315° C at 25° C/minute, hold at 315° C for 4.0 minutes
- Sample volume: 1 uL
- Carrier Gas: ultra high purity helium at 1.0 mL/minute in constant flow mode

9.1.3 Injector

9.1.3.1 A Septum Programmable Injector (SPI), temperature programmed. The injector is capable of sub-ambient initial injector temperatures, followed by rapid heating up to 315° C, and is used for Ion Trap applications.

9.1.3.2 A Split/splitless injector in the splitless mode held at 250° C, is used for the Mass Selective Detector (MSD).

9.1.4 Fused Silica Capillary Columns

9.1.4.1 A 30 m x 0.32mm i.d. 1.0 um film thickness silicon coated DB-5MS or equivalent, is used for the Ion Trap.

9.1.4.2 A 30 m x 0.25 mm, i.d. 0.25 um film thickness silicon coated DB-5MS or equivalent, is used for the Mass Selective Detector (MSD).

9.1.4.3 A 20 m x 0.15 mm. i.d. 0.15 um film thickness silicon coated factorFour VF-5MS is also used for the Mass Selective Detector (MSD).

9.1.5 Data system:

9.1.5.1 A multi-tasking personal computer, capable of the continuous acquisition and storage on magnetic tape or compact disk, of all mass spectra obtained throughout a chromatographic program.

9.1.5.2 The software can search acquired GC/MS data files, for specific mass ions, and can plot ion abundances versus time or scan number. This type of plot is an Extracted

Ion Current Profile (EICP). The software integrates abundances in any EICP between specified time or scan number limits. The 1992 (Ion Trap) or 1998 (MSD) version of the NIST mass spectral library is referenced for identification of unknowns.

9.1.6 Autosampler: A Leap A200SE or equivalent, programmed for 1.0 uL injections with multiple rinses before and after injection.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 When chromatography degrades or sensitivity decreases, routine maintenance consists of cleaning/changing the injection port liner, clipping capillary column, changing injection port septum, and cleaning the source.

10.2 The rough pump oils must be replaced at least annually.

10.3 Check the MSD diffusion pump oil every six months and refill, if necessary.

10.4 Check the Ion Trap turbo pump wick annually and re-oil or replace, if necessary.

10.5 If column performance deteriorates, performance may be brought back by repeated injections of Toluene overnight. If this is ineffective, the column will need replaced.

11.0 CHEMICALS AND REAGENTS

11.1 All chemicals and reagents used must be reagent grade or better, and must be determined free of interferences.

11.2 Acetone, hexane, methylene chloride, and toluene must be pesticide grade or better, and must be determined free of interferences.

12.0 STANDARDS PREPARATION

12.1 Stock calibration standard solutions: Stock solutions are prepared from pure standard materials or purchased as certified solutions.

12.1.1 Commercially purchased stock solutions are used to prepare the majority of working standards. The purchased concentration for stock mixes is typically 2000 mg/L. Solutions arrive at the laboratory sealed in various glass ampules, which are combined during preparation.

12.1.1.1 Stock standards are combined with the surrogate currently being used during sample extraction, to create a 100 mg/L calibration standard in methylene chloride.

12.1.1.2 Diluting from the 100 mg/L standard, make up the 8-point initial calibration at concentrations of 25, 20, 15, 10, 5.0, 2.0, 1.0, and 0.5 mg/L. Due to low response

in the lowest calibration point, prepare extra concentrations of 2,4-dinitrophenol, 4-nitrophenol, benzoic acid and benzidine for a final concentration of 1.0 mg/L to 50 mg/L.

12.1.2 Stock standard solutions from neat compounds are prepared by weighing out each analyte to the nearest tenth of a milligram. The material is dissolved in methylene chloride or other suitable solvent and diluted to volume in a volumetric flask. If compound purity is 96% or greater, the weighed mass is used without correction to calculate stock standard concentration.

12.1.3 Transfer stock standard solutions to screw-cap vials with PTFE-lined lids and store below -5° C, protected from light. Stock standard solutions must be monitored for degradation or evaporation.

12.1.4 All stock standard mixes must be replaced annually or sooner if comparison with quality control checks indicates a problem. Working calibration standards must be replaced every six months or sooner if comparison with quality control checks indicates a problem. Continuing calibration standards must be prepared weekly.

12.2 Internal standard solutions: The six internal standards used are 1,4-dichlorobenzene-d_4, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, naphthalene-d_8, and perylene-d_{12}. The stock standard is prepared in a volumetric flask at 1000 ug/mL by dissolving 100 mg of each compound with a small volume of methylene chloride then diluting to 100 mL. Each 1.0 mL extract analyzed must be spiked with 5.0 uL of the internal standard solution. The resulting concentration of internal standards in extracts is 5.0 ug/mL.

12.3 GC/MS tuning standard: The standard is used for tuning the mass spectrometers and contains 5.0 ng/uL of decafluorotriphenylphosphine (DFTPP), 50 ng/uL of pentachlorophenol, 50 ng/uL of benzidine, and 50 ng/uL of 4,4'-DDT, in methylene chloride. Stock standards for the tuning mixture are purchased from a vendor at 1000 mg/L. Appropriate dilutions for the working concentrations are prepared from these standards. The tuning standard also verifies injection port inertness and monitors GC column performance.

12.4 Surrogate standards: Surrogate solution contains o-terphenyl, 2-fluorophenol, phenol-d6, 2,4,6-tribromophenol, nitrobenzene-d5, and 2-fluorobiphenyl. These are made as follows.

12.4.1 Acid and base compound surrogate solutions are made separately from neat materials:

12.4.1.1 Add 0.5 g of each base-neutral surrogate to a 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.

12.4.1.2 Add 1.0 g of each acid surrogate to another 250 mL volumetric flask, half-filled with methanol. Swirl, shake or sonicate to dissolve, then dilute to volume with methanol.

12.4.1.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. The finished solution must be checked for accuracy by analyzing on the GC/MS. Once approved, the prep lab and the

mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.

12.4.1.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the organic prep laboratory refrigerator at 4 ± 2° C.

Surrogate	Mass (g)	Dilution Volume (mL)	Stock Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d_5	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d_6	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

12.4.2 The volume spiked is 100 uL for each 1.0 mL of extract volume, resulting in a concentration of 10 ng/uL of base/neutral surrogates and 20 ng/uL acids.

12.5 Spiking standards applicable to Laboratory Blank Spikes (BS) and Matrix Spikes (MS/MSD): The base/neutral spike solution contains acenaphthene, 1,4-dichlorobenzene, 2,4,-dinitrotoluene, naphthalene, N-nitrosodi-n-propylamine, pyrene, and 1,2,4-trichlorobenzene. The acid spike contains 4-chloro-3-methyl phenol, 2-chlorophenol, 4-nitrophenol, pentachlorophenol, and phenol. The spikes are made up in methanol at 100 ug/mL for the base/neutrals and 200 ug/mL for the acids, as detailed in the appropriate extraction procedure. The volume spiked is 100 uL per 1.0 mL of extract, resulting in a concentration of 10 ng/uL for base/neutrals, and 20 ng/uL for acids.

NOTE: For all State of Wisconsin samples and when other projects require it, every analyte of interest must be spiked into the BS and MS/MSDs or only the BS, as applicable. All analytes must be spiked at 10 ng/uL before extraction.

13.0 SAMPLE PREPARATION

13.1 For sample extractions, refer to the appropriate procedures in section 5.0.

13.2 All samples must be spiked with surrogates before extracting (Refer to section 12.4).

13.3 All extracts must be spiked with internal standards, immediately prior to injection (Refer to section 12.2).

13.4 Extracts that are dark in color or thick and viscous will be diluted at least 1:10 prior to analysis.

14.0 CALIBRATION PROCEDURES

14.1 Set up instrumentation as specified in section 9.1.1 and 9.1.2.

14.2 All samples and standards are introduced into the GC/MS, by 1.0 uL direct injection. All injections are performed using an autosampler.

14.3 Each GC/MS system is hardware-tuned to meet Table 3 or Table 3A criteria for a 5.0 ng, 1.0 uL DFTPP injection. No analysis may begin until these criteria are met. Mass spectra are taken across the 198 ion apex. Background subtraction is designed only to eliminate column bleed or instrument background ions.

14.3.1 The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and/or DDD must not exceed 20%. Calculate degradation as follows:

$$\%\ \text{degradation} = \frac{\text{Area}_{DDE} + \text{Area}_{DDD}}{\text{Area}_{DDE} + \text{Area}_{DDD} + \text{Area}_{DDT}} \times 100$$

14.3.2 The degradation result must be recorded in the daily instrument run logbook.

14.3.3 Pentachlorophenol and benzidine must be present at their normal responses, with minimal peak tailing. If peak tailing is suspect, calculate a tailing factor using the pentachlorophenol total ion chromatogram. To determine if corrective action is required, follow Figure 1 instructions. A tailing factor of 1 to 2 is considered acceptable, a tailing factor of 2 to 4 is considered the warning limit, and a tailing factor of 4 or higher is considered unacceptable, requiring corrective action. Corrective action for degradation and/or poor chromatography can include cleaning the injection port and clipping the first 6 to 12 cm off the capillary column.

14.4 All analytes must be quantified using the method of internal standards. Analytes and their corresponding internal standards are provided in Tables 1 and 1A. For analytes not listed, the corresponding internal standard must be the one eluting nearest the unlisted analyte. Primary quantitation ions are also provided in Tables 1 and 1A. A secondary ion may be used for quantitation, only if interferences are noted. If a secondary ion is used, the compound RF must be recalculated before quantitation.

14.5 Initial Calibration:

14.5.1 Calibration standards: The initial calibration for all analytes of interest must contain a minimum of five points (three for method 625). Table 1 standards calibration is typically run at 0.5, 1.0, 2.0, 5.0, 10, 15, 20, 25 ug/mL. The 25 ug/mL standard may be omitted. However, omitting lowers the linear calibration range. Each 1.0 mL calibration standard aliquot must be spiked with 5.0 uL of internal standard solution, immediately prior to analysis. Table 1A and other analytes, are run at the appropriate concentration, based on response. The lowest concentration of the initial calibration must be at the reporting limit.

14.5.2 Injections of 1.0 uL for each calibration standard, including surrogates and internal standards are analyzed. Figures 2 and 2A show chromatograms of a continuing calibration standard. A Response Factor (RF) is calculated for each compound using the Table 1 or 1A primary ions. Response factors are calculated as follows:

$$RF = \frac{A_X \times C_{IS}}{A_{IS} \times C_X}$$

where:

A_x = Area of the primary ion for the compound being measured
A_{is} = Area of the primary ion for the specific internal standard
C_x = Concentration of the compound being measured (ng/uL)
C_{is} = Concentration of the specific internal standard (ng/uL)

14.5.3 A system performance check must be performed to ensure that average RFs are acceptable for use in quantitation. System Performance Check Compounds (SPCCs) are listed in Table 4. The minimum acceptable average RF for SPCC compounds is 0.050. SPCCs typically have very low RFs (0.1to 0.2) and tend to decrease in response as the chromatographic system or the standards deteriorate. SPCCs are usually the first to show poor performance, and must meet the minimum requirement when the system is calibrated. The minimum recommended RF for all non-SPCC analytes of interest is 0.01, however, spectra quality and background (signal to noise) must also be considered.

14.5.4 Percent relative standard deviation (RSD) must be calculated for each compound using the following equation:

$$\%RSD = \frac{SD}{RF} \times 100$$

where:

SD = The standard deviation of the average response factor across the initial calibration curve.
RF = Average response factor across the initial calibration curve.

14.5.5 Percent RSD for each compound must be less than 15% to use an RF for quantitation, which assumes linearity through the origin. Percent RSD for each Table 5 calibration check compound (CCC) **must** be less than 30%. If percent RSD for any CCC is between 15% and 30%, a calibration curve must be constructed as outlined in 14.5.8. If percent RSD is **greater** than 30% for any CCC, the system is too unstable to proceed. Corrective action, including replacing the injection port liner, replacing or clipping the capillary column, and recalibration of the GC/MS system, must be initiated.

14.5.6 The relative retention time of each compound of interest in each calibration run must agree within 0.06 relative retention time units.

14.5.7 If percent RSD of any non-CCC compound is 15% or less, the response factor is assumed to be constant over the calibration range, and the average response factor can be used for quantitation.

14.5.8 If percent RSD for any compound is greater than 15%, a regression curve must be constructed by plotting area ratio ($Area_{analyte}/Area_{IS}$) against concentration. A first order or higher regression fit is used. The curve must not be forced through the origin:

14.5.8.1 Linear regression requires five calibration standards and is calculated as follows:

$$C_s = [((A_s * C_{is})/A_{is}) - b]/a$$

Where:
A_s = Area of the target analyte peak, in sample "s"
A_{is} = Area of the "is" internal standard peak
C_s = Concentration of the target analyte in calibration standard "s"
C_{is} = Concentration of the internal standard "is"
a = Slope of the curve (also called the coefficient of C)
b = The intercept

14.5.8.2 A quadratic (second order) fit requires six calibration standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s * C_{is})/A_{is}] = a * C_s^2 + b * C_s + c$$

Where:
c = the constant

14.5.8.3 A third order polynomial requires seven standards and a chromatographic curve fitting program, to solve the following:

$$[(A_s * C_{is})/A_{is}] = a * C_s^3 + b * C_s^2 + c * C_s + d$$

Where:
d = the constant

14.5.8.4 Analysts may select each analyte regression curve giving the least calibration error, as indicated by the correlation coefficient. To be acceptable, the correlation coefficient "r" for a first order regression must be 0.99 or higher. For second and third order regressions, the coefficient of determination (COD) "r^2" must be 0.99 or higher.

NOTE: Non-linear regression calibration must not be used as compensation for improper instrument maintenance. Non-linear regression for any analyte must not be employed on an instrument previously shown to **routinely** exhibit linearity.

14.5.9 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

14.5.9.1 A minimum of six calibration points are required to use second order regression curves. Five data points are needed for first order.

14.5.9.2 The lowest calibration point defines an analyte reporting limit. The low point can only be dropped provided required reporting limits are not affected.

14.5.9.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.

14.5.10 Run a second-source calibration standard containing all analytes at a mid-range concentration following the initial calibration. All analytes must have recoveries between 75-125% for the initial calibration to be considered valid. If these criteria are not met, analyze the second-source standard a second time. If the second analysis fails, take corrective action up to and including preparation and running of new initial calibration standard solutions. Repeat the second-source analysis for confirmation that corrective action was effective.

14.6 Continuing Calibration:

14.6.1 Prior to analysis of samples or standards, the DFTPP tuning standard is analyzed and evaluated, as specified in section 14.3. Acceptable DFTPP tuning must be demonstrated at the beginning of each 12-hour shift.

14.6.2 Immediately following every acceptable DFTPP analysis, a continuing calibration standard must be run at a mid-level concentration. For method 8270C analytes listed in Table 1, the concentration is 10 ug/mL. A continuing calibration standard contains all semi-volatile target analytes, including all surrogates and internal standards. Table 1A and other analytes, are run at the appropriate concentration, based on response. Each analyte RF is compared with the initial calibration average RF, from the same instrument.

14.6.3 System Performance Check Compounds (SPCC): A system performance check is made on every continuing calibration standard. All SPCC criteria must be met for a continuing calibration standard to pass. Table 4 lists SPCC compounds used for the continuing calibration. The minimum SPCC RF is 0.050. The recommended minimum RF for all non-SPCC compounds of interest is 0.01. However, spectra quality and background (signal to noise) must also be considered. If minimum response factors are not met for SPCCs, the system must be evaluated and corrective action taken, before analysis can continue. Possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front of the analytical column, and active sites in the column or chromatographic system.

14.6.4 Calibration Check Compounds (CCC): After the system performance check is met, Table 5 CCCs are used to check the initial calibration validity.

When average response factor is used for calibration, calculate percent difference between the initial calibration average RF and the continuing calibration RF:

$$\%\ Difference = \frac{RF_v - RF_m}{RF_m} \times 100$$

where:

RF_v = response factor from verification standard
RF_m = average calibration factor from initial calibration

Use percent drift to determine acceptance when calibrating using a regression fit model:

$$\%\ Drift = \frac{C_C - C_I}{C_I} \times 100$$

where:

C_I = theoretical CCC standard concentration
C_C = measured concentration using appropriate regression fit.

14.6.5 If percent difference or drift for every CCC is less than ±20%, the initial calibration remains valid. If any CCC compound differs from the initial calibration curve by greater than ±20%, corrective action must be taken. Problems similar to those listed under section 14.6.3 can also affect CCC results. If the problem source cannot be determined after corrective action has taken place, the calibration is considered out of control. At a minimum, the continuing calibration standard must be re-run. If necessary, a new calibration will be run. All CCC criteria must be met before sample analysis can continue. For non-CCC analytes, the percent difference or drift must be ≤25%, with the exception of poor performing analytes, which can have up to four outside of ≤25%, but must be ≤40%. Examples of poor performing analytes include: Chloro- and Nitro-anilines, Benzoic acid, Benzidine, Hexachlorocyclopentadiene, 4-Nitrophenol, and 2,4-Dinitrophenol. In addition, if response for any non-CCC compound is outside this criteria, but is recovering on the high side (response for the analyte is out of control high), then extracts with non-detect results for these compounds may still be used since there is no question of analyte sensitivity for the day. For method 625, all compounds must have a percent difference of less than ≤20%.

14.6.6 Internal standard responses and retention times in continuing calibration verification standards and samples must be evaluated in each run. If retention time for any internal standard varies by more than 30 seconds from the last calibration verification, the system must be inspected for malfunctions, and corrective action performed as necessary. If the EICP area for any internal standard varies by a factor of 2 (-50% to +100%) from the midpoint standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrective action performed as necessary. All sample extracts analyzed under either of these out of control conditions must be reanalyzed.

15.0 ANALYTICAL PROCEDURE

15.1 Every 12-hour shift **must** begin with the analysis of DFTPP, followed by a new calibration or a continuing calibration verification standard, then followed by an instrument blank.

15.2 The DFTPP tune **must** pass the tuning criteria in section 14.3 before subsequent analysis can begin. The 12-hour acquisition window **must** begin at the injection time of DFTPP. The entire 12-hour shift must be run under the same mass spectrometer conditions used for DFTPP. All samples must be run under the same gas chromatographic conditions as the initial calibration. Refer to sections 9.1.1 and 9.1.2 for recommended operating conditions.

15.3 Any calibrated compound **must** have an acceptable average Response Factor (RF) generated from the initial calibration curve, or be calibrated using a first or higher order regression curve. If Method 8270C is being utilized, the calibration **must** pass the SPCC and CCC criteria specified in section 14.5.

15.4 A continuing calibration standard is run on days an initial calibration is not. The continuing calibration standard **must** pass the criteria specified in section 14.6.

15.4.1 After the DFTPP and continuing calibration criteria are met, the analytical system **must** be demonstrated free from contamination by running an instrument blank before sample analysis during each 12-hour shift (Refer to section 18.3).

15.5 Once the above criteria are met, sample analysis may proceed for the remainder of the 12-hour shift.

15.6 If response for any analyte in the sample exceeds the initial calibration range, a dilution of the extract must be made. Attempt to dilute the sample only enough to bring the analyte(s) in question into the upper half of the calibration range. Additional internal standard must be added to the diluted extract to maintain the required 5.0 ng/uL internal standard concentration. The diluted extract is then reanalyzed.

15.7 A matrix spike, matrix spike duplicate, method preparation blank, and laboratory fortified blank **must** be run for each quality control batch to assess extraction efficiency. Up to a maximum of 20 samples/batch is allowed. It is preferred but not required that matrix quality control be analyzed with samples from the same extraction batch during one 12-hour shift. Additional quality control requirements are specified in section 18.0.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative analysis:

16.1.1 An analyte is identified by a mass spectra comparison between the sample and a standard of the suspected compound (standard reference spectrum). Standard reference spectra are obtained through analysis of the calibration. Two criteria must be satisfied to verify identification. First, a sample component must elute at the same GC relative retention time (RRT) as the standard compound. Secondly, the mass spectra of the sample component and standard compound must agree.

16.1.1.1 The sample component RRT must fall within ± 0.06 RRT units of the standard compound, in the daily continuing calibration standard. If interfering co-elution

prohibits accurate sample component RRT measurement from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles unique to the compound of interest.

16.1.1.2 All ions in standard mass spectra at a relative intensity greater than 10% are automatically checked by the software to be in a sample spectrum. The most abundant ion in standard mass spectra equals 100%. Relative ion intensities between standard and sample spectra, must agree within ± 30%. For example, an ion with 50% abundance in standard spectra, means the corresponding sample abundance must be between 20% and 80%.

16.1.2 For samples containing components not associated with calibration standards, a library search can be made for a tentative identification. Only after visual comparison of sample spectra with the nearest library searches must the analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description must be given to identify functional groups. TIC nomenclature by functional group is as follows:

Name	CAS #
Unknown Alcohol	xx-xx-xx
Unknown Freon	xx-xxx-xxxx
Unknown Ketone	xx-xxx-xx
Unknown Acid	xxx-xx-xxxx
Unknown Hydrocarbon	xxx-xxx
Unknown Glycol Ethers	xxx-xx-x
Unknown Substituted Benzenes	xxxxx
Hydrocarbons (sub. Benzenes)	xxxxx-x-x
Hydrocarbons, Total	xxxxxx

Guidelines for making tentative identification are:

16.1.2.1 Relative intensities of major ions in a reference spectrum (ions with >10% of the most abundant ion) should be present in the sample spectrum.

16.1.2.2 Relative intensities of major ions should agree within ± 20%. For example, an ion with 50% abundance in a standard spectrum corresponds to a sample ion abundance between 30% and 70%.

16.1.2.3 Molecular ions in the reference spectrum should be present in the sample spectrum.

16.1.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or co-eluting components.

16.1.2.5 Ions in the reference spectrum but not in the sample spectrum should be reviewed for subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create such discrepancies. TIC peaks are determined by the following search criteria:

16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 20 potential TICs must be reviewed, unless otherwise required. The match quality must be 70% or higher to report a positive identification, in the absence of interfering peaks.

NOTE: A 70% match corresponds to a fit of 700 on the Ion Trap or a Q-value of 70 on the MSD. If there are interfering peaks, analyst discretion must be employed when reporting matches.

16.2 Quantitative analysis:

16.2.1 When a compound has been identified, quantitation is performed using area of the appropriate quantitation ion(s), listed in Tables 1 and 1A. The internal standards technique is used. Internal standards are specified in Tables 1 and 1A. For target analytes in neither table, the internal standard nearest the analyte retention time of the analyte must be used. All qualitative results must be reported without correction for recovery, and without blank subtraction.

16.2.2 Calculate analyte concentration as follows:

Water:

$$\text{Concentration in ug/L} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(V_o)}$$

where:

A_x = Characteristic ion area, for the compound being quantitated
I_s = Internal standard concentration injected, in ug/mL
V_t = Total extract volume, in mL
A_{is} = Internal standard characteristic ion area
RF = Average response factor, for compound being quantitated (section 14.5.2)
V_o = Sample volume extracted, in liters
Dil = Any post extraction dilution factor

Sediment/Soil Sludge:

$$\text{Concentration in mg/kg} = \frac{(A_x)(I_s)(V_t)(\text{Dil})}{(A_{is})(\text{RF})(W_s)(D)}$$

where:

W_s = Sample mass extracted, in grams
D = Sample % solids, as a decimal fraction

All other variables are the same..

NOTE: If any analyte initial calibration RF has not met the 15% RSD requirement, a regression curve must be used to determine extract concentration and the calculations modified accordingly.

16.2.3 When requested, the concentration of non-calibrated sample components must be estimated. The formulas above are used with the following modifications:

16.2.3.1 Areas A_x and A_{is} are from total ion chromatograms, and the compound RF is assumed to be 1.

16.2.3.2 Use the nearest interference-free internal standard.

16.2.3.3 The concentration reported must be qualified as estimated.

16.3 Manual Integrations

16.3.1 The TriMatrix policy concerning manual integration must be followed without exception. Every effort must be made to set up data processing software integration parameters, to avoid manual integration.

16.3.2 All manual integration must be documented by printing both the original integration performed by the data system, and the manually integrated ion chromatograms. Manual integrations must be initialed and dated, and must have the rationale for the new integration clearly stated on the print out. These must be included in all client package deliverables.

16.3.3 When required, manual integration must be consistently applied to standards and samples. Examples where manual integration may be necessary include: closely eluting isomers (1,3- and 1,4-Dichlorobezene), jagged analyte peak shapes split by the integrator, and analytes like benzidine that have significant peak-tailing.

16.3.4 **Under no circumstances must manual integration be applied solely for the purpose of meeting quality control. Peak shaving and peak enhancing are strictly prohibited.**

16.3.5 For further details, refer to TriMatrix Laboratories SOP GR-10-115, *Manual Integrations*, latest revision.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running samples are also responsible for correctly filling in, turning in, and filing the associated paperwork. It is essential to perform these tasks, to provide defensible data to clients.

17.2 ELEMENT Reporting

17.2.1 When an analysis sequence is completed, all data must be uploaded to the ELEMENT DataSystem. For details on how to upload data, refer to TriMatrix SOP GR-10-123. Carefully

review the *data entry* table to insure that results are accurately reported and associated with the correct quality control batch. Manually enter a dilution factor for each corresponding sample. Dilution factors are used to calculate elevated reporting limits from diluting an extract. The dilution factor is a multiplier that may or may not reflect actual dilution volumes.

17.2.2 If internal chain-of-custody is required, it is important that a COC form be filled in correctly, and returned to the COC file location.

17.3 Laboratory Required Paperwork

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a write-over, and must be dated and initialized. Blank lines in run logbooks must be Z'd out. Refer to Figures 3 and 4 for standards forms and run logbook examples.

17.3.2 All tune, calibration, and continuing calibration standard runs must be placed in the correct file.

17.3.3 All ELEMENT documentation and raw data must be placed in the correct client folder and given to a data management technician. The technician must record the date and contents of the data then archive the folder.

17.4 CLP-like Deliverables

17.4.1 Include the following in data packages requiring CLP-like deliverables:

17.4.1.1 Copies of tunes, including the EICP. These copies must contain ion peaks for DFTPP, Benzidine, and Pentachlorophenol with tailing factor calculations and results, ion abundance results and requirements including mass listings, and associated Form Vs.

17.4.1.2 Copies of calibrations, including raw data quantitation reports, chromatograms, and associated Form VIs.

17.4.1.3 Copies of continuing calibration standards, including raw data quantitation reports, chromatograms, and associated Form VIIs.

17.4.1.4 Copies of raw sample data, including applicable quality control, quantitation reports, triple plots for positive results, chromatograms, and TIC reports when requested

17.4.1.5 Completed MPB summary Form IVs.

17.4.1.6 Completed Internal Standard recovery Form VIIIs.

17.4.1.7 Copies of supporting information, including: internal chain of custody forms, run and standards logbooks, method detection limits, and extraction summaries.

NOTE: Forms I, II, and III are generated from ELEMENT and must be included with the deliverables package.

18.0 QUALITY ASSURANCE

18.1 Quality control requirements are included in the procedure, as follows:

18.1.1 DFTPP tune (section 14.3)

18.1.2 Initial Calibration (section 14.5)

18.1.3 Continuing Calibration (section 14.6)

18.1.4 In addition, quality control must include sections 18.3 through 18.7:

18.2 Blanks

18.2.1 After the DFTPP tune and calibration or continuing calibration have been run, a blank is required before sample analysis showing that the system is free from interferences and contamination at or below the reporting limit. The blank is a Method Preparation Blank (BLK) or a solvent instrument blank (IBL). An MPB is an extracted blank from a sample batch. An IBL is methylene chloride spiked only with internal standard. A BLK may be used for the blank if uncontaminated. Otherwise an IBL must be run. Run a BLK or IBL at least once each 12-hour shift. Run blanks more frequently if carryover is suspected from a high-concentration sample, or if laboratory contamination is in question. Extract a BLK for each batch of up to 20 samples. BLKs must be spiked with surrogates and carried through the same preparation as samples. Acceptance criteria for both the IBL and BLK are that all target analytes must have concentrations below the lowest reporting limit for samples analyzed that day. Common laboratory contaminants such as phthalates must have blank concentrations less than five times the reporting limit.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following the continuing calibration standard must be evaluated during or immediately after data acquisition. All internal standard retention times must be within ± 30 seconds of the current 12-hour continuing calibration standard. All internal standard quantitation ion areas must stay within a factor of two (-50% to +100%) of the mid-point standard from the initial calibration.

18.3.2 If at any time an internal standard fails the -50% to +100% area window, the ability to accurately quantitate analytes is reduced. Every effort must be made to prevent an internal standard failure, including dilution and reanalysis of samples. Refer to TriMatrix GR-04-101 for the corrective action sequence, should internal standards fail. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for a malfunction and appropriate repairs made. Once the problem is resolved, samples analyzed with the faulty spectrometer must be re-analyzed, with acceptable internal standard responses.

18.4 Surrogates

18.4.1 All samples must be spiked with surrogates. Until twenty samples of a given matrix have been analyzed, a default recovery window of 50% - 150% will be used. When twenty samples of a given matrix have been analyzed, in-house recovery limits will be generated (section 18.5.2-18.5.4) and used. Table 6 provides examples of in-house surrogate recovery limits. These are examples only. At a minimum, surrogate recovery limits must be updated annually, on a matrix-by-matrix basis.

18.4.2 Calculate upper and lower control limits for each surrogate, as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where
p = average percent recovery
s = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If surrogate recovery is not within limits, take corrective action. Refer to TriMatrix GR-04-101 for the corrective action sequence, including re-extraction when sample volumes and holding times permit, and data qualification. If many surrogates are out-of-control for no apparent reason, the instrument must be inspected for a malfunction, and appropriate repairs made. When the problem is resolved, samples analyzed with the faulty instrument must be reanalyzed with acceptable surrogate recovery.

18.5 Matrix Spikes and Laboratory Fortified Blanks

18.5.1 To assess extraction efficiency, a Matrix Spike (MS), Matrix Spike Duplicate (MSD), and Blank Spike (BS) must be extracted. Spiking must contain the compounds in Table 7 and must be performed at least once every 20 samples for each matrix extracted. If only one to ten samples are analyzed in a month, at least one matrix spike, one matrix spike duplicate and one laboratory fortified blank is required. A BS must be extracted daily or with each sample batch, whichever is more frequent. Generally, the MPB and BS are run at the beginning of the 12-hour shift, and matrix spikes at the end. Running matrix spikes last helps document that the instrument is still operating correctly toward the end of the 12-hour shift.

NOTE: For State of Wisconsin samples and where project specifications require, all target analytes must be added for associated spikes, spike duplicates and BSs.

18.5.2 Until five matrix spikes, matrix spike duplicates, and laboratory fortified blanks have been analyzed, recoveries must be validated against default recovery limits of 50% - 150%. Matrix Spike/Matrix Spike Duplicate relative percent difference (RPD) limits must use a default maximum limit of 20%.

18.5.3 When five data sets of a given matrix (water, soil, other) have been analyzed, calculate average percent recovery (R) and standard deviation of the average (SD). Express the recovery as the percent recovery interval from R ± 3SD. For example, if R = 90% and SD = 10%, spike recovery limits are 60% - 120%. Separate limits must be calculated for laboratory fortified blanks. The relative percent difference limit must be calculated by averaging five MS/MSD percent differences ± 3 standard deviations. Acceptance limits will be updated at least annually.

18.5.4 Calculate percent recovery as follows:

$$\%\text{recovery} = \frac{(A_{spk} - A_{smp})}{SPK} \times 100$$

where:
A_{spk} = The spiked sample concentration, in ug/L or mg/kg
A_{smp} = The non-spiked sample concentration, in ug/L or mg/kg

$$SPK = \left(\frac{\text{concentration of spiked standard in ug/L x mL spiked}}{\text{initial sample volume in L}} \right)$$

18.5.5 Calculate relative percent difference (RPD) as follows:

$$\%RPD = \frac{|(MS - MSD)|}{\left[\frac{(MS + MSD)}{2}\right]} \times 100$$

where:

MS = The matrix spike concentration, in ug/L or mg/kg
MSD = The matrix spike duplicate concentration, in ug/L or mg/kg

18.5.6 If recovery or duplication are not within limits, refer to TriMatrix GR-04-101 for the corrective action sequence, to determine if or how data is qualified.

18.5.7 If a BS analyte is out-of-control, the problem must be immediately identified and corrected. Samples for the analyte must not be run until the problem is corrected. The purpose of a BS is to verify that out-of-control compounds in a MS or MSD are the result of matrix interference, rather than extraction or system error. Failure of any analyte in a BS, requires corrective action specific to the failed parameter. Every effort must be made to determine the cause for failure (mis-spiked, mis-extracted, or any other reason). When the problem is located, corrective action must be taken following TriMatrix SOP GR-04-101. If it is determined that re-extraction is necessary, the entire batch of samples must be re-extracted.

NOTE: If a BS recovery is biased high and there are no positive results for the analyte in the associated samples, results can be reported without the need for corrective action or qualification.

18.6 Method Detection Limit Studies

18.6.1 A Method Detection Limit (MDL) study must be performed annually for every target compound analyzed. MDL studies must be performed on each instrument involved in sample analysis. Analyte results for which a study has not been performed must be reported as estimated. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. Actual reporting limits are derived from the MDL study. Minimum reporting limits are equal to the concentration spiked for the MDL study, provided the MDL passes. Reporting limits actually achieved for any given analysis will vary depending on instrument sensitivity, matrix, and extract dilutions. MDL studies must be run for aqueous and soil matrices.

18.6.2 The procedure followed for MDL studies is based on the method given in 40 Code of Federal Regulations, Part 136, Appendix B, latest revision. Method 8270C quality control governing the analysis must be followed.

18.6.3 Seven replicate extractions and subsequent analyses are performed using reagent water or Ottawa Sand spiked with the target compounds of interest at estimated minimum reporting limit concentrations.

18.6.4 For each target compound, standard deviation of the average is calculated and multiplied by 3.143. The resulting number is the calculated MDL.

18.6.5 If the concentration spiked is greater than or equal to the calculated MDL, and less than or equal to five times the calculated MDL, and there are no zero percent recoveries in the seven results, the MDL is acceptable. The MDL study must be repeated, only for the analyte(s) that did not pass. If a study needs repeated at a different concentration, the entire set of seven needs to be repeated. If a study does not pass due to poor reproducibility on one result, only that result needs repeated. Only one result may be rejected and repeated however. All seven results do not need to be obtained during the same 12-hour shift.

18.6.6 If a reporting limit is above a client or state-desired reporting limit, the calculated MDL may be used provided results are narrated. Narration must state that the reporting limit is the calculated MDL and when spiked at that level, analyte was not observed.

18.6.7 Table 6 is an MDL spreadsheet example. The spreadsheet is used to calculate and verify MDLs and reporting limits.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running a one time demonstration of capability study. While the demonstration of capability is not instrument dependent, a study is required on every instrument running samples, to demonstrate each instrument's ability to generate acceptable accuracy and precision.

19.2 Prepare a 100 ug/L concentration of all compounds of interest in four 1000 mL aliquots of reagent water, by spiking with 0.100 mL of a 100 ng/uL standard. Extract to a 10 mL final volume. The spiking solution must be prepared independently from stock standards used for quantitation.

19.3 Analyze the four extracts using Method 8270C quality control, and following every step of the procedure. For each target compound, calculate average percent recovery "x" and standard deviation "s" in ug/L. Use all four results. Calculate by inputting into the IDC spreadsheet located on the laboratory intranet subdirectory "\\library\spreadsheets\training documents\qc_idc-demonstration of capability(doc).xls".

19.3.1 For each target compound, "x" must pass the current ELEMENT BS acceptance limits. Also, relative standard deviation must be less than or equal to 20%. Calculate %RSD as [("s"/"x")*100]. If "s" and %RSD for all analytes pass both acceptance criteria, the demonstration of capability study is complete. The analyst and associated instrument may run samples. If %RSD exceeds 20% or "x" falls outside the current ELEMENT BS acceptance limits, analyst or instrument performance is unacceptable for the analyte.

19.3.2 When one or more analytes fail for "x" or %RSD, proceed according to section 19.4.

19.4 Locate and correct the source of any problem and repeat the study for failed analytes. Repeated failure however, will confirm a general problem with the procedure or technique used. If this occurs, locate and correct the problem, then repeat the study for all unacceptable compounds beginning with section 19.1. Samples may not be analyzed by any analyst or on any instrument, until a demonstration of capability study has been successfully completed. Copies of successful demonstration of capability studies, IDC spreadsheets and raw data must be given to Quality Assurance.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 3, December 1996

22.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

22.3 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. B, Definition and Procedure for the Determination of the Method Detection Limit

23.0 ATTACHMENTS

23.1	Table 1	Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
23.2	Table 1A	Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions
23.3	Table 2	Default Reporting Limits
23.4	Table 2A	Default Reporting Limits
23.5	Table 3	DFTPP Key Ions and Ion Abundance Criteria (EPA CLP)
	Table 3A	DFTPP Key Ions and Ion Abundance Criteria (Method 8270C Default Criteria)
23.6	Table 4	System Performance Check Compounds (SPCC)
23.7	Table 5	Continuing Check Compounds (CCC)
23.8	Table 6	Method Detection Limit Study Example (Partial Table)
23.9	Table 7	Demonstration of Capability Study Example (Partial Table)
23.10	Figure 1	Pentachlorophenol Tailing Factor
23.11	Figure 2	Continuing Calibration Standard Total Ion Chromatogram, Varian Ion Trap (10 ug/mL)
23.12	Figure 2A	Continuing Calibration Standard Total Ion Chromatogram, Agilent 5973 MSD (10 ug/mL)
23.13	Figure 3	Standards Form Example
23.14	Figure 4	Analytical Run Logbook Example

23.15 Method 8270C Selected Ion Monitoring (SIM) instructions

Attachment 23.1 (Table 1)
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.76	162	164, 160	3
Chrysene-d_{12} (I.S.)	25.04	240	120, 236	5
Dichlorobenzene-1,4-d_4 (I.S.)	10.06	152	150, 115	1
Naphthalene-d_8 (I.S.)	13.21	136	68	2
Perylene-d_{12} (I.S.)	29.83	264+265 (264)**	260, 265	6
Phenanthrene-d_{10} (I.S.)	20.50	188	94,80	4
2-Fluorobiphenyl (surr.)	16.00	172	171	3
2-Fluorophenol (surr.)	7.18	112	64	1
Nitrobenzene-d_5 (surr.)	11.38	127+128 (128)**	82, 54	2
Phenol-d_6 (surr.)	9.13	99		1
2,4,6-Tribromophenol (surr.)	19.33	330	332, 141	3
Terphenyl-o (surr.)	21.03	230	215,229	5
Acenaphthene	17.84	152+154 (154)**	153, 152	3
Acenaphthylene	17.44	152	151, 153	3
Anthracene	20.63	178	176, 179	4
Benzoic acid	12.39	105	77,122	2
Benzo(a)anthracene	25.00	228	229, 226	5
Benzo(b and k)fluoranthene	28.24/28.40	252	253, 125	6
Benzo(g,h,i)perylene	37.32	276+277 (276)**	138, 277	6
Benzo(a)pyrene	29.60	252	253, 125	6
Benzyl alcohol	10.35	108	79, 77	1
Bis(2-chloroethoxy)methane	12.51	92+93 (93)**	95, 123	2
Bis(2-chloroethyl)ether	9.38	92+93 (93)**	63, 95	1
Bis(2-chloroisopropyl)ether	10.65	45	77, 121	1
Bis(2-ethylhexyl)phthalate	24.59	149	167, 279	5
4-Bromophenyl phenyl ether	19.73	248	250, 141	4
Butyl benzyl phthalate	23.56	149	91, 206	5
4-Chloroaniline	13.40	127	129	2
2-Chloronaphthalene	16.34	162	127, 164	3
4-Chloro-3-methylphenol	14.60	107	144, 142	2
2-Chlorophenol	9.57	128	64, 130	1
4-Chlorophenyl phenyl ether	18.87	204	206, 141	3

Attachment 23.1 (Table 1) - Continued
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Chrysene	25.10	228	226, 229	5
Dibenz(a,h)anthracene	35.57	278	139, 279	6
Dibenzofuran	18.20	168	139	3
Di-n-butylphthalate	21.23	149	150, 104	4
1,3-Dichlorobenzene	9.93	146	148, 111	1
1,4-Dichlorobenzene	10.10	146	148, 111	1
1,2-Dichlorobenzene	10.45	146	148, 111	1
3,3'-Dichlorobenzidine	24.80	252	254, 126	5
2,4-Dichlorophenol	12.84	162	164, 98	2
Diethylphthalate	18.59	149+177 (149)**	177, 150	3
2,4-Dimethylphenol	12.26	107	121, 122	2
Dimethylphthalate	17.03	163	194, 164	3
4,6-Dinitro-2-methylphenol	18.94	198	51, 105	4
2,4-Dinitrophenol	17.86	184	63, 154	3
2,4-Dinitrotoluene	18.12	165	63, 89	3
2,6-Dinitrotoluene	17.20	165	63, 89	3
1,2-Diphenylhydrazine	19.15	77	105, 182	4
Di-n-octylphthalate	26.32	149	167, 43	5
Fluoranthene	22.35	202	101, 203	4
Fluorene	18.89	166	165, 167	3
Hexachlorobenzene	19.87	284	142, 249	4
Hexachlorobutadiene	13.54	225	223, 227	2
Hexachlorocyclopentadiene	15.45	237	235, 272	3
Hexachloroethane	11.30	119	201, 199	1
Indeno(1,2,3-cd)pyrene	35.52	276+277 (276)**	138, 227	5
Isophorone	12.00	82	95, 138	2
2-Methylnapthalene	15.06	142	141	2
2-Methylphenol (o-cresol)	10.58	108	79,107	1
3- and/or 4-Methylphenol (m,p-cresol)	10.95	108	79,107	1
Naphthalene	13.26	128	129, 127	2

Attachment 23.1 (Table 1) - Continued
Routine Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
2-Nitroaniline	16.59	138	92, 138	3
3-Nitroaniline	17.62	138	108, 92	3
4-Nitroaniline	18.90	108+138 (138)**	108, 92	3
Nitrobenzene	11.43	77+123 (77)**	65	2
2-Nitrophenol	12.21	109+139 (139) **	109, 65	2
4-Nitrophenol	18.02	65	109, 65	3
N-Nitrosodimethylamine	4.58	73+74 (74)**	42	1
N-Nitrosodiphenylamine	19.08	169	168, 167	4
N-Nitrosodipropylamine	10.95	70	42, 101, 1	1
Pentachlorophenol	20.18	266	264, 268	4
Phenanthrene	20.54	178	179, 176	4
Phenol	9.17	94	65, 66	1
Pyrene	22.74	202	200, 203	5
1,2,4-Trichlorobenzene	13.05	180	182, 145	2
2,4,5-Trichlorophenol	15.91	196	198, 200	3
2,4,6-Trichlorophenol	15.79	196	198, 200	3

I.S. = Internal Standard
surr. = Surrogate
* retention time provided for example only.
** for MSD

Attachment 23.2 (Table 1A)
Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
Acenaphthene-d_{10} (I.S.)	17.05	162	160,164	3
Chrysene-d_{12} (I.S.)	24.14	240	120,236	5
Dichlorobenzene-1,4-d_4 (I.S.)	9.39	152	150,115	1
Naphthalene-d_8 (I.S.)	12.49	136	68	2
Perylene-d_{12} (I.S.)	28.22	264+265 (264)***	260	6
Phenanthrene-d_{10} (I.S.)	19.96	188	94,80	4
Acetophenone	10.45	105	77,70	2
2-Acetylaminofluorene	23.74	181	223,180	3
4-Aminobiphenyl	18.63	169	168,167	3
Aniline	8.76	93	66,65	1
Aramite**	22.64/22.73/22.85	185	63,57	4
2-sec-Butyl-4,6-dinitrophenol	19.89	211	163,147	3
2,6-Dichlorophenol	12.69	162	164,63	2
p-(Dimethylamino)azobenzene	22.71	120	225,77	3
7,12-Dimethylbenz(a)anthracene	26.79	256	252,241	5
3,3'-Dimethylbenzidine	23.26	212	213,106	3
2,2-Dimethylphenethylamine	12.64	58	91	2
1,3-Dinitrobenzene	16.55	76	139,169,50, 168	2
Carbazole	17.97	167	83, 139	4
Ethyl methacrylate	4.98	69	41,99	1
Ethyl methanesulfonate	7.74	79	109,97	1
Hexachlorophene	27.48	196	198,209	6
Hexachloropropene	12.75	213	215,211	2
Isosafrole	15.58	162	131,104	2
Methapyrilene	21.39	58	97,72	3
3-Methylcholanthrene	29.28	268	126,252	5
Methyl methacrylate	3.67	69	41,100	1
Methyl methanesulfonate	6.34	80	65,79	1
1,4-Naphthoquinone	16.23	158	104,102	2
1-Naphthylamine	17.87	143	115,116	2
2-Naphthylamine	18.05	143	115,116	2
4-Nitroquinoline-1-oxide	21.38	190	89,101	4

Approved By: 1-22-09 **QA Officer** **Approved By:** 1/23/09 **Area Supervisor**

Attachment 23.2 (Table 1A) - Continued
Additional Target Compounds, Internal Standards, Surrogates, Retention Times, Quantitation and Secondary Ions

Compound	Retention Time (min.)*	Quantitation Ion	Secondary Ion(s)	Internal Standard
N-Nitrosodi-n-butylamine	13.64	84	57,41	2
N-Nitrosodiethylamine	7.14	102	42,44	1
N-Nitrosomethylethylamine	5.75	88	42,43	1
N-Nitrosomorpholine	10.48	56	116,86	2
N-Nitrosopiperidine	11.24	114	42,56	2
N-Nitrosopyrrolidine	10.39	100	41,42	1
5-Nitro-o-toluidine	18.42	152	106,77	3
Pentachlorobenzene	17.46	250	252,248	3
Pentachloroethane	8.74	167	93,117	2
Pentachloronitrobenzene	19.78	237	169,142	4
Phenacetin	19.17	108	109,179	3
p-Phenylenediamine	13.80	108	80,107	2
2-Picoline	6.73	93	66,92	1
Pronamide	20.20	173	175,145	3
Pyridine	6.32	79	52,51	1
Safrole	14.24	162	131,101	2
1,2,4,5-Tetrachlorobenzene	14.76	216	214,218	3
2,3,4,6-Tetrachlorophenol	17.83	232	230,234	3
o-Toluidine	10.54	107	106,79	2
sym-Trinitrobenzene	19.09	75	74,213	3
Octachlorocyclopentene (C-58)	19.03	307+309	237	3

I.S. = Internal Standard
* retention time provided for example only
** Aramite chromatographs into three peaks
*** For MSD

Attachment 23.3 (Table 2)
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Acenaphthene	83-32-9	5	0.33
Acenaphthylene	208-96-8	5	0.33
Anthracene	120-12-7	5	0.33
Benzoic acid	65-85-0	50	3.3
Benzo(a)anthracene	56-55-3	5	0.33
Benzo(b and k)fluoranthene	205-99-2 / 207-08-9	5	0.33
Benzo(g,h,i)perylene	191-24-2	5	0.33
Benzo(a)pyrene	50-32-8	5	0.33
Benzyl alcohol	100-51-6	50	1.3
Bis(2-chloroethoxy)methane	111-91-1	5	0.33
Bis(2-chloroethyl)ether	111-44-4	5	0.33
Bis(2-chloroisopropyl)ether	39638-32-9	5	0.33
Bis(2-ethylhexyl)phthalate	117-81-7	5	0.33
4-Bromophenyl phenyl ether	101-55-3	5	0.33
Butyl benzyl phthalate	85-68-7	5	0.33
Carbazole	86-74-8	5	0.33
4-Chloroaniline	106-47-8	20	1.3
2-Chloronaphthalene	91-58-7	5	0.33
4-Chloro-3-methylphenol	59-50-7	5	0.33
2-Chlorophenol	95-57-8	5	0.33
4-Chlorophenyl phenyl ether	7005-72-3	5	0.33
Chrysene	218-01-9	5	0.33
Dibenz(a,h)anthracene	53-70-3	5	0.33
Dibenzofuran	132-64-9	5	0.33
Di-n-butylphthalate	84-74-2	5	0.33
1,3-Dichlorobenzene	541-73-1	5	0.33
1,4-Dichlorobenzene	106-46-7	5	0.33
1,2-Dichlorobenzene	95-50-1	5	0.33
3,3'-Dichlorobenzidine	91-94-1	20	2.0
2,4-Dichlorophenol	120-83-2	5	0.33
Diethylphthalate	84-66-2	5	0.33

Approved By: 1-22-09 **QA Officer** **Approved By:** 1/23/19 **Area Supervisor**

Attachment 23.3 (Table 2) - Continued
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
2,4-Dimethylphenol	105-67-9	5	0.33
Dimethylphthalate	131-11-3	5	0.33
4,6-Dinitro-2-methylphenol	534-52-1	20	1.7
2,4-Dinitrophenol	51-28-5	20	1.7
2,4-Dinitrotoluene	121-14-2	5	0.33
2,6-Dinitrotoluene	606-20-2	5	0.33
Diphenylamine	122-39-4	5	0.33
Di-n-octylphthalate	117-84-0	5	0.33
Fluoranthene	206-44-0	5	0.33
Fluorene	86-73-7	5	0.33
Hexachlorobenzene	118-74-1	5	0.33
Hexachlorobutadiene	87-68-3	5	0.33
Hexachlorocyclopentadiene	77-47-4	5	0.33
Hexachloroethane	67-72-1	5	0.33
Indeno(1,2,3-cd)pyrene	193-39-5	5	0.33
Isophorone	78-59-1	5	0.33
2-Methylnaphthalene	91-57-6	5	0.33
2-Methylphenol (o-cresol)	95-48-7	5	0.33
3- and/or 4-Methylphenol (m,p-cresol)	106-44-5	5	0.33
Naphthalene	91-20-3	5	0.33
2-Nitroaniline	88-74-4	20	1.7
3-Nitroaniline	99-09-2	20	1.7
4-Nitroaniline	100-01-6	20	1.7
Nitrobenzene	98-95-3	5	0.33
2-Nitrophenol	88-75-5	5	0.33
4-Nitrophenol	100-02-7	20	1.7
N-Nitrosodimethylamine	62-75-9	5	0.33
N-Nitrosodiphenylamine	86-30-6	5	0.33
N-Nitrosodipropylamine	621-64-7	5	0.33
Pentachlorophenol	87-86-5	20	1.7

Approved By: 1-22-09 **QA Officer** **Approved By:** 1/23/9 **Area Supervisor**

Attachment 23.3 (Table 2) - Continued
Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/Ground/ Wastewater ug/L	Low Soil/ Sediment mg/kg
Phenanthrene	85-01-8	5	0.33
Phenol	108-95-2	5	0.33
Pyrene	129-00-0	5	0.33
1,2,4-Trichlorobenzene	120-82-1	5	0.33
2,4,5-Trichlorophenol	95-95-4	5	0.33
2,4,6-Trichlorophenol	88-06-2	5	0.33

* Sample reporting limits are highly matrix-dependent. The reporting limits listed are provided for guidance and may not always be achievable.

** Water reporting limits are based on an extraction of 1000 mL to 10 mL, in the MDL study. Reporting limits listed for soil/sediment are based on an extraction of 30 g to 10 mL.

Attachment 23.4 (Table 2A)
Additional Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
Acetophenone	98-86-2	10	0.33
2-Acetylaminofluorene	53-96-3	50	2.0
4-Aminobiphenyl	92-67-1	10	0.33
Aniline	62-53-3	5	1.7
Aramite	140-57-8	50	2.0
2-sec-Butyl-4,6-dinitrophenol	88-85-7	5	2.0
2,6-Dichlorophenol	87-65-0	5	0.33
p-(Dimethylamino)azobenzene	60-11-7	10	0.33
7,12-Dimethylbenz(a)anthracene	57-97-6	10	0.33
3,3'-Dimethylbenzidine	119-93-7	50	2.0
2,2-Dimethylphenethylamine	122-09-8	20	0.70
1,3-Dinitrobenzene	99-65-0	5	0.33
Ethyl methacrylate	97-63-2	50	2.0
Ethyl methanesulfonate	62-50-0	10	1.0
Hexachlorophene	70-30-4	***	***
Hexachloropropene	1888-71-7	50	2.0
Isosafrole	120-58-1	20	0.70
Methapyrilene	91-80-5	10	1.0
3-Methylcholanthrene	56-49-5	50	2.0
Methyl methacrylate	80-62-6	50	2.0
Methyl methanesulfonate	66-27-3	50	2.0
1,4-Naphthoquinone	130-15-4	1000	30
1-Naphthylamine	134-32-7	50	2.0
2-Naphthylamine	91-59-8	50	2.0
4-Nitroquinoline-1-oxide	56-57-5	500	20
N-Nitrosodi-n-butylamine	924-16-3	20	0.90
N-Nitrosodiethylamine	55-18-5	5	2.0
N-Nitrosomethylethylamine	62-75-9	5	2.0
N-Nitrosomorpholine	59-89-2	20	0.70
N-Nitrosopiperidine	100-75-4	20	0.70

Attachment 23.4 (Table 2A) - Continued
Additional Default Reporting Limits

Semivolatiles	CAS Number	Default Reporting Limits** Surface/ Ground Water ug/L	Low Soil/ Sediment mg/kg
N-Nitrosopyrrolidine	930-55-2	20	0.70
5-Nitro-o-toluidine	99-55-8	10	0.33
Pentachlorobenzene	608-93-5	5	0.33
Pentachloroethane	76-01-7	50	2.0
Pentachloronitrobenzene	82-68-8	20	0.50
Phenacetin	62-44-2	10	0.33
p-Phenylenediamine	106-50-3	***	***
2-Picoline	109-06-8	20	0.70
Pronamide	23950-58-5	10	0.33
Pyridine	110-86-1	10	0.33
Safrole	94-59-7	10	0.33
1,2,4,5-Tetrachlorobenzene	95-94-3	10	0.50
2,3,4,6-Tetrachlorophenol	58-90-2	50	2.0
o-Toluidine	95-53-4	10	0.33
sym-Trinitrobenzene	99-35-4	20	0.70
Octachlorocyclopentene (C-58)	706-78-5	0.1	0.02

* Sample reporting limits are highly matrix-dependent. The listed values are provided for guidance and may not always be achievable.

** Water reporting limits are based on extracting 1000 mL to 1.0 mL, in the MDL study. Reporting limits listed for soil/sediment are based on extracting 30 g to 1.0 mL.

*** These compounds are demonstrated to be difficult to extract from water, or difficult to chromatograph. Method detection limits are not available.

Attachment 23.5 (Table 3)
DFTPP Key Ions and Ion Abundance Criteria
(EPA CLP)

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	<2% of mass 69
69	Present
70	<2% of mass 69
127	25-75% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance (see note)
199	5-9% of mass 198
275	10-30% of mass 198
365	>0.75% of mass 198
441	Present but less than mass 443
442	40-110% of mass 198
443	15-24% of mass 442

NOTE: All ion abundances MUST be normalized to m/z 198 which is the nominal base peak, even though ion abundances of m/z 442 may be up to 110% that of m/z 198.

Attachment 23.5 (Table 3A)
DFTPP Key Ions and Ion Abundance Criteria
(Method 8270C Defaults)

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

Attachment 23.6 (Table 4)
System Performance Check Compounds (SPCC)

Base/Neutral Fraction	Acid Fraction
N-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

Attachment 23.7 (Table 5)
Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Attachment 23.8 (Table 6)
Method Detection Limit Study Example

TriMatrix Laboratories, Inc.

SEMI-VOLATILE LABORATORY
INSTRUMENT NUMBER 195 2004 WATER
METHOD DETECTION LIMIT STUDY

Parameter / Compound	Reference Citation	Date Analyzed	Amount Spiked	Units	Rep. #1	Rep. #2	Rep. #3	Rep. #4	Rep. #5	Rep. #6	Rep. #7	Average Amount Found	Average % Recovery	Standard Deviation	MDL
N-Nitrosodimethylamine	8270C	12/29/2003	1.00	ug/L	0.550	0.490	0.370	0.180	0.710	0.620	0.310	0.461	46%	0.185	0.582
Phenol	8270C	12/31/2003	0.500	ug/L	0.273	0.264	0.270	0.340	0.325	0.343	0.281	0.299	60%	0.0350	0.110
Aniline	8270C	12/29/2003	1.00	ug/L	0.450	0.400	0.330	0.260	0.550	0.470	0.290	0.393	39%	0.105	0.330
Bis (2-chloroethyl)ether	8270C	12/31/2003	0.500	ug/L	0.397	0.466	0.483	0.537	0.501	0.429	0.514	0.475	95%	0.0490	0.154
2-Chlorophenol	8270C	12/31/2003	0.500	ug/L	0.425	0.372	0.411	0.476	0.460	0.482	0.431	0.437	87%	0.0391	0.123
1,3-Dichlorobenzene	8270C	12/31/2003	0.500	ug/L	0.411	0.377	0.393	0.465	0.456	0.457	0.424	0.426	85%	0.0344	0.108
1,4-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.01	0.910	0.780	0.580	1.06	0.880	0.620	0.834	83%	0.184	0.578
Benzaldehyde	8270C	12/29/2003	1.00	ug/L	0.880	0.790	0.900	0.710	0.980	1.02	0.770	0.864	86%	0.114	0.357
Benzyl Alcohol	8270C	12/29/2003	1.00	ug/L	0.490	0.420	0.320	0.280	0.690	0.540	0.330	0.439	44%	0.146	0.458
1,2-Dichlorobenzene	8270C	12/29/2003	1.00	ug/L	1.04	0.870	0.740	0.590	1.10	0.870	0.610	0.831	83%	0.198	0.621
2-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.488	0.454	0.521	0.416	0.516	0.539	0.400	0.476	95%	0.0542	0.170
Bis-(2-chloroisopropyl)-ether	8270C	12/31/2003	0.500	ug/L	0.512	0.476	0.496	0.701	0.578	0.519	0.502	0.541	108%	0.0775	0.244
Acetophenone	8270C	12/29/2003	1.00	ug/L	0.810	0.720	0.710	0.700	0.860	0.840	0.670	0.759	76%	0.0760	0.239
4-Methylphenol	8270C	12/31/2003	0.500	ug/L	0.345	0.422	0.474	0.451	0.449	0.435	0.409	0.426	85%	0.0416	0.131
N-Nitrosodi-n-propylamine	8270C	12/29/2003	1.00	ug/L	1.16	0.780	0.640	0.570	1.12	0.860	0.600	0.819	82%	0.242	0.761
Hexachloroethane	8270C	12/29/2003	1.00	ug/L	0.920	0.830	0.650	0.480	0.940	0.760	0.550	0.733	73%	0.179	0.563
Nitrobenzene	8270C	12/31/2003	0.500	ug/L	0.450	0.528	0.449	0.609	0.542	0.491	0.466	0.505	101%	0.0586	0.184
Isophorone	8270C	12/31/2003	0.500	ug/L	0.532	0.481	0.563	0.619	0.641	0.547	0.601	0.569	114%	0.0553	0.174
2-Nitrophenol	8270C	12/31/2003	0.500	ug/L	0.526	0.604	0.636	0.554	0.654	0.596	0.647	0.602	120%	0.0483	0.152
2,4-Dimethylphenol	8270C	12/31/2003	0.500	ug/L	0.367	0.315	0.375	0.295	0.374	0.398	0.364	0.355	71%	0.0366	0.115
Benzoic Acid	8270C	2/11/2004	5.00	ug/L	3.36	3.78	3.23	1.52	3.48	3.39	3.43	3.17	63%	0.747	2.35
Bis(2-chloroethoxy)methane	8270C	12/31/2003	0.500	ug/L	0.469	0.399	0.513	0.550	0.477	0.510	0.484	0.486	97%	0.0472	0.148
2,4-Dichlorophenol	8270C	12/29/2003	1.00	ug/L	0.840	0.710	0.530	0.440	0.900	0.840	0.510	0.681	68%	0.187	0.587
1,2,4-Trichlorobenzene	8270C	12/29/2003	1.00	ug/L	0.970	0.820	0.650	0.500	1.00	0.800	0.560	0.757	76%	0.194	0.611

Approved By: PD 1-22-09
QA Officer

Approved By: JMW 1/23/9
Area Supervisor

Attachment 23.9 (Table 7)
Demonstration of Capability Study Example

TriMatrix Laboratories, Inc.

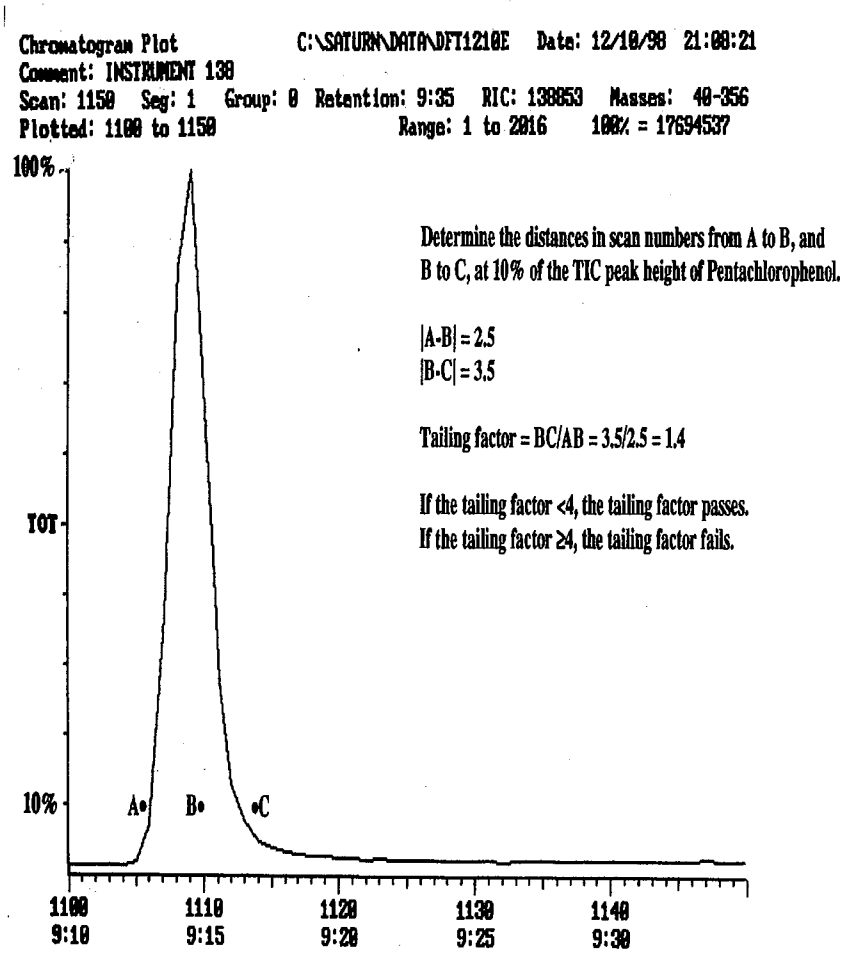
SEMI-VOLATILE LABORATORY
DEMONSTRATION OF CAPABILITY FOR SCOTT J. PUGH

Parameter	Date Analyzed	Method	Inst. Number	Units	Amount Spiked	Cert. #1 Amount Found	Cert. #2 Amount Found	Cert. #3 Amount Found	Cert. #4 Amount Found	Average Percent Recovery	Percent Recovery Window	Pass/Fail Percent Recovery	Percent RSD	Percent RSD Window	Percent RSD Pass/Fail	Overall Pass/ Fail
Phenol	12/15/2003	8270c	133	ug/L	10.0000	3.9900	3.9300	4.3400	4.0800	40.9%	19 -55	PASS	4.4%	20	PASS	*PASS*
Aniline	12/15/2003	8270c	133	ug/L	10.0000	5.2300	5.3400	5.8100	5.5100	54.7%	23 -84	PASS	4.6%	20	PASS	*PASS*
Bis(2-Chloroethyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	8.9700	8.8900	9.6100	9.3900	92.2%	70 -130	PASS	3.7%	20	PASS	*PASS*
2-Chlorophenol	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9400	8.8800	8.2500	82.2%	70 -130	PASS	5.8%	20	PASS	*PASS*
1,3-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.4600	7.8600	8.2400	7.8000	78.4%	70 -130	PASS	4.1%	20	PASS	*PASS*
1,4-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.1900	7.6200	8.2100	7.7500	76.9%	70 -130	PASS	5.5%	20	PASS	*PASS*
Benzyl Alcohol	12/15/2003	8270c	133	ug/L	10.0000	8.1300	8.2300	8.6500	8.7800	84.5%	70 -130	PASS	3.7%	20	PASS	*PASS*
1,2-Dichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	7.8900	8.0300	8.6100	8.2500	82.0%	70 -130	PASS	3.8%	20	PASS	*PASS*
2-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.5500	7.6700	8.0200	7.7800	77.6%	70 -130	PASS	2.6%	20	PASS	*PASS*
Bis(2-Chloroisopropyl)Ether	12/15/2003	8270c	133	ug/L	10.0000	9.3000	9.3700	9.7200	9.5600	94.9%	70 -130	PASS	2.0%	20	PASS	*PASS*
4-Methylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2900	7.1100	7.9500	7.7100	75.2%	70 -130	PASS	5.1%	20	PASS	*PASS*
N-Nitrosodi-N-Propylamine	12/15/2003	8270c	133	ug/L	10.0000	9.3500	9.2300	10.4000	10.0200	97.5%	70 -130	PASS	5.7%	20	PASS	*PASS*
Hexachloroethane	12/15/2003	8270c	133	ug/L	10.0000	7.8000	7.9000	8.3300	7.8200	79.6%	70 -130	PASS	3.1%	20	PASS	*PASS*
Nitrobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.8400	9.3700	9.4300	8.9500	91.5%	70 -130	PASS	3.2%	20	PASS	*PASS*
Isophorone	12/15/2003	8270c	133	ug/L	10.0000	9.3100	9.7400	9.3600	9.5900	95.0%	70 -130	PASS	2.1%	20	PASS	*PASS*
2-Nitrophenol	12/15/2003	8270c	133	ug/L	10.0000	8.8100	9.5200	9.5200	9.3600	93.0%	70 -130	PASS	3.6%	20	PASS	*PASS*
2,4-Dimethylphenol	12/15/2003	8270c	133	ug/L	10.0000	7.2100	7.2400	7.0800	6.8600	71.0%	70 -130	PASS	2.4%	20	PASS	*PASS*
Benzoic Acid	12/15/2003	8270c	133	ug/L	10.0000	4.3400	5.4500	5.3600	5.5800	51.8%	20 -69	PASS	11.0%	20	PASS	*PASS*
Bis(2-Chloroethoxy)Methane	12/15/2003	8270c	133	ug/L	10.0000	9.1600	9.5800	9.5900	9.2700	94.0%	70 -130	PASS	2.3%	20	PASS	*PASS*
2,4-Dichlorophenol	12/15/2003	8270c	133	ug/L	10.0000	9.0800	9.3200	9.4000	9.3400	92.9%	70 -130	PASS	1.5%	20	PASS	*PASS*
1,2,4-Trichlorobenzene	12/15/2003	8270c	133	ug/L	10.0000	8.2000	8.6700	9.0400	8.5100	86.1%	70 -130	PASS	4.1%	20	PASS	*PASS*
Naphthalene	12/15/2003	8270c	133	ug/L	10.0000	9.2200	9.6100	9.3200	8.9200	92.7%	70 -130	PASS	3.1%	20	PASS	*PASS*

Approved By: RD 1-22-09
QA Officer

Approved By: JMK 1/23/09
Area Supervisor

Attachment 23.10 (Figure 1)
Pentachlorophenol tailing factor



Approved By: _____ QA Manager

Approved By: _____ Area Manager

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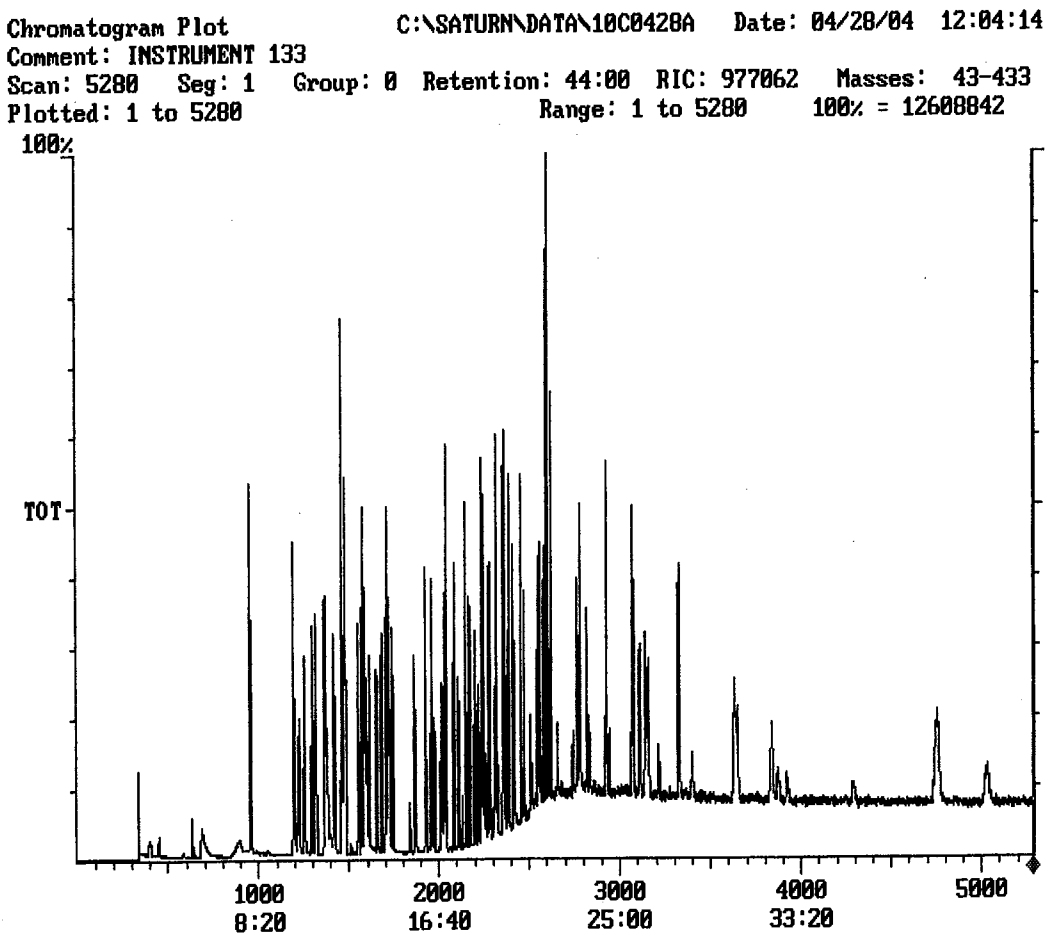
Attachment 23.11 (Figure 2)
Continuing Calibration Standard (10 ug/mL)
Total Ion Chromatogram, Varian Ion Trap

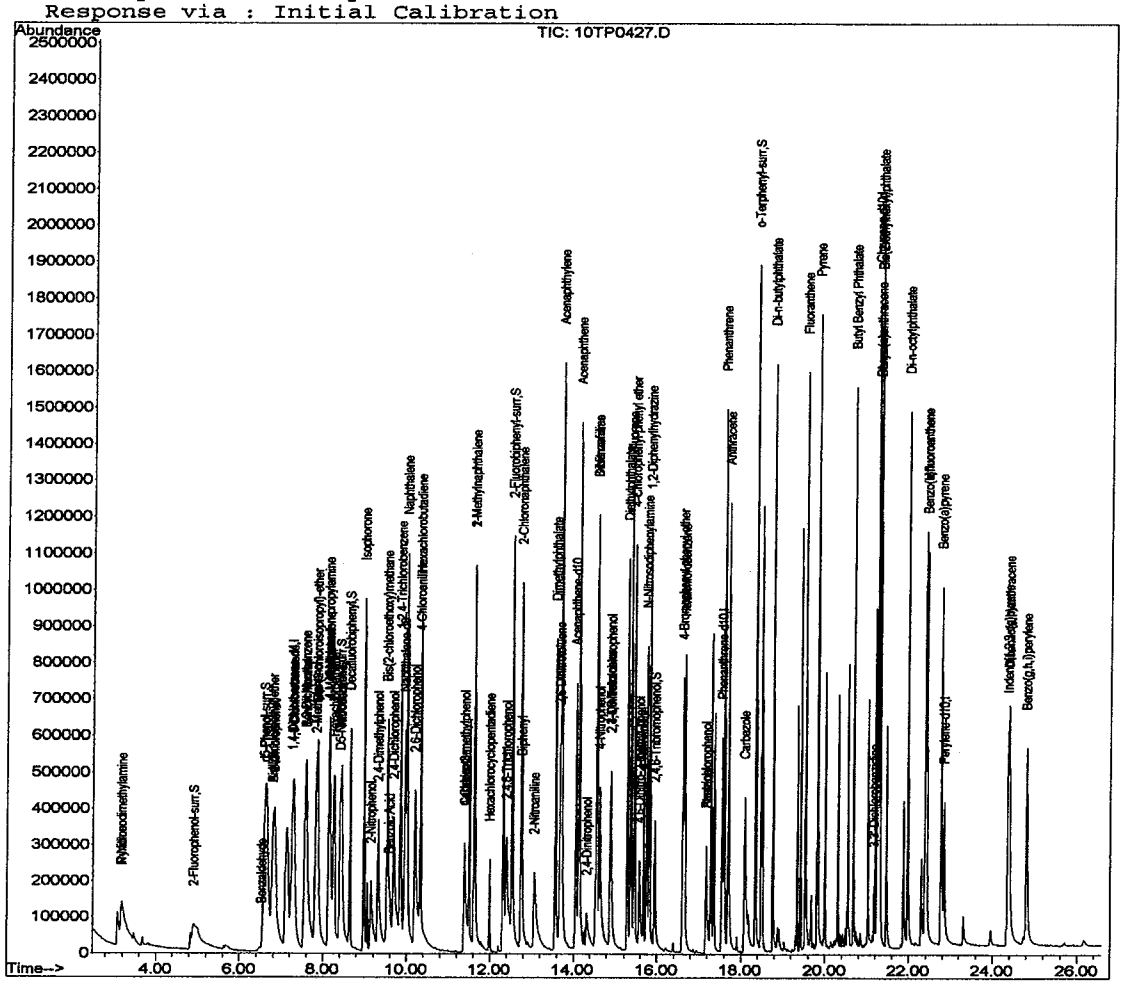


Attachment 23.12 (Figure 2A)
Continuing Calibration Standard (10 ug/mL)
Total Ion Chromatrogram, Agilent 5973 MSD

Quantitation Report

```
Data File : C:\HPCHEM\1\DATA\04-27-04\10TP0427.D          Vial: 74
Acq On    : 27 Apr 2004  10:17                        Operator:
Sample    : 10ppm bna pest                            Inst     : GC/MS Ins
Misc      :                                           Multiplr: 1.00
MS Integration Params: rteint.p
Quant Time: Apr 27 10:44 2004                 Quant Results File: BNA04074.RES

Method       : C:\HPCHEM\1\METHODS\BNA04074.M (RTE Integrator)
Title        : 8270C/625 BNA
Last Update  : Tue Apr 27 17:24:56 2004
Response via : Initial Calibration
```



10TP0427.D BNA04074.M Thu Apr 29 14:34:16 2004 Page 4

Approved By: 1-22-09 **QA Officer** **Approved By:** 1/23/9 **Area Supervisor**

Attachment 23.13 (Figure 3)
Standards Form Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

A605395

Description:	DFTPP Tune Standard	Expires:	Aug-27-06
Standard Type:	MS Tune	Prepared:	May-11-06
Solvent:	Solvent Lot # C07H05	Prepared By:	Scott J. Pugh
Final Volume (mls):	1	Department:	Semivolatiles MS
Vials:	1	Last Edit:	May-11-06 09:04 by SJP

Solvent: MeCl

Analyte	CAS Number	Concentration	Units
4,4'-DDT	50-29-3	50	ppm
Benzidine	92-87-5	50	ppm
Decafluorotriphenylphosphine		5	ppm
Pentachlorophenol	87-86-5	50	ppm

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A409098	Pentachlorophenol, SVMS	Aug-27-01	** Vendor **	Aug-27-06	Dec-01-05 11:57 by RGJ	0.05
A512731	4,4-DDT SVMS	Dec-08-05	** Vendor **	Dec-08-06	Dec-27-05 09:53 by SJP	0.05
A601262	Benzidine, SVMS	Feb-24-05	** Vendor **	Feb-24-08	Jan-09-06 15:23 by SJP	0.05
A603057	DFTPP, SVMS	Feb-27-06	** Vendor **	Feb-27-07	Mar-02-06 08:58 by SJP	0.005

Approved By: 1-22-09 **QA Officer** **Approved By:** 1/23/09 **Area Supervisor**

Attachment 23.14 (Figure 4)
Analytical Run Logbook Example

TriMatrix Laboratories, Inc.

Instrument ID: Saturn GC/MS Instrument #133 Date: 4/23/04 Taped: _____ Disk ID: _____ Analyst: SDS

Instrument Settings	Acquisition Information	Temperature Program					Miscellaneous
Multiplier: 1550	Scan Range: 40-450 amu	GC	Temp. (C)	Rate (C/min)	Time (min)	Total (min)	New Column Date: 4/6/4
AGC 7000	Seconds/Scan: 0.500						New Septa: Yes (No)
Interface Temp: 250C	Filament Delay: 3.5 min.	Step #1	45	0.0	1.5	1.5	Working Std. #: 544.33-9
Source Temp: 190C	Threshold: 2 counts	Step #2	200	10	15.5	17.0	Int Std. #: 544.31.5
Head Pressure: 8 psi	Mass Defect: 0	Step #3	315	25	4.6	21.6	Tune Std. #: 544.23-8
	Background Mass: 35 amu	Step #4	315	0.0	16.4	38	Inj. Vol.: 1 uL
	Curve Date: 1/30/14	Inject.	45	0.0	0.5	0.5	DFTPP Area 198: 99.307%
		Inject.	300	100	2.55	3.5	DDT Breakdown: 0%
		Inject.	300	0.0	15	15.05	

Sample / File ID	Client	Inj Time	Dil	Matrix	Fraction	Method	Notes
DFT 0423C		8:47		QC	DFTPP	8270	Passed
SC 0423A		9:21		↓	BNA		Passed
BLK 0423A		10:10	STR	QC			
358328	[redacted]	11:22		WTR			
358329		12:16					
358330		13:05					
358331		13:45					
358332		14:25					
358333		15:04					
358334		15:44					
358335		16:24					
358336	[redacted]	17:04	STR	WTR	BNA	8270	

file: 133_RUN.XLS page: 16 of 61 revision: 1.0

Approved By: [signature] 1-22-09 QA Officer

Approved By: [signature] 1/23/09 Area Supervisor

Attachment 23.15
Method 8270C Selected Ion Monitoring (SIM) Instructions

Selected Ion Monitoring (SIM) allows the mass spectrometer to detect specific compounds with very high sensitivity. In SIM mode the instrument obtains data at select masses of interest instead of stepping the mass filter over a wide mass range.

To set up SIM monitoring, the compounds of interest must first be analyzed by acquiring a full GCMS scan to determine the ions to use. Two to three ions are selected for each target compound. Include the most abundant ion if it is unique to the compound. Higher mass ions are usually more specific and separated from interference.

After choosing spectral fragments (ions) to monitor, an accurate mass must be determined (±0.1 AMU). The easiest way to determine mass accuracy is by tabulating the full-scan spectra of the compound. Print the mass table and use the m/z value in the tabulation to the nearest 0.1 AMU for the SIM acquisition mass.

After SIM acquisition masses are determined for each compound, ions are grouped according to elution time. Limit the number of ions being monitored in a group to between 3 and 8. Only one group can be monitored at a time. The end-time for one group becomes the start-time for the next group. Using these groups, the important ions for a list of compounds can be monitored by switching from one group to another at the appropriate time during a chromatographic run.

Once the monitoring ions are determined and the ions grouped, set a dwell time for each group to optimize the cycle time. Obtain 15 to 20 scans across each peak. A dwell time of 50 msec for each group is a good starting point. Acquire a run with this dwell time and note the number of scans across each peak. If the number of scans is less than 10, the dwell time needs reduced. If more than 25 scans, the dwell time needs increased.

Follow every step in the analytical procedure when using the SIM. A full-scan tune is acquired and must pass at the start of each 12-hour shift. Use a calibration curve of at least five points that pass acceptance criteria. Curve concentrations will depend on compound response and client requirements. Depending upon response, the low calibration standard is generally between 0.002 ug/mL and 0.1 ug/mL. Internal standard concentration is 0.5 ug/mL.

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Volatile Organic Compounds by Purge and Trap Capillary Column Gas Chromatography/Mass Spectrometry

EPA Method 624
SW-846 Method 8260B

APPROVALS:

Area Supervisor: Diane VanMale — Date: 1-26-09
Diane L. VanMale

QA Officer: Tom C. Boocher — Date: 1-22-09

Operations Manager: Jeff P. Glaser — Date: 1/26/09

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By: Diane L. VanMale
Total Number of Pages: 42

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure uses gas chromatography/mass spectrometry (GC/MS) for determining volatile organic compounds in a variety of matrices. It can be used to quantify most volatile organic compounds having a boiling point below 200° C. Volatile water-soluble compounds have higher quantitation limits due to poor purging efficiencies. Such compounds include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides.

1.2 Nearly all matrices, including ground water, wastewater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments can be analyzed. Sample is introduced into the system using a purge and trap concentrator, or by direct injection if concentrations are extremely high.

1.3 For analysis of nonaqueous matrices, refer to TriMatrix SOP GR-04-105.

Note: Low-level bulk soil analysis by method 8260B is not allowed for state of Ohio samples under Ohio VAP (Voluntary Action Program) oversight and must not be used for Ohio VAP samples unless reported by method 8260A.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

2.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables

2.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

2.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

3.0 SUMMARY OF PROCEDURE

3.1 When using purge and trap technique, an inert gas is purged through a sample, at an ambient or slightly elevated temperature. Volatile components are transferred from the aqueous to vapor phase. The vapor is swept onto a sorbent column, where volatiles are trapped.

3.2 After purging, the sorbent column is heated and back flushed with inert gas to desorb onto a capillary column. The capillary column is temperature programmed to separate and elute components, which are then transferred to a mass spectrometer, via jet separator or a direct connection, using an open-split interface or a capillary-direct interface with a split at the injection port.

3.3 Component analytes are detected by the mass spectrometer which is capable of qualitative and quantitative analysis. Identification is done by comparing analyte mass spectra against calibration standard spectra. Quantitation is performed by comparing analyte ion response (using an internal standard) to a minimum five-point calibration.

4.0 PARAMETER OR COMPOUND LIST

4.1 Compounds available for analysis and referenced to method 8260B or method 624 are listed in Attachment 23.1 (Table 1) and Attachment 23.2 (Table 1A).

4.2 Other analytes may be determined providing an acceptable demonstration of capability is performed following every step in the procedure. Additionally, all described quality control must be within laboratory established control limits.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-105, *Closed System Purge and Trap Extraction for Volatile Organics in Soil and Waste Samples*, latest revision

5.2 TriMatrix SOP GR-03-124, *Volatile Laboratory Corrective Actions*, latest revision

5.3 TriMatrix SOP GR-15-102, *Waste Disposal,* latest revision

5.4 TriMatrix SOP GR-07-115, *Percent Solids, Gravimetric, Dried At 103-105° C*, latest revision

5.5 TriMatrix SOP GR-10-115, *Manual Integration*, latest revision

5.6 TriMatrix SOP GR-10-123, *Element™ Data Transfer and Review*, latest revision

5.7 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Whether purged or injected directly onto a column, interferences naturally present in samples can elevate reporting limits and interfere with the analysis. Naturally occurring interferences can vary considerably from site to site.

6.2 Sample contamination can also raise reporting limits or give false positives. Contamination can come from a variety of sources. Improper sampling techniques can contaminate at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through septa. A trip blank prepared from reagent water and carried through sampling and handling serves as a check on such contamination. All volatile sample storage units must contain a storage blank, which must be replaced and analyzed weekly.

6.3 During analysis, contamination can come from impurities in the purge gas or from organic compounds outgassing from the plumbing ahead of the trap, if deposited by a previously analyzed high-level sample. To minimize, the non PTFE tubing, non PTFE thread sealants, and flow controllers with rubber components in the purging device, have been eliminated where possible.

6.4 Carryover can occur whenever high and low level samples are analyzed sequentially. To reduce carryover, the autosampler rinses the purging device and sample syringe with reagent water between samples. Samples with unusually high concentrations must be followed by analysis of a reagent water blank to check

for carryover. If compounds present in high concentrations are present at any level in a subsequent sample, demonstrate that there is no carryover by reanalyzing the sample. If compounds are not present in the subsequent sample, reanalysis is not necessary.

6.5 The trap and other parts are also subject to contamination. Frequent bakeout and purging of the entire system is required. If contamination persists, the complete purge and trap system must be purged first with 100° C reagent water then if necessary, with methanol. If methanol is used, disconnect the trap or install a blank trap, as methanol will adversely affect the trap's performance.

6.6 For samples containing large amounts of water-soluble material, suspended solids, high boiling compounds, or high organohalide levels, it may be necessary to wash out purging devices with a detergent solution, rinse with reagent water then dry in a 105° C oven between analyses.

6.7 Methanol content in blanks, standards, and samples must be kept constant on the ion trap. Varying methanol can suppress analyte signal response, and can alter certain spectra (chloroethane spectra will not look like the reference spectra).

Note: The maximum methanol volume purged must be no more than 100 µL.

6.8 Corrective actions for unacceptable quality control are outlined in TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used has not been precisely defined. All chemicals must be treated as a potential health hazard. From this viewpoint, chemical exposure must be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel

7.3 The following analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromomethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds must be handled only in a hood, with disposable gloves, a laboratory coat and approved safety glasses.

7.4 Approved safety glasses must be worn in the laboratory except when inputting data at a computer terminal.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 To achieve reporting limits, a minimum of 40 mL aqueous sample (in duplicate) needs to be collected. More sample volume will be necessary if matrix spikes are required. Collect soil, sludge and solid waste samples as specified by TriMatrix SOP GR-04-105.

8.1.1 Collect all aqueous samples and liquid waste in 40 mL borosilicate glass screw-cap VOA vials with PTFE-lined silicone septa. All sample collection vials provided by TriMatrix contain acid preservative. Gently fill sample vials such that a meniscus develops at the top rim of the vial (fill to almost overflowing). To minimize analyte loss, no air bubbles must pass through a

sample as a vial is filled, or be trapped in the sample when the vial is sealed. Invert each filled and capped vial to confirm no headspace or bubbles are present. If there is headspace or bubbles larger than 5-6 mm in diameter, fill a new vial.

8.1.1.1 If vials were not supplied by TriMatrix and are not pre-preserved, fill to just overflowing, then adjust to a pH <2 by carefully adding two drops of 1:1 HCl to each vial. Seal the filled vials with a septa screw-cap lid (PTFE face must be down), then cool to 4 ±2° C.

8.1.1.2 If a sample contains residual chlorine, collect first in a 125 mL bottle, pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl the capped bottle to mix. Proceed with Section 8.1.1, then Sections 8.1.1.3 and 8.1.1.4 if applicable.

8.1.1.3 If analysis includes acrolein and/or acrylonitrile, and a project specifies it, collect additional sample in vials that do not contain preservative. Adjust to a pH of 4-5 with 1:1 HCl, then cool to 4 ±2° C.

8.1.1.4 Analysis on unpreserved or insufficiently preserved samples (where a sample has a high buffering capacity), which reference method 8260B, will not be conducted except by specific client or project request. In such cases, analysis will be conducted within 7 days, instead of 14.

Note: This is an uncommon exception to the 14-day holding time, handled on a case by case basis.

8.1.1.5 Method 624 samples may be collected with preservative as described above, or collected with no preservative. In either case, samples must be cooled to 4 ±2° C.

8.1.1.5.1 If collected without preservative, and acrolein is an analyte, sample analysis must be completed within 3 days. Acrylonitrile has a hold time of 14 days whether preserved or not. Regardless of preservation, acrolein and/or acrylonitrile results must be qualified as screening data only under method 624.

8.1.1.5.2 Samples collected for purgeable *aromatic* hydrocarbons analysis can be collected without a chemical preservative. However, the analysis must be completed within 7 days of sample collection.

8.1.1.5.3 Samples collected for purgeable *chlorinated* hydrocarbons analysis *only* can be collected without a chemical preservative. Analysis must be completed within 14 days of sample collection.

8.1.2 At least one trip blank must accompany each aqueous sample set collected from the same general site at approximately the same time, to the site and back. A trip blank is a pre-preserved 40 mL VOA vial filled at the laboratory with reagent water, sealed, and shipped to the sampling site with empty sample containers.

8.1.3 For trip blanks associated with soil, sludge and waste collection, refer to TriMatrix SOP GR-04-105

8.2 Aqueous samples and soil samples to be prepared following TriMatrix SOP GR-04-105 must be chilled to 4 ±2° C on the day of collection, and maintained at that temperature until received by the laboratory. Samples not received by the laboratory on the day of collection, must be packaged for shipment with sufficient ice to ensure a transit temperature of 4 ±2° C.

8.2.1 Once received, TriMatrix must store samples at 4 ±2° C until analysis. Aqueous, solid, and waste samples are stored separately. Each storage area must be free of organic solvent vapors.

8.2.2 With certain exceptions (noted in Sections 8.1.1.4 and 8.1.1.5), all samples must be analyzed within 14 days of collection or qualified as estimated.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Glassware and Hardware

9.1.1 Class A volumetric flasks (10 mL, 50 mL, 100 mL, 250 mL, 1000 mL)

9.1.2 Microsyringes (1.0 µL, 5.0 µL, 10 µL, 25 µL, 50 µL, 100 µL, 1000 µL)

9.1.3 Gastight luer lock syringes, 5.0 mL

9.1.4 60 or 125 mL wide mouth glass jars with PTFE-lined caps

9.1.5 Borosilicate glass vials with PTFE-lined septum caps, 20 mL and 40 mL

9.1.6 Various size PTFE-lined screw cap vials

9.1.7 Mini-inert vials, 1.0 mL for standards preparation and storage

9.1.8 Refrigerator, capable of maintaining 4 ±2° C

9.1.9 pH test strips

9.1.10 Metal spatulas

9.1.11 Analytical balance capable of reading to 0.0001 g

9.1.12 Top-loading balance capable of reading to 0.01 g

9.1.13 Pasteur pipettes (disposable) with rubber pipette bulb

9.2 Purge and trap/gas chromatograph/mass spectrometer system:

9.2.1 Concentrators

9.2.1.1 Encon (Environmental Sample Technology) concentrator conditions used:

Trap: EST Vocarb 3000
Purge: 11 minutes at 38-42 mL/minute
Dry purge: 1 minute

Desorb preheat: 245° C
Desorb: 250° C for 2.0 minutes
Bake: 10 minutes at 260° C
Valve oven: 150° C
Transfer line: 150° C

9.2.2 Autosamplers:

9.2.2.1 Archon (O.I. Analytical 4552 or Varian) – with needle sparger for heated purging of water and low-level soil samples; preheat for 1.0 minute and purge at 45° C

9.2.2.2 Centurion (Environmental Sample Technology) for water samples and MeOH preserved soil samples

9.2.3 Gas Chromatograph

9.2.3.1 Hewlett Packard Model 5890 Series II with a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
Column temperature program:45° C for 10 minutes, then to 73° C at 7° C/minute, then to 110° C at 10°, then to 220° at 20° C/minute, hold 2.25 minutes .

9.2.3.2 Hewlett Packard Model 6890 equipped with electronic pressure control (EPC), a split/splitless injection port and a capillary direct interface. Conditions used:

Injector temperature: 200° C
Transfer line temperature: 280° C
EPC setting: Constant flow mode at 0.7 mL/minute
Split ratio: 50:1
Column temperature program: 45° C for 3 minutes, then to 200° C at 15° C/minute, hold for 1 minute

9.2.4 Columns:

9.2.4.1

9.2.4.2 30 m x 0.25 mm ID, 1.4 um film thickness, narrow bore capillary column DB-VRX (J&W Scientific). Used in HP 5890 II with capillary direct interface (Section 9.2.3.1).

9.2.4.3 20 m x 0.18 mm ID, 1.0 um film thickness, narrow bore capillary column DB-624 (J&W Scientific). Used in HP 6890 Series II with capillary direct interface (Section 9.2.3.3).

9.2.5 Mass Spectrometer (Hewlett Packard 5971A or 5973 MSD) capable of scanning from at least 35-650 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact mode, and producing a mass spectrum that meets all criteria in Attachment 23.3 (Table 2), BFB Key Ion Abundance Criteria, when 50 ng of 4-bromofluorobenzene (BFB) are purged or injected onto the analytical column.

GC/MS operating conditions:

9.2.5.1 Electron energy: 70 volts (nominal)

9.2.5.2 Mass range: 35-300 amu

9.2.5.3 Scan time: 1.5 scans/second

9.2.5.4 Source temperature: 160 - 220° C

9.2.6 Data Acquisition:

9.2.6.1 The HP MSD data acquisition system is a DOS-based HP Chemstation equipped with EnviroQuant environmental data analysis software. It is also capable of plotting EICPs and has a 120,000 compound NIST spectral library.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 GC Maintenance

10.1.1 Change septa weekly or as needed

10.1.2 Change gas scrubber traps yearly or as needed

10.1.3 Clip or replace GC column as needed

10.1.4 Check column head pressure daily

10.1.5 Check gas cylinder pressure daily and change if needed

10.1.6 Replace injection port liner as needed

10.2 Mass Spectrometer Maintenance

10.2.1 Mass Selective Detector (MSD) Maintenance Schedule

10.2.1.1 Drain and replace rough pump oil every 6 months

10.2.1.2 Check diffusion pump oil annually and change if discolored or low

10.2.1.3 Clean ion source at least annually (may be needed more frequently as shown by detector performance)

10.2.1.4 Examine and replace large o-ring seal on 5971 if worn

10.2.1.5 Replace electron multiplier as needed (maximum voltage 3000)

10.2.1.6 Check calibration gas and refill as necessary

10.3 Purge and Trap Maintenance Schedule

10.3.1 Before initial use, new traps must be conditioned for one hour.

10.3.2 Check and record the purge flow weekly

10.3.3 Empty waste bottles and fill rinse bottles daily

10.3.4 Check and record the concentrator pressures weekly

10.3.5 Check and if necessary fill internal/surrogate standard module daily

10.3.6 Clean sparge tube as necessary.

11.0 CHEMICALS AND REAGENTS

11.1 Reagent water (organic free) ASTM Type II

11.2 Methanol (purge and trap grade)

11.3 Certified stock standard materials (96% pure or greater) or certified stock solutions

11.4 1:1 Hydrochloric acid

11.5 Sodium bisulfate, $NaHSO_4$, ACS reagent grade or equivalent

12.0 STANDARDS PREPARATION

12.1 All laboratory standards must be recorded in the standards logbook and/or in the laboratory information management system (Element™). Information including the vendor, lot number, concentration, purity must be recorded. Each standard is given a unique ID number. All vendors and standard concentrations provided below are recommended and subject to change.

12.2 Hold Time and Storage Requirements

12.2.1 Unopened ampoules of purchased standard solutions and neat materials used in stock standards preparation, may be kept for up to 12 months from the date received or as directed by the manufacturer.

12.2.2 Due to evaporative loss and reactivity, laboratory prepared stock, and nongaseous intermediate stock standards are given an expiration date of six months. An intermediate stock gas standard typically requires weekly replacement. Within expiration dates, all standards must not be kept past acceptable performance.

12.2.2.1 Continuing calibration standards must meet the calculated percent difference or drift criteria of Section 14.6 to be acceptable. This requirement applies to all analytes, both gaseous and non-gaseous. Once this criterion has been exceeded, the intermediate stock and/or the stock must be replaced unless exceedance is due to instrument performance degradation.

12.2.2.2 Dichlorodifluoromethane will generally be the first gaseous compound to fail.

12.2.2.3 Because 2-chloroethyl vinyl ether is reactive, it will typically be among the first of the nongaseous compounds to fail.

12.2.3 Store all prepared standards with minimal headspace, protected from light, in PTFE sealed screw-cap vials at -10° C or below. Purchased standards are stored as instructed by the manufacturer. Standards must be stored separately from samples.

12.3 Stock Standard Preparation

12.3.1 Stock standards are prepared from neat materials or certified solutions. All purchased solutions must be accompanied by a Certificate of Analysis. Two separate standard sources must be obtained. This applies to both purchased standards and neat compounds. One set will be used to prepare calibration, laboratory fortified blanks (blank spikes) and matrix spikes, and the other will be used for laboratory control samples secondary calibration verifications). The two must be prepared from dissimilar or entirely separate sources. It is permissible to purchase different lots from the same vendor. However, a dissimilar lot means preparation from dissimilar chemical lots. Not prepared twice from the same lot. It is not necessary to purchase/prepare Internal Standard/Surrogate mixes from separate sources.

12.3.2 When preparing stock standards from neat material, the mass weighed out may be used without mathematical correction when the compound has a purity greater than or equal to 96%. Prepare stock standard solutions in methanol.

12.3.3 Gravimetric Method:

12.3.3.1 A 10,000 mg/L stock solution: 0.5 g of each solid analyte (neat) is transferred to a 50 mL volumetric flask partially filled with methanol which has been tared on an analytical balance. Record mass to the nearest 0.0001 g, make sure the solid is dissolved then fill to the mark with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the flask's neck and transfer the remaining solution to a PTFE-capped vial.

12.3.4 Purchased Commercial Stock Standards:

12.3.4.1 The following standards are combined to make Stock Standard I.

- Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH
- Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH
- Mix C: Acrolein & Acrylonitrile @ 2.0 mg/mL in MeOH
- Mix D: Ketone Mix @ 2.0 mg/mL in MeOH
- Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH
- Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH

- Mix G: Internal Standard and Surrogate Standard Mix at 5.0 mg/mL in MeOH.
- Mix H: BFB Standard Mix at 2.0 mg/mL in MeOH.

12.3.4.2 The following standards are combined to make Stock Standard II.

- Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH
- Epichlorohydrin at 1.0 mg/mL in MeOH.
- 2-Chloro-1,3-butadiene (chloroprene) at 2.0 mg/mL in MeOH.
- N-butyl acetate at 2.0 mg/mL in MeOH.
- Methyl Methacrylate at 2.0 mg/mL in MeOH.

12.4 Intermediate Stock Standards

12.4.1 8260B Intermediate Stock Standard I

12.4.1.1 This solution is prepared in a 1.0 mL mini-inert vial. Add 650 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.1, add the following volumes to produce 1.0 mL of a 100 mg/L intermediate stock standard:

50 µL Mixes A, B, C, D, and E
100 µL Mix F

12.4.2 Appendix IX Intermediate Stock Standard II

12.4.2.1 This standard is also prepared in a 1.0 mL mini-inert vial. Add 425 µL of methanol to the mini-inert vial. From the mixes specified in Section 12.3.4.2 and the neat stocks from 12.3.3.2, adding the following quantities will produce 1.0 mL of a 250 mg/L intermediate stock standard:

40 µL Mix A
100 µL Epichlorohydrin
50 µL 2-Chloro-1,3-butadiene
100 µL Isopropanol
100 µL Ethyl methacrylate
50 µL Methyl methacrylate
50 µL N-Butyl acetate
100 µL Cyclohexanone

12.5 Working Standards

12.5.1 Internal Standard/Surrogate Intermediate Stock Standard III

12.5.1.1 This standard is prepared at a concentration of 200 mg/L. Fill a 25 mL volumetric flask approximately three-quarters full with methanol. Add 1.0 mL of mix G specified in Section 12.3.4.1 and dilute to volume with methanol. Cap and invert three times to ensure proper mixing. Discard the contents in the flask's neck, and

transfer the remaining contents to a PTFE-capped vial. The purge and trap auto sampler adds this standard automatically to samples during the purge cycle.

12.5.2 BFB Intermediate Stock Standard IV

12.5.2.1 This standard is prepared in a 1.0 mL mini-inert vial. Add 990 µL of methanol to the mini-inert vial. Add 10 µL of mix H specified in Section 12.3.4.1 to produce 1.0 mL of a 20 mg/L intermediate stock standard. This standard is used for performing BFB tunes.

12.5.3 Initial calibration and calibration verification standards are prepared by spiking different volumes of intermediate stock standard into a 50 mL volumetric flask or a 5.0 mL gas-tight syringe, depending on the auto sampler used. These standards are prepared as needed and not stored. Calibration verification standards are typically prepared at 40 ug/L for all except Appendix IX compounds, which are prepared at 100 ug/L. Default spike volumes used in preparing the initial and continuing calibration verification standards are discussed in Section 14. Stock volumes necessary to prepare other concentrations can be calculated using the following formula:

$$V_s = \frac{C_f \times V_f}{C_s}$$

where:
V_s = Volume of stock standard to inject (µL)
C_f = Final concentration of working standard (ug/L)
V_f = Final volume of working standards (mL)
C_s = Concentration of stock standard (ug/mL)

12.6 Matrix Spike Standards

12.6.1 The second source intermediate stock standards prepared in Section 12.4 are used for matrix spiking. Matrix spikes are prepared using the formula given in Section 12.5.3. Matrix spike standards must be prepared at concentrations near the midpoint of the calibration, typically the same concentration as the continuing calibration standard. Other concentrations may be specified for certain projects.

12.6.2 The following analytes are default matrix spike compounds: 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene and benzene. Additional analytes are reported if requested by clients.

12.6.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.6.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.6.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

12.7 Laboratory Fortified Blank/Blank Spike (LFB/BS) Standards

12.7.1 Primary source intermediate stock standards prepared in Section 12.4 are used for blank spikes. An LFB/BS is prepared using the calculation given in Section 12.5.3. LFB/BSs must be prepared at the same concentration as the matrix spike.

12.7.2 The same analytes used in matrix spikes must be used for the LFB/BS. Additional analytes are reported if requested by the client. The same analyte list must be reported for matrix spikes and the LFB/BS.

12.7.2.1 Samples that must meet data quality objectives of the State of Wisconsin must be spiked with all target analytes.

12.7.2.2 Samples the must meet data quality objectives of the Department of Defence must be spiked with all target analytes.

12.7.2.3 Any client sample requiring all target analytes to be quality control spiked must be input to the laboratory information management system (Element™) to spike all target analytes.

13.0 SAMPLE PREPARATION

13.1 Soils are prepared in accordance with TriMatrix SOP GR-04-105 with reference to SW-846 method 5035.

13.2 Waters are prepared using the Archon or Centurion Autosampler

13.2.1 Samples analyzed on these autosamplers require no preparation. The 40 mL vial is loaded directly into the autosampler.

13.2.2 If a dilution is required, the sample must be diluted into the appropriate size volumetric flask and an aliquot transferred to a 40 mL VOA vial for analysis. An ideal dilution results in an analyte concentration in the upper half of the calibration range.

13.2.3 The autosampler withdraws a five mL aliquot from the 40 mL vial, and adds the required internal standards and surrogates before transferring to the sparge vessel.

14.0 CALIBRATION PROCEDURES

14.1 Initial Calibration

14.1.1 A 7-point calibration must be run before sample analysis. The low point of the initial calibration curve must be at or below the minimum reporting limit for each analyte. The high point defines the linear range. Calibration standards must be matrix matched to samples whenever possible, and analyzed under the same operating conditions. All target compounds must be included in the initial calibration. A calibration must be analyzed only after a successful BFB analysis.

14.1.2 For the Archon or Centurion autosampler, prepare standards in 50 mL volumetric flasks using 10 times the volumes listed in Table 3 and 3A. Transfer to a 40 mL vial, then load in the autosampler. The autosampler takes a 5.0 mL aliquot and adds 1.0 µL of internal

standard/surrogate mixture before purging. The Archon and Centurion are used to analyze aqueous samples, and methanol extracts after dilution into reagent water.

Note: **SODIUM BISULFATE ADDITION DEGRADES 2-CHLOROETHYL VINYL ETHER IN STANDARDS AND SAMPLES. TRIMATRIX LABORATORIES HAS DEMONSTRATED THAT ACCEPTABLE RESULTS FOR THIS COMPOUND ARE NOT ACHIEVED WITH THIS TECHNIQUE.**

14.2 Calculate Response Factors (RF) for each compound using the quantitation and internal standard ions listed in Attachments 23.1 or 23.2. Calculate RF using the following formula:

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured
C_{is} = Concentration of the specific internal standard

14.2.1 For compounds not listed in Attachments 23.1 or 23.2, choose a major ion that is dissimilar to any potential coeluting or interfering ions, for the quantitation ion. Choose an internal standard with a retention time closest to the compound being measured.

14.2.2 Calculate average response factors as follows:

$$RF_{avg} = (RF_1 + RF_2 + RF_3 + \ldots RF_n)/n$$

where:
RF_{avg} = Average calibration response factor
RF_1 = Calibration response factor for standard 1
RF_2 = Calibration response factor for standard 2
RF_3 = Calibration response factor for standard 3
RF_n = Calibration response factor for standard n
N = number of calibration standards

14.3 The average RF for all calibration points must be calculated for each compound. Five System Performance Check Compounds (SPCCs) must be checked against a minimum average RF limit.

14.3.1 These SPCCs are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. These minimum acceptable average response factors are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	>0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

14.3.2 An SPCC monitors compound instability and degradation caused by contaminated lines or active sites in the system. Examples of such occurrences are:

14.3.3 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

14.3.4 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

14.3.5 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated purge-and-trap transfer lines and/or active sites in trapping materials.

14.3.6 All non-SPCC compounds must adhere to the following minimum average RF limits:

14.3.6.1 The average RF of Alcohols, 1,4-Dioxane, and Epichlorohydrin must be at least 0.001 to be acceptable.

14.3.6.2 The average RF of all other compounds must be 0.010 or higher.

14.4 Always check for carryover and memory effects (ghosting) from high concentration standards or samples when analyzing for late eluting compounds such as hexachlorobutadiene and 1,2,3-trichlorobenzene. Adequate purge chamber rinsing minimizes such effects. Newer purge-and-trap systems address this problem with a bakeout step following trap desorb and newer traps retain less moisture. However, high concentrations can still affect subsequent runs.

14.5 Calculate Percent Relative Standard Deviation (%RSD) for all compounds. Using the initial calibration average RF, calculate percent RSD using the following formula:

$$\text{Percent RSD} = \frac{SD}{X} \times 100$$

where:

RSD = Relative standard deviation.
SD = Standard deviation of average RF for a compound.
X = Mean of the five initial response factors for a compound.

14.5.1 All compounds must have a percent RSD of ≤15%. Six compounds are used as Calibration Check Compounds (CCCs): 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethyl benzene, vinyl chloride. These compounds **must** have a percent RSD of ≤30%. If percent RSD is greater than 30 percent for any CCC, corrective action must be initiated and the entire system recalibrated.

14.5.1.1 If percent RSD of a compound is 15% or less, the calibration is assumed to be linear through the origin, and an average response factor may be used for quantitation.

14.5.1.2 If percent RSD of any compound is greater than 15%, then a regression curve of area ratio (Area analyte/Area IS) against concentration must be constructed, using first or higher order regression fit. Analysts must select a regression algorithm providing the best fit.

14.5.1.2.1 Generally this will be a first order linear regression using the following equation:

$C_i = A_i\text{-}b)/m$

Where:
C_i = Concentration in ug/mL
A_i = Area ratio ($A_{analyte}/A_{IS}$)
b = Intercept of the regression curve
m = Slope of the regression curve
i = Compound "i"

14.5.1.2.2 Second order regression must only be used for analytes with a definite quadratic response, as results produced by this algorithm may give erroneous results at concentrations near the reporting limit. A minimum of six calibration points are required to use second order calibration curves. The Enviroquant data processing software performs all regression functions, and uses coefficient of determination (r^2) to measure calibration validity. To be considered an acceptable calibration curve, r^2 must be 0.990 or higher.

14.5.1.3 If initial calibration criteria are not achieved using all calibration points run, the lowest or highest point on the curve may be dropped, provided enough points remain for the calibration model chosen.

14.5.1.3.1 A minimum of six calibration points are required to use second order regression curves. Five date points are needed for first order.

14.5.1.3.2 The lowest calibration point must be at the analyte reporting limit. The low point can not be dropped without elevating reporting limits.

14.5.1.3.3 Dropping the highest point shortens the calibration range, which could lead to a greater number of sample dilutions.

14.6 Initial calibration verification must be performed immediately after the initial calibration by running a second-source calibration verification (SCV) standard at 40 ug/L.

14.6.1 The SCV must be run after each initial calibration.

14.6.2 The SCV must include all targeted analytes.

14.6.3 Acceptance criteria for all analytes are ±25% of expected value unless otherwise specified in the laboratory information management system (Element™).

14.6.4 If any SCV analyte fails, locate and correct the problem (up to and including remaking the SCV solution) then repeat the SCV analysis successfully.

14.6.5 If the second SCV attempt fails, review the initial calibration solutions and remake or obtain a new calibration stock then repeat the SCV successfully.

14.6.5 Sample analysis may not begin until an acceptable initial calibration and SCV have been run successfully.

14.7 Every 12 hours a 40 ug/L continuing calibration verification (CCV) containing each compound quantified must be run after the BFB (Section 14.1). If a calibration has just been run, then the 40 ug/L standard from the calibration is used. Analysis of the CCV verifies instrument sensitivity and confirms acceptability of the initial calibration curve. A CCV is verified the same as an initial calibration, by checking SPCCs and CCCs. The CCV must be analyzed and quantified against a regression fit or average RF under conditions identical to the initial calibration.

14.7.1 System Performance Check Compounds (SPCC): This is the same check applied during initial calibration. SPCC compound response factors must be as listed in Section 14.3.1 (Section 14.2 for calculating response factors) In addition, non-SPCC compounds must meet the initial calibration criteria (Section 14.3.6) If minimum response factors are not met, the system must be evaluated and corrective action taken before sample analysis begins. Possible problems include standards degradation, injection port contamination and/or buildup in the first six inches of the analytical column and active sites in the column or chromatographic system.

14.7.2 Calibration Check Compounds (CCCs): After SPCCs are met, CCCs are used to check initial calibration validity. Calculate percent drift or percent difference using the following equations:

Percent Drift $= [(C_c - C_1)/C_1]*100$

where:
C_1 = CCC concentration
C_c = Measured concentration using selected average RF or regression.

Percent Difference $= [(RF_{ccc} - RF_{avg})/ RF_{avg}]*100$

where:
RF_{avg} = Average Response Factor from Initial Calibration
RF_{ccc} = Response Factor from Continuing Calibration

14.7.3 If CCC percent drift or difference does not exceed ±20%, the initial calibration continues to be valid. If acceptance is not met, then corrective action must be taken. An acceptable CCC **MUST** be run before sample analysis can begin. If CCC compounds are not target analytes, then all target analytes must be used to meet the ±20% control limits.

14.7.4 For non-CCC compounds in the CCV, the following criteria must be used.

14.7.4.1 All percent differences or drifts must not exceed ±25%, with the following exceptions:

14.7.4.2 Alcohols, ketones, 2-Methylnaphthalene, 2-Chloroethyl vinyl ether, 1,4-Dioxane, Vinyl acetate, and Iodomethane must not exceed ±40%.

14.7.4.3 Also, if non-CCC compound responses are high and out of control, then non-detect sample results for those compounds may still be reported since there is no question of analyte sensitivity for that day.

14.7.5 It is not permitted to choose a quantitation technique dissimilar to that used during initial calibration to achieve CCC criteria. For example: An analyte initial calibration %RSD was within ±15% drift, and average RF is used for quantitation. The last CCC is not acceptable for percent drift. To attain a passing CCC, it is not permitted to switch to a regression curve in an attempt to pass the CCC, even if the regression curve passes coefficient of determination criteria.

14.7.6 Internal standard responses and CCV retention times must be evaluated during or immediately after data acquisition.

14.7.6.1 If retention time for an internal standard changes by more than 30 seconds compared to the 40 ug/L initial calibration, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.2 If the area for an internal standard changes by a factor of two (-50% to +100%) compared to the 40 ug/L initial calibration standard, the system must be inspected for malfunctions. Corrections must be made as needed.

14.7.6.3 When corrections are made, reanalysis of samples analyzed while the system was malfunctioning are necessary.

15.0 ANALYTICAL PROCEDURE

15.1 Before initial use, a Vocarb 3000 trap must be conditioned for at least one hour by baking at 260° C and purging with helium. If other trapping materials are substituted for the Vocarb 3000, follow the manufacturer's conditioning recommendations. After periods of inactivity, GC columns may be run through the temperature program, or ramped to the final temperature and held for 15 minutes.

15.2 Set up the autosampler, purge-and-trap, and GC/MS as instructed in Section 9.0. Sample prep and purging conditions are specific to each matrix. The procedure for soil samples is described in TriMatrix SOP GR-04-105.

15.3 BFB Tuning

15.3.1 At the beginning of every 12-hour shift 4-Bromofluorobenzene (BFB) must be used to tune the mass spectrometer. Analyze the BFB and compare the spectra obtained to the criteria given in Attachment 23.3. No other analysis may begin prior to running an acceptable BFB tune.

15.3.2 The standard prepared in Section 12.5.2.1 contains 20 mg/L of 4-Bromofluorobenzene (BFB).

15.3.2.1 For the Archon autosampler, spike 25 µL into a 50 mL volumetric flask containing reagent water. Transfer the contents to a 40 mL sample vial. Alternately, 2.5 µL may be directly injected into the gas chromatograph.

15.3.2.2 The result will be to inject 50 ng of BFB into the GC/MS system. All subsequent analysis must commence within 12 hours of the BFB injection. BFB must be analyzed using the same acquisition parameters as standards and samples.

15.3.3 The following evaluation sequence must be used to determine BFB tune performance.

15.3.3.1 All BFB tunes must be initially evaluated using the CLP BFB tuning procedure, which takes the scan average at the peak apex and two scans immediately before and after the apex, followed by a background subtraction.

15.3.3.2 If the above does not generate an acceptable BFB tune, then take a scan average of the entire peak, followed by a background subtraction.

15.3.3.3 If the above two options do not yield an acceptable BFB tune, then evaluate with a single scan at the apex.

15.3.3.4 If none of the above give an acceptable BFB tune, then corrective action is required. The first step is to reinject and reanalyze the same BFB standard. Next, prepare a new BFB working solution, then inject and analyze. The last step is to optimize the tuning acquisition parameters by tweaking manually or by performing a new autotune. Once acquisition parameters are optimized, restart the tuning sequence.

15.4 Initial/Continuing Calibration

15.4.1 After a successful BFB analysis, an initial curve or continuing calibration verification must be analyzed (Section 14.0). Initial and continuing calibrations must match sample methanol and/or sodium bisulfate concentrations wherever possible, and be run under the same analytical conditions as samples. Follow initial or continuing calibration with the quality control described in Section 18.0.

15.5 Sample Analysis

15.5.1 For aqueous samples analyzed using the Archon or Centurion autosampler, the 40 mL sample collection vial is placed directly into the autosampler without opening. The autosampler removes 5.0 mL and transfers it to the purge vessel automatically. Internal standards and surrogate are added by the autosampler as well. The purge sequence will start automatically, using the operating conditions in Section 9.0. At the end of the purge cycle, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2 Low concentration analysis for bulk and sodium bisulfate preserved soils (approximate concentration range 0.010 - 1.0 mg/kg):

15.5.2.1 If a sample is preserved in sodium bisulfate, remove the vial from storage, and let warm to room temperature. Agitate the vial gently so contents move freely and stirring will be effective. For bulk soils prepared in the laboratory, 5.0 mL of reagent water and a PTFE stir bar must be added during preparation. Place the vials in the autosampler.

15.5.2.2 The autosampler adds 10 mL of organic-free reagent water, internal standards, and surrogates before initiating the purge sequence. Other volumes may be used but **it is imperative that all samples, blanks and calibration standards have exactly the same added volume of organic-free reagent water**.

15.5.2.3 Before purging, vials are heated to 45° C and held for 1.0 minute. Purge follows the operating conditions provided in Section 9.0. Heat and stir using the magnetic

stir bar, for the entire purge cycle. After purging, analytes remain on the adsorbent trap, and are ready for desorption to the gas chromatograph.

15.5.2.4 This technique must be used only by client request.

15.5.4 The following is for extraction of high concentrations (greater than 1 mg/kg), from solid and oily waste:

15.5.4.1 Remove sample methanol extracts from storage and let warm to room temperature. Using a microsyringe, transfer an appropriate extract volume to organic-free reagent water in a 5.0 mL syringe or into a 50 mL volumetric flask. Minimally a 1:50 dilution is done.

15.5.4.2 Transfer the diluted extract to a 40 mL vial and place in the autosampler. The autosampler will add 10 mL of organic-free reagent water if using the Archon autosampler and internal standards/surrogates before purging.

15.5.4.3 Report soil results on a dry weight basis. Report waste results on a wet weight basis.

15.5 Sample Desorption

15.5.1 After the 11 minute purge, the system will automatically advance to the desorb mode. The trap will preheat to 245° C without desorption gas flow. When the trap reaches this setpoint, it will desorb at 20 mL/minute for two minutes. Desorbing initiates the gas chromatograph oven temperature program, and data acquisition begins.

15.6 After desorbing, the trap is reconditioned by baking out at 260° C (or the temperature recommended by the trap packing material manufacturer). After 10 minutes, the trap heater is turned off and purge flow through the trap is halted. When the trap is cool, the next analysis can begin.

15.7 If any compound response exceeds the calibration range, prepare a sample dilution. An ideal dilution will result in analyte response at midrange in the calibration. However, a dilution with a response in the upper 60% of the range is acceptable.

15.7.1 For aqueous sample dilutions, prepare a sample dilution from an aliquot of the second (duplicate) 40 mL vial or from unused sample saved under zero headspace from the first vial. A dilution may be prepared in a 5 mL syringe (for 5.0 mL purge volumes) or in a volumetric flask (50 mL or larger). The minimum volume that can be diluted is 1.0 mL. The total volume purged must equal the volume used for calibration standards (5.0, 10 or 25 mL). Organic-free reagent water must be used for all dilutions. Matrix-matching must be done by including the same methanol volume to dilutions as used in calibration standards. Pour the contents into a 40 mL vial then load into the autosampler, which will automatically add internal standards/surrogates.

15.7.2 For bulk soil dilutions, prepare a 1.0 g sample instead of 5.0 g if the expected concentration is within the calibration range. If a larger dilution is needed, refer to TriMatrix SOP GR-04-105.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Qualitative Analysis:

16.1.1 An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard. Standard spectra are obtained from calibration standards.

16.1.1.1 Two criteria must be satisfied to verify analyte identification:

16.1.1.1.1 Elution at the same GC relative retention time (RRT) as the standard.

16.1.1.1.2 Positive correlation of the sample analyte mass spectrum with the standard.

16.1.1.2 The sample component RRT must compare within ±0.06 RRT units of the daily continuing calibration standard. If coelution interferes with accurate component RRT assessment from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles, for ions unique to the compound.

16.1.1.3 All ions present in standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%), are automatically checked by the software to be present in the sample spectrum. Relative ion intensities must agree within plus or minus 20%, between standard and sample spectra.

Example: For an ion having a relative intensity of 50 percent in standard spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2 For sample compounds not associated with a calibration standard, a library search can be made for tentative identification. Only after visual comparison of sample spectra with the nearest library searches will an analyst assign a tentative identification. When a tentatively identified compound (TIC) cannot be identified by name, a generic description will be given to help identify functional groups. These TICs will have names such as:

Name	CAS #
Unknown Alcohol	xx-xx-xx
Unknown Freon	xx-xxx-xxxx
Unknown Ketone	xx-xxx-xx
Unknown Acid	xxx-xx-xxxx
Unknown Hydrocarbon	xxx-xxx
Unknown Glycol Ethers	xxx-xx-x
Unknown Substituted Benzenes	Xxxxx
Hydrocarbons (sub. Benzenes)	xxxxx-x-x
Hydrocarbons, Total	Xxxxxx

Guidelines for making a tentative identification are:

16.1.2.1 Relative intensities of major ions in the library spectrum (ions >10% of the most abundant ion) must be present in a sample spectrum.

16.1.2.2 Relative intensities of major ions must agree within ±20%. As an example: For an ion having a relative intensity of 50 percent in library spectra, corresponding sample ions must be between 30 and 70 percent.

16.1.2.3 Library spectra molecular ions must be present in a sample spectrum.

16.1.2.4 Sample spectra ions not present in the library spectrum must be reviewed for the presence of background contamination or coeluting compounds.

16.1.2.5 Library spectra ions not in sample spectra must be reviewed for possible subtraction from the sample spectrum, because of background contamination or coeluting peaks. Data system library reduction can sometimes create such discrepancies. When TIC searches are performed, the criteria for determining whether or not a peak is a TIC are:

16.1.2.5.1 The peak in question must be >10% of the nearest internal standard.

16.1.2.5.2 The top 10 potential TICs must be reviewed, unless otherwise required. The match quality must be 70 percent (corresponding to a fit of 700 on the Ion Trap) or higher, to report a positive identification in the absence of interfering peaks. If there are interfering peaks, analyst discretion must be employed when reporting positive matches.

16.1.2.5.3 Concentrations <1 ug/L or <0.01 mg/kg (assuming a nominal sample extraction volume or mass, and instrument response) will not be reported as a TIC unless otherwise specified for the project, and deemed achievable following supervisor data review.

16.1.2.6 Concentrations obtained must be reported indicating the values are estimated, and are flagged with an E. Calibrated analytes not part of a client's target analyte list will also be reported when TIC's are requested. They are to be given a Q flag (quantitated) if the concentration is within the calibration range or CE flag (curve estimated) if outside the range. If no valid identification can be made, the compound must be reported as an unknown aromatic, hydrocarbon or other class, if classification is possible.

16.2 Quantitative Analysis

16.2.1 Quantitation is performed using the same technique used during initial calibration (average response factor or regression). Use analyte area and internal standard ions as specified in Attachment 23.1 (Table 1A) and Attachment 23.2 (Table 1A), and the equations below.

16.2.1.1 Aqueous Samples

$$\frac{A_x \times C_{is}}{A_{is} \times RF} \times DF = ug/L$$

where:
A_x = Area of the characteristic ion for the target compound
A_{is} = Area of the characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard in ug/L
RF = Average Response Factor
DF = Dilution factor

16.2.1.2 Low Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{5\ mL}{W \times \%S} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
W = Wet weight of sample (g)
%S = Percent solids in decimal form (example: 0.90, not 90%). Used to calculate dry weight results for soils and sludges. Wastes are calculated on a wet weight basis only. Refer to TriMatrix SOP GR-07-115.

16.2.1.3 High Level Soil Samples

$$\frac{mg}{kg} = \frac{A_x \times C_{is}}{A_{is} \times RF} \times \frac{DF}{\%S} \times \frac{V}{W} \times \frac{L}{1000\ mL} \times \frac{mg}{1000\ ug} \times \frac{1000\ g}{kg}$$

where additionally:
V = Volume of solvent added to sample during purging or dilution (mL)
W = Wet weight of sample purged or diluted (g)

16.2.1.4 Equations given above will also be used to quantitate TICs, substituting total ion areas for A_x and A_{is}. The internal standard chosen must be the one closest in elution time to an unknown peak, provided it is free from most interferences. C_{is} remains the same and RF will be a default of 1.0.

16.3 All manual integration must be performed with strict adherence to TriMatrix SOP GR-10-115.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts running sample sets are responsible for correctly filling in, handing in and filing associated paperwork. It is essential to perform these tasks to provide defensible data and client reporting.

17.2 Reporting to the laboratory information system must be done in accordance with TriMatrix SOP GR-10-123.

17.3 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in and archived correctly. The time each analyst has sample possession must be accounted for on the form.

17.3 All laboratory hardcopy (including CoC forms) must be archived appropriately and correctly.

17.3.1 All run and maintenance logbooks must be filled in completely and correctly. Corrections must be made with a lineout, not a writeover and must be dated and initialed. Blank lines must be Z'd out, initialed and dated.

17.3.2 All tunes, calibrations and continuing calibration verification runs must be placed in the correct archival folder.

17.3.3 All LIMS reports must be kept and placed in the correct archival folder.

17.4 The following are required for data packages requiring CLP-like deliverables:

17.4.1 Copies of all tunes including the EICP that has BFB ion peaks and ion abundance results and requirements, including mass listings, and associated Form Vs

17.4.2 Copies of all associated curves, including raw data quantitation reports, chromatograms, and associated Form VIs

17.4.3 Copies of all associated continuing calibration verifications, including raw data quantitation reports, chromatograms, and associated Form VIIs

17.4.4 Copies of all sample raw data, including all applicable quality control samples, quantitation reports, including triple plots for positive sample results, chromatograms and all associated TIC reports when requested

17.4.5 Method Blank Summary Form IVs

17.4.6 Internal standard recovery Form VIIIs

17.4.7 Copies of all applicable supporting information including: internal chain of custody forms, run and standard logbooks, method detection limits and extraction summaries

Note: For most projects, Forms I, II and III will be included with the deliverables package.

17.5 Rounding and significant figure adjustment is only performed on the final reported result by Element™.

18.0 QUALITY ASSURANCE

18.1 Quality control requirements include the following previously covered requirements and the quality control that follows:

18.1.1 BFB (Section 15.3)

18.1.2 Initial Calibration (Section 14.1)

18.1.3 Initial Calibration Vericiation (Section 14.6)

18.1.3 Continuing Calibration (Section 14.7)

18.2 Method Preparation Blanks (BLK)

18.2.1 After the BFB and initial calibration or continuing calibration have been run, a purged blank is required before sample analysis showing the analytical system to be free of interference and contamination.

18.2.2 The blank concentration must be at or below the maximum acceptance limit listed in the laboratory information management system. However methylene chloride and acetone may be up to five times the reporting limit unless further restricted by specific data quality objectives.

18.2.3 At a minimum, a BLK is run every 12-hour shift. The BLK must be run more frequently if carryover is suspected from a high concentration sample analysis or if laboratory contamination is in question. The BLK must be carried through all steps of sample preparation and analysis, including the addition of internal standards/surrogates spiking.

18.2.4 The BLK must be prepared in the same matrix as samples:

18.2.4.1 For methanol extractions, the BLK must be prepared as for a methanol extraction using a 1:50 ratio of methanol.

18.2.4.2 If samples are prepared with sodium bisulfate preservative, the BLK must also be prepared with sodium bisulfate.

18.2.4.3 For low-level soil analyses, the BLK must be prepared in a clean solid matrix.

18.3 Internal Standards

18.3.1 Internal standard responses and retention times in all runs following continuing calibration verification must be evaluated during or immediately after data acquisition. The retention time for internal standards must be within ±30 seconds from the current 12-hour continuing calibration standard. The quantitation ion area for all internal standards must stay within a factor of two (-50% to +100%) from the current 12-hour continuing calibration verification.

18.3.2 If at any time an internal standard fails the -50% to +100% area criteria, the ability to accurately quantitate analyte is reduced. Every effort must be made to prevent an internal standard failure, including sample dilution and reanalysis. Refer to TriMatrix SOP GR-03-124, for when an internal standard fails. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.4 Surrogates

18.4.1 All samples and quality control must be spiked with surrogates. Until thirty samples of a given matrix have been analyzed, default recovery limits of 70 - 130% will be used. Once thirty samples of a given matrix have been analyzed, in-house recovery limits will be generated. At a minimum, surrogate acceptance limits must be updated annually on a matrix by matrix basis. Calculate surrogate recovery for each analysis. If recovery or precision is not within acceptable limits, consult TriMatrix SOP GR-03-124 to determine when and how data is qualified.

18.4.1.1 High recovery may be due to a co-eluting matrix interference from the sample. Examine the chromatogram for evidence of co-elution. No corrective action is required in this instance.

18.4.1.2 Low recovery may be due to poor purge efficiency. This should be verified by re-purging the sample if hold times permit.

18.4.1.3 If surrogate recovery in a purge blank (BLK) is below the lower control limit, only samples with failing surrogate recoveries will require re-purging. If BLK

surrogate recovery is above the upper control limit, no corrective action is required as long as sample surrogate recoveries are acceptable.

18.4.1.4 If surrogate recovery fails in the MS/MSD, re-analysis is only required if LFB/BS spike recovery also fails. If the LFB/BS is still out-of-control after re-analysis, all associated samples must be re-analyzed.

18.4.2 Calculate upper and lower control limits for each surrogate. This must be done as follows:

Upper Control Limit (UCL) = p + 3s
Lower Control Limit (LCL) = p - 3s

where
p = average percent recovery
s = standard deviation of the average

18.4.3 Two standard deviations will be used when three give a negative lower control limit.

18.4.4 If recovery fails, refer to TriMatrix SOP GR-03-124. If many samples are out-of-control for no apparent reason, the mass spectrometer must be inspected for malfunctions and appropriate maintenance performed. After maintenance has resolved the problem, samples run while the instrument was malfunctioning must be reanalyzed.

18.5 Laboratory Fortified Blank/Blank Spike (LFB/BS)

18.5.1 An LFB/BS is required with each 12-hour shift or with each batch of up to 20 samples, whichever is more frequent. The LFB/BS serves as a check of methanol extraction/purging efficiency should matrix spike recoveries not be within quality control limits.

18.5.1.1 When possible, the daily CCV doubles as the blank spike.

18.5.1.2 The LFB/BS is run at the beginning of the 12-hour shift following analysis of the BLK.

18.5.1.3 Recoveries must be calculated and compared to LIMS control limits. Analysis must be stopped and the problem corrected if recoveries are outside quality control limits. Samples analyzed in a batch with a failing LFB/BS must be reanalyzed for failed analytes. If this is not possible due to holding time or lack of sample, data for the failed analytes must be qualified as estimated.

18.5.1.4 An LFB/BS may be from the same source as the calibration or from the second-source used to verify the intial calibration.

18.5.2 At a minimum, the LFB/BS must contain the following analytes:

18.5.2.1 1,1-Dichloroethylene

18.5.2.2 Trichloroethylene

18.5.2.3 Chlorobenzene

18.5.2.4 Benzene

18.5.2.5 Toluene

Note: As noted previously, where project specifications require, all target analytes must be in the matrix spike, matrix spike duplicate and LFB/BS.

18.5.3 Until at least twenty blank spikes have been analyzed in a given matrix, recovery will be validated against default recovery limits of 70 - 130%. The blank spike must be prepared in the same matrix as samples:

18.5.3.1 For methanol extractions, the blank spike must be prepared as for a methanol extraction using a ratio of 1:50 of methanol.

18.5.3.2 If samples are prepared with sodium bisulfate preservative, the blank spike must also be prepared with sodium bisulfate.

18.5.3.3 For low-level soil analyses, the blank spike must be prepared in a clean solid matrix.

18.5.4 If an LFB/BS is out-of-control, the problem must be immediately identified and corrected before samples can be run.

18.5.4.1 The LFB/BS verifies that out-of-control compounds in an MS or MSD are the result of matrix interference rather than extraction or purging error.

18.5.4.2 Failure of any targeted analyte in an LFB/BS requires corrective action depending upon the failed parameter. Every effort must be made to determine the reason for failure (spike, extraction or purge error). Appropriate corrective action must then be taken.

18.5.4.3 Take corrective action in accordance with TriMatrix SOP GR-03-124.

18.5.4.4 Samples may not be reported without an acceptable LFB/BS.

18.6 Matrix Spikes (MS, MSD)

18.6.1 To assess extraction and purge efficiency from specific matrices, extract a Matrix Spike (MS/SPK) and Matrix Spike Duplicate (MSD/SPK) at least once every 20 samples prepared for each matrix. If less than 20 samples are analyzed in a month, at least one matrix spike and one matrix spike duplicate is required. Generally, matrix spikes are analyzed at the end of the 12-hour shift. Running matrix spikes at the end of the shift helps document the instrument is still functioning correctly.

18.6.2 Until at least twenty matrix spike/matrix spike duplicates have been analyzed, recovery will be validated against default recovery limits of 70 - 130%. The maximum default precision acceptance limit is 20% relative percent difference (RPD).

18.6.3 Once twenty MS/MSD sets of a given matrix (water, soil or waste) have been analyzed, statistical acceptance limits will be calculated by the laboratory information management system and listed there.

18.6.4 If MS/MSD recovery or duplication is not within acceptable limits, take corrective action in accordance with TriMatrix SOP GR-03-124.

18.7 This procedure is written primarily in reference to SW-846 8260B, however the following modifications are allowed when used for EPA method 624 sample analyses:

18.7.1 The 12-hour shift is replaced by a 24-hour shift. BFB and continuing calibration verifications are still required but only run once in 24 hours instead of every 12.

18.7.2 A 3-point calibration can be used in place of the 7-point.

18.7.3 SPCCs and CCCs are not used. Instead response factors for every compound listed on the EPA 624 list must have ≤35% RSD for the calibration to be valid.

18.7.4 The continuing calibration verification does not use SPCCs and CCCs. The RF for every compound in the 40 ug/L continuing calibration verification is compared with the corresponding calibration acceptance criteria found in Attachment 23.8 (Table 5). If parameter responses fall within the designated ranges, analysis of samples can begin. If any individual RF falls outside the range, a new continuing calibration verification or a new calibration must be run.

18.7.5 There are no criteria for internal standard areas or retention times in method 624. However, method 8260B criteria will be followed.

18.7.6 Every targeted analyte must be spiked in the MS/MSD, SCV and LFB/BS. Not just the limited list in Section 18.5.2.

18.7.7 Acrolein and Acrylonitrile may only be screened by GC/MS. All positive results must be qualified in the report as estimated.

18.7.8 Refer to Section 8.1.1.5 for sample collection requirements unique to method 624.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis to demonstrate acceptable accuracy and precision.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Spike four aliquots of organic-free reagent water so analyte concentrations are in the lower half of the calibration range. Process the four spikes following every step outlined in the procedure, including quality control.

19.1.1.2 Input all four results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery must be within the LFB/BS acceptability limits listed in the laboratory information management system. Relative standard deviation must be ≤20%.

19.1.1.3 If any criterion in the study fails, locate and correct the source of the problem and repeat the study successfully.

19.1.1.4 Repeated failure, will confirm a general problem with the procedure and/or techniques used. If this occurs, locate the problem and correct the procedure and/or techniques used then repeat the study successfully.

19.1.1.5 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.6 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.1.2 A Continuing Demonstration of Capability (CDC) must be performed annually by all analysts running samples by any of the following approaches:

19.1.2.1 By using the last four of seven results obtained from a method detection limit study if run exclusively by the analyst. ONLY the last four results may be used.

19.1.2.2 BY repeating the IDC study.

19.1.2.3 By using four consecutively run blank spike results obtained during the course of routine analysis and if run exclusively by the analyst.

19.1.2.4 By running an acceptable blind performance testing sample during the course of routine sample analysis.

19.1.2.5 Copies of successful demonstration of capability studies and raw data must be submitted to the Quality Assurance department.

19.2 A Method Detection Limit (MDL) Study must be performed annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update III, Method 8260B, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 2, December 1996

22.2 *40 Code of Federal Regulations, most current edition*, Part 136, Appendix A, Method 624-Purgeables

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 8260A, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Revision 1, September, 1994

22.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition*, Final Update II, Method 5030B, "Purge and Trap for Aqueous Samples", Revision 2, December, 1996

23.0 ATTACHMENTS

23.1 Attachment 23.1, Table 1, Compound List, CAS #, and Routine Reporting Limits, for Standard I

23.2 Attachment 23.2, Table 1A, Compound List, CAS #, and Routine Reporting Limits, For Standard II

23.3 Attachment 23.3, Table 2, BFB Key Ion Abundance Criteria

23.4 Attachment 23.4, Table 3, Standard 8260B Volumes Required For Curve

23.5 Attachment 23.5, Table 3A, Appendix IX Volumes Required For Curve

23.6 Attachment 23.6, Table 4, Elution Order, Quantitation and Characteristic Ions, Internal Standards, and Surrogates for Standard I

23.7 Attachment 23.7, Table 4A, Elution Order, Quantitation and Characteristic Ions, for Standard II

23.8 Attachment 23.8, Table 5, Continuing Calibration QC Acceptance Criteria, Method 624

23.9 Attachment 23.9, Example Chromatogram, DB-624

23.10 Attachment 23.10, Example Chromatogram, DB-VRX

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
D	Acetone	67-64-1	100	5	0.1	0.75
C	Acrolein	107-02-8	100	5	0.01	0.05
C	Acrylonitrile	107-13-1	100	1	0.01	0.05
A	Benzene	71-43-2	100	1	0.01	0.05
A	Bromobenzene	108-86-1	100	1	0.01	0.05
A	Bromochloromethane	74-97-5	100	1	0.01	0.05
A	Bromodichloromethane	75-27-4	100	1	0.01	0.05
A	Bromoform	75-25-2	100	1	0.01	0.05
B	Bromomethane	74-83-9	100	1	0.01	0.05
A	n-Butylbenzene	104-51-8	100	1	0.01	0.05
A	sec-Butylbenzene	135-98-8	100	1	0.01	0.05
A	tert-Butylbenzene	98-06-6	100	1	0.01	0.05
D	Carbon disulfide	75-15-0	100	1	0.1	0.5
A	Carbon tetrachloride	56-23-5	100	1	0.01	0.05
A	Chlorobenzene	108-90-7	100	1	0.01	0.05
B	Chloroethane	75-00-3	100	1	0.01	0.05
D	2-Chloroethyl vinyl ether	110-75-8	100	10	0.1	0.5
A	Chloroform	67-66-3	100	1	0.01	0.05
B	Chloromethane	74-87-3	100	1	0.01	0.05
A	2-Chlorotoluene	95-49-8	100	1	0.01	0.05
A	4-Chlorotoluene	106-43-4	100	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Dibromochloromethane	124-48-1	100	1	0.01	0.05
A	1,2-Dibromo-3-chloropropane	96-12-8	100	1	0.01	0.05
A	1,2-Dibromoethane	106-93-4	100	1	0.01	0.05
A	Dibromomethane	74-95-3	100	1	0.01	0.05
A	1,2-Dichlorobenzene	95-50-1	100	1	0.01	0.05
A	1,3-Dichlorobenzene	541-73-1	100	1	0.01	0.05
A	1,4-Dichlorobenzene	106-46-7	100	1	0.01	0.05
E	trans-1,4-Dichloro-2-butene	110-57-6	100	1	0.05	0.25
B	Dichlorodifluoromethane	75-71-8	100	1	0.01	0.05
A	1,1-Dichloroethane	75-34-3	100	1	0.01	0.05
A	1,2-Dichloroethane	107-06-2	100	1	0.01	0.05
A	1,1-Dichloroethylene	75-35-4	100	1	0.01	0.05
A	cis-1,2-Dichloroethylene	156-59-2	100	1	0.01	0.05
A	trans-1,2-Dichloroethylene	156-60-5	100	1	0.01	0.05
F	Dichlorofluoromethane	73-43-4	100	1	0.01	0.05
A	1,2-Dichloropropane	78-87-5	100	1	0.01	0.05
A	1,3-Dichloropropane	142-28-9	100	1	0.01	0.05
A	2,2-Dichloropropane	594-20-7	100	1	0.01	0.05
A	1,1-Dichloropropylene	563-58-6	100	1	0.01	0.05
A	cis-1,3-Dichloropropylene	10061-01-5	100	1	0.01	0.05
A	trans-1,3-Dichloropropylene	10061-02-6	100	1	0.01	0.05
A	Ethylbenzene	100-41-4	100	1	0.01	0.05
E	Ethyl ether	60-29-7	100	5	0.1	0.5
E	Heptane	142-82-5	100	10	0.1	0.5

Attachment 23.1
Table 1
Compound List, CAS Number and Routine Reporting Limits for Standard I (continued)

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting Limit		
				Aqueous ug/L	Low Level Soil, mg/kg	High Level Soil, mg/kg
A	Hexachlorobutadiene	87-68-3	100	1	0.01	0.05
E	Hexachloroethane	67-72-1	100	1	0.01	0.05
D	2-Hexanone	591-78-6	100	5	0.1	2.5
D	Iodomethane	74-88-4	100	1	0.01	0.05
E	Isopropanol	67-63-0	100	25	0.1	2.5
A	Isopropylbenzene	88-82-8	100	1	0.01	0.05
A	p-Isopropyltoluene	99-87-6	100	1	0.01	0.05
E	Methyl tert-butyl ether (MTBE)	1634-04-4	100	1	0.1	2.5
E	Methyl Acetate	79-20-9	100	10	0.01	0.25
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
A	Methylene chloride	75-09-2	100	1	0.01	0.25
D	Methyl ethyl ketone (2-Butanone)	78-93-3	100	5	0.1	0.75
E	2-Methylnaphthalene	91-57-6	100	5	0.05	0.25
D	4-Methyl-2-pentanone (MIBK)	108-10-1	100	5	0.1	2.5
A	Naphthalene	91-20-3	100	5	0.01	0.5
A	n-Propylbenzene	103-65-1	100	1	0.01	0.05
A	Styrene	100-42-5	100	1	0.01	0.05
A	Tetrachloroethylene	127-18-4	100	1	0.01	0.05
A	1,1,1,2-Tetrachloroethane	630-20-6	100	1	0.01	0.05
A	1,1,2,2-Tetrachloroethane	79-34-5	100	1	0.01	0.05
A	Toluene	108-88-3	100	1	0.01	0.05
A	1,2,3-Trichlorobenzene	87-61-6	100	1	0.01	0.05
A	1,2,4-Trichlorobenzene	120-82-1	100	1	0.01	0.05
A	1,1,1-Trichloroethane	71-55-6	100	1	0.01	0.05
A	1,1,2-Trichloroethane	79-00-5	100	1	0.01	0.05
A	Trichloroethylene	79-01-6	100	1	0.01	0.05
B	Trichlorofluoromethane	75-69-4	100	1	0.01	0.05
A	1,2,3-Trichloropropane	96-18-4	100	1	0.01	0.05
E	1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	100	1	0.01	0.05
A	1,2,4-Trimethylbenzene	95-63-6	100	1	0.01	0.05
A	1,3,5-Trimethylbenzene	108-67-8	100	1	0.01	0.05
D	Vinyl acetate	108-05-4	100	5	0.01	0.05
B	Vinyl chloride	75-01-4	100	1	0.01	0.05
A	m,p-Xylene	106-42-3	200	1	0.02	0.1
A	o-Xylene	95-47-6	100	1	0.01	0.05
A	Xylene, total	1330-20-7	300	1	0.03	0.15

*Mix A: Volatile Organic Compounds - Liquids @ 2.0 mg/mL in MeOH (Accustandard M502A-R-10X)

*Mix B: Volatile Organic Compounds - Gases @ 2.0 mg/mL in MeOH (Accustandard M502B-10X)

*Mix C: Acrolein & Acrylonitrile at 2.0 mg/mL in MeOH (Accustandard S-948)

*Mix D: Ketone Mix @ 2.0 mg/mL in MeOH (Accustandard M-8260-ADD-10X)

*Mix E: Custom Additions to Method 8260B @ 2.0 mg/mL in MeOH (Accustandard S-3439-R1)

*Mix F: Dichlorofluoromethane @ 1.0 mg/mL in MeOH (Absolute #70904)

Attachment 23.2
Table 1A
Compound List, CAS#, And Routine Reporting Limits For Standard II

Mix*	Analyte	CAS Number	Concentration mg/L	Minimum Reporting		
				Aqueous ug/L	Low Level Soil mg/kg	High Level Soil mg/kg
A	Allyl chloride	107-05-1	250	5	0.05	0.25
A	2-Butanol	15892-23-6	250	50	0.5	2.5
A	n-Butanol	71-36-3	250	50	0.5	2.5
A	t-Butanol	75-65-0	250	50	0.5	2.5
Neat	n-Butyl acetate	123-86-4	250	10	0.02	0.5
B	2-Chloro-1,3-butadiene (Chloroprene)	126-99-8	250	5	0.05	0.25
Neat	1-Chlorohexane	544-10-5	250	1	0.01	0.05
E	Cyclohexane	110-82-7	100	10	0.01	0.25
A	Cyclohexanone	108-94-1	250	50	0.5	2.5
A	2,3-Dichloro-1-propene	78-88-6	250	5	0.05	0.25
A	1,4-Dioxane	123-91-1	250	25	0.1	2.5
C	Epichlorohydrin	106-89-8	250	25	0.05	1.3
A	Ethanol	64-17-5	250	50	0.5	2.5
A	Ethyl acetate	141-78-6	250	10	0.5	2.5
Neat	Ethyl methacrylate	97-63-2	250	5	0.05	0.25
A	Hexachloroethane	67-72-1	250	5	0.05	0.25
A	Hexane	110-54-3	250	10	0.1	0.5
A	Isobutanol	78-83-1	250	50	0.5	2.5
A	Isobutyl acetate	110-19-0	250	5	0.1	0.5
Neat	Isopropanol	67-63-0	250	25	0.05	2.5
A	Isopropyl ether	108-20-3	250	5	0.01	0.05
A	Methacrylonitrile	126-98-7	250	5	0.5	2.5
E	Methylcyclohexane	108-87-2	100	10	0.01	0.25
Neat	Methyl methacrylate	80-62-6	250	5	0.05	0.25
A	2-Nitropropane	79-46-9	250	5	0.1	0.5
A	n-Propanol	71-23-8	250	50	0.5	2.5
A	Propionitrile	107-12-0	250	5	0.5	2.5
A	Tetrahydrofuran	109-99-9	250	5	0.1	0.5

*Mix A: Appendix IX Standard @ 2.5 mg/mL in MeOH (Accustandard S-3651 Volatile Custom Solution)
*Mix B: 2-Chloro-1,3-butadiene (Chloroprene) @ 2.0 mg/mL in MeOH (Accustandard App-9-048-R1-20X)
*Mix B: Epichlorohydrin @ 1.0 mg/mL in MeOH (Accustandard 70377)

Attachment 23.3
Table 2
BFB Key Ion Abundance Criteria

Table 2
BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Attachment 23.4
Table 3
Standard 8260B Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.1.1	Additional Volume (µL) of Methanol Required
1.0	0.5	999.5
5.0	2.5	997.5
10	5.0	995
20	10	990
40	20	980
100	50	950
200	100	900

Attachment 23.5
Table 3A
Appendix IX Volumes Required For Curve

Concentration of Working Standard (ug/L)	Volume (µL) of Standard 12.4.2.1	Additional Volume (µL) of Methanol Required
5.0	1.0	999
25	5.0	995
50	10	990
100	20	980
250	50	950
500	100	900
1000	20*	980

*Note: when preparing the 1000 ug/L Appendix IX calibration standard, use the stock standard listed in 12.3.4.2.

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Dichlodifluoromethane	85	87, 50	1
2	Chloromethane	50	52, 49	1
3	Vinyl Chloride	62	64, 47	1
4	Bromomethane	94	96, 93	1
5	Chloroethane	64	66	1
6	Trichlorofluoromethane	101	103, 105	1
7	Dichlorofluoromethane	67	69, 47	1
8	Ethyl Ether	45	59, 73	1
9	Acrolein	55	56, 53	1
10	1,1-Dichloroethylene	96	61, 98	1
11	1,1,2-Trichloro-1,2,2-trifluoroethane	101	103, 151	1
12	Iodomethane	142	127, 141	1
13	Carbon Disulfide	76	78, 77	1
14	Acetone	43	58, 42	1
17	Methylene Chloride	49	51, 84	1
18	Acrylonitrile	52	54, 53	1
19	Trans-1,2-Dichloroethylene	96	61, 98	1
20	Methyl tert-Butyl Ether (MTBE)	73	43, 57	1
21	1,1-Dichloroethane	63	65,83	1
22	Vinyl Acetate	43	42, 44	1
23	2,2-Dichloropropane	77	41, 79	1
24	cis-1,2-Dichloroethylene	96	61, 63	1
25	Methyl Ethyl Ketone (2-Butanone)	43	72, 57	1
26	Bromochloromethane	49	130, 128	1
27	Tetrahydrofuran	71	41, 42	1
28	Chloroform	83	85, 47	1
29	1,1,1-Trichloroethane	97	99, 61	1
30	SUR:Dibromofluoromethane	113	111, 192	1
31	IS:Fluorobenzene	96	70,50	1
32	Carbon Tetrachloride	117	119, 121	1
33	Cyclohexane	41	39, 57	1
34	1,1-Dichloropropylene	75	110, 77	1
35	Benzene	78	51, 50	1
36	1,2-Dichloroethane	62	64, 49	1
37	1,2-Dichloroethane-d4	65	67,51	1
38	Trichloroethylene	130	95, 132	1
39	1,2-Dichloropropane	63	62, 76	1
40	Dibromomethane	93	95, 174	1
41	Bromodichloromethane	83	85, 129	1

Attachment 23.6
Table 4
Elution Order, Quantitation And Characteristic Ions, Internal Standards, And Surrogates For Standard I (Cont.)

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
44	cis-1,3-Dichloropropylene	75	77, 110	1
45	4-Methyl-2-Pentanone (MIBK)	43	58, 85	1
46	SUR:d8-Toluene	98	100,70	1
47	Toluene	91	92, 65	1
48	Trans-1,3-Dichloropropylene	75	77, 110	1
49	1,1,2-Trichloroethane	97	83, 85	1
50	Tetrachloroethylene	166	129, 168	2
51	1,3-Dichloropropane	76	78, 41	2
52	2-Hexanone	43	58, 85	2
53	Dibromochloromethane	129	127, 131	2
54	1,2-Dibromoethane	109	107, 188	2
55	IS:d5-Chlorobenzene	82	117, 119	2
56	Chlorobenzene	112	77, 114	2
57	1,1,1,2-Tetrachloroethane	131	133, 119	2
58	Ethylbenzene	91	106, 51	2
59	m,p-Xylene	91	106, 51	2
60	o-Xylene	91	106, 51	2
61	Styrene	104	78, 51	2
62	Bromoform	173	175, 79	2
63	Isopropylbenzene	105	120, 79	3
64	SUR:4-Bromofluorobenzene	95	174, 176	2
65	Bromobenzene	77	156, 158	3
66	1,1,2,2-Tetrachloroethane	83	85, 95	3
67	1,2,3-Trichloropropane	75	49, 110	3
68	n-Propylbenzene	91	120, 65	3
69	2-Chlorotoluene	126	91, 63	3
70	1,3,5-Trimethylbenzene	105	120, 77	3
71	4-Chlorotoluene	91	126, 63	3
72	tert-Butylbenzene	119	91, 134	3
73	sec-Butylbenzene	105	134, 91	3
74	1,3-Dichlorobenzene	146	111, 148	3
75	p-Isopropyltoluene	119	134, 91	3
76	IS:d4-1,4-Dichlorobenzene	152	150	3
77	1,4-Dichlorobenzene	146	111, 148	3
78	1,2-Dichlorobenzene	146	111, 148	3
79	n-Butylbenzene	91	92, 134	3
80	1,2-Dibromo-3-Chloropropane	75	155, 157	3
81	1,2,4-Trichlorobenzene	180	182, 145	3
82	Hexachlorobutadiene	225	227, 260	3
83	Naphthalene	128	102, 51	3
84	1,2,3-Trichlorobenzene	180	182, 145	3

Attachment 23.7
Table 4A
Elution Order, Quantitation And Characteristic Ions, For Standard II

Elution Order	Analyte	Quantitation Ion	Secondary Ions	Internal Standard
1	Ethanol	45	43	1
2	Acetonitrile	41	40, 54	1
3	Allyl Chloride	76	39, 41	1
4	Isopropanol	45	59, 43	1
5	t-Butanol	59	41, 43	1
6	Hexane	41	56, 57	1
7	Isopropylether	45	69, 86	1
8	n-Propanol	42	59, 41	1
9	Methylcyclopentane	56	41, 69	1
10	Propionitrile	54	52, 56	1
11	Ethyl Acetate	43	61, 45	1
12	Methacrylonitrile	67	41, 52	1
13	2-Butanol	45	59, 57	1
14	Sur: Dibromofluoromethane	113	111, 79	1
15	Cyclohexane	41	39, 57	1
16	IS: Fluorobenzene	96	70,50	1
17	Isobutanol	41	43, 42	1
18	n-Butanol	41	39, 56	1
19	1,2-Dichloroethane-d4	65	67,51	1
20	2,3-Dichloro-1-propylene	75	77, 110	1
21	Methyl Methacrylate	41	69, 100	1
22	1,4-Dioxane	88	57, 43	1
23	2-Nitropropane	41	43, 46	2
24	Epichlorohydrin	49	57, 62	2
25	Hexachloroethane	117	119, 201	3
26	Ethyl methacrylate	41	99, 69	2
27	Sur: d-8-Toluene	98	100, 70	2
28	n-Butyl acetate	43	56, 57	2
29	IS: d5-Chlorobenzene	82	117, 119	2
30	Cyclohexanone	55	42, 98	2
31	Sur: 4-Bromofluorobenzene	95	174, 176	2
32	trans-1,4-Dichloro-2-butylene	53	75, 89	3
33	IS: d4-1,4-Dichlorobenzene	152	150	3

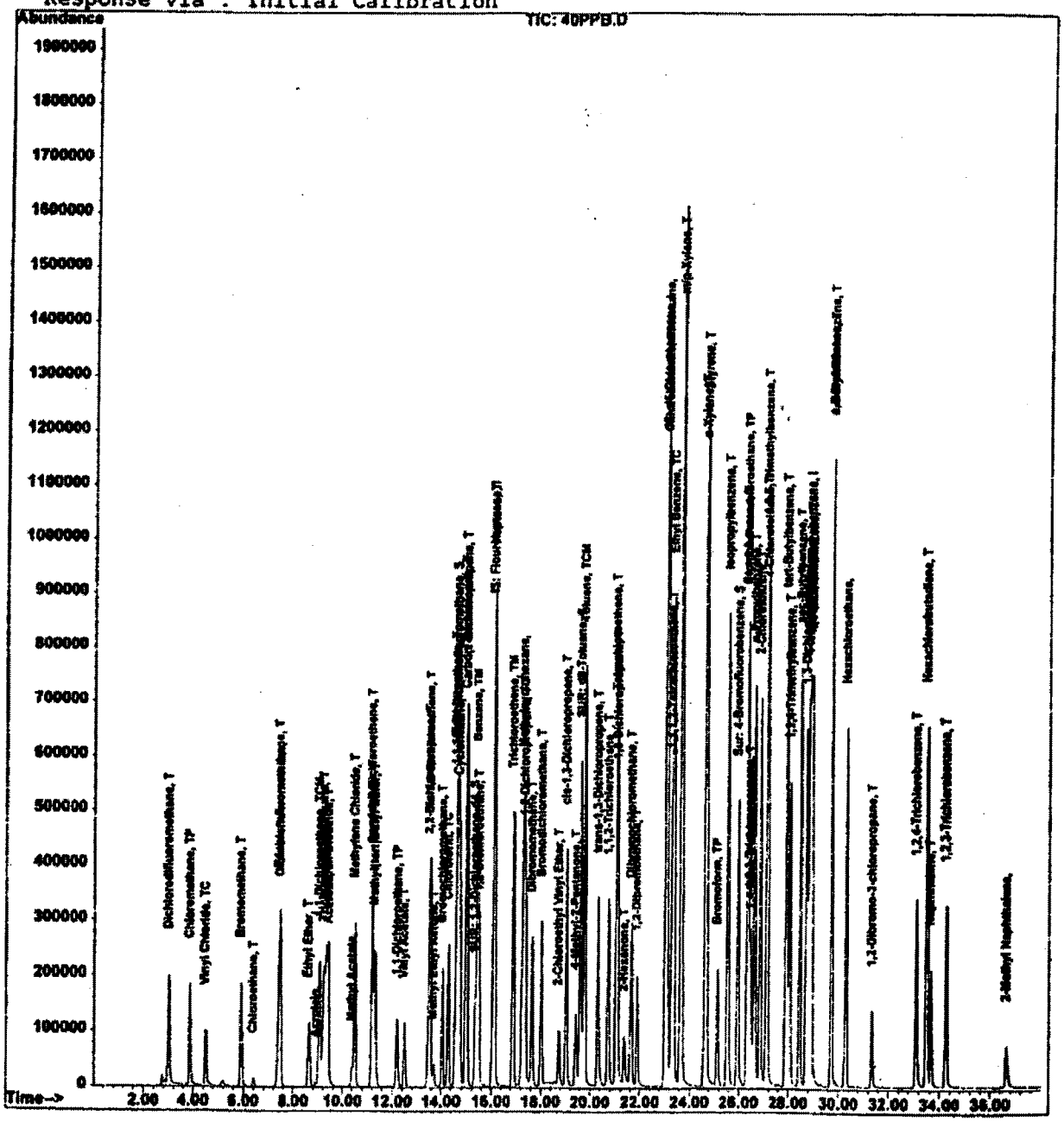
Attachment 23.8
Table 5
Continuing Calibration QC Acceptance Criteria
Method 624

Parameter	**Range for Q (ug/L)**
Benzene	25.6-54.4
Bromodichloromethane	26.2-53.8
Bromoform	28.4-51.6
Bromomethane	5.6-74.4
Carbon tetrachloride	29.2-50.8
Chlorobenzene	26.4-53.6
Chloroethane	15.2-64.8
2-Chloroethylvinyl ether	D-89.6
Chloroform	27.0-53.0
Chloromethane	D-81.6
Dibromochloromethane	27.0-53.0
1,2-Dichlorobenzene	25.2-54.8
1,3-Dichlorobenzene	29.2-50.8
1,4-Dichlorobenzene	25.2-54.8
1,1-Dichloroethane	29.0-51.0
1,2-Dichloroethane	27.2-52.8
1,1-Dichloroethylene	20.2-59.8
trans-1,2-Dichloroethylene	27.8-52.2
1,2-Dichloropropane	13.6-66.4
cis-1,3-Dichloropropylene	9.6-70.4
trans-1,3-Dichloropropylene	20.0-60.0
Ethyl benzene	23.6-56.4
Methylene chloride	24.2-55.8
1,1,2,2-Tetrachloroethane	24.2-55.8
Tetrachloroethylene	29.4-50.6
Toluene	29.8-50.2
1,1,1-Trichloroethane	30.0-50.0
1,1,2-Trichloroethane	28.4-51.6
Trichloroethylene	26.6-53.4
Trichlorofluoromethane	19.2-60.8
Vinyl chloride	1.6-78.4-

D=Detected result must be greater than zero.

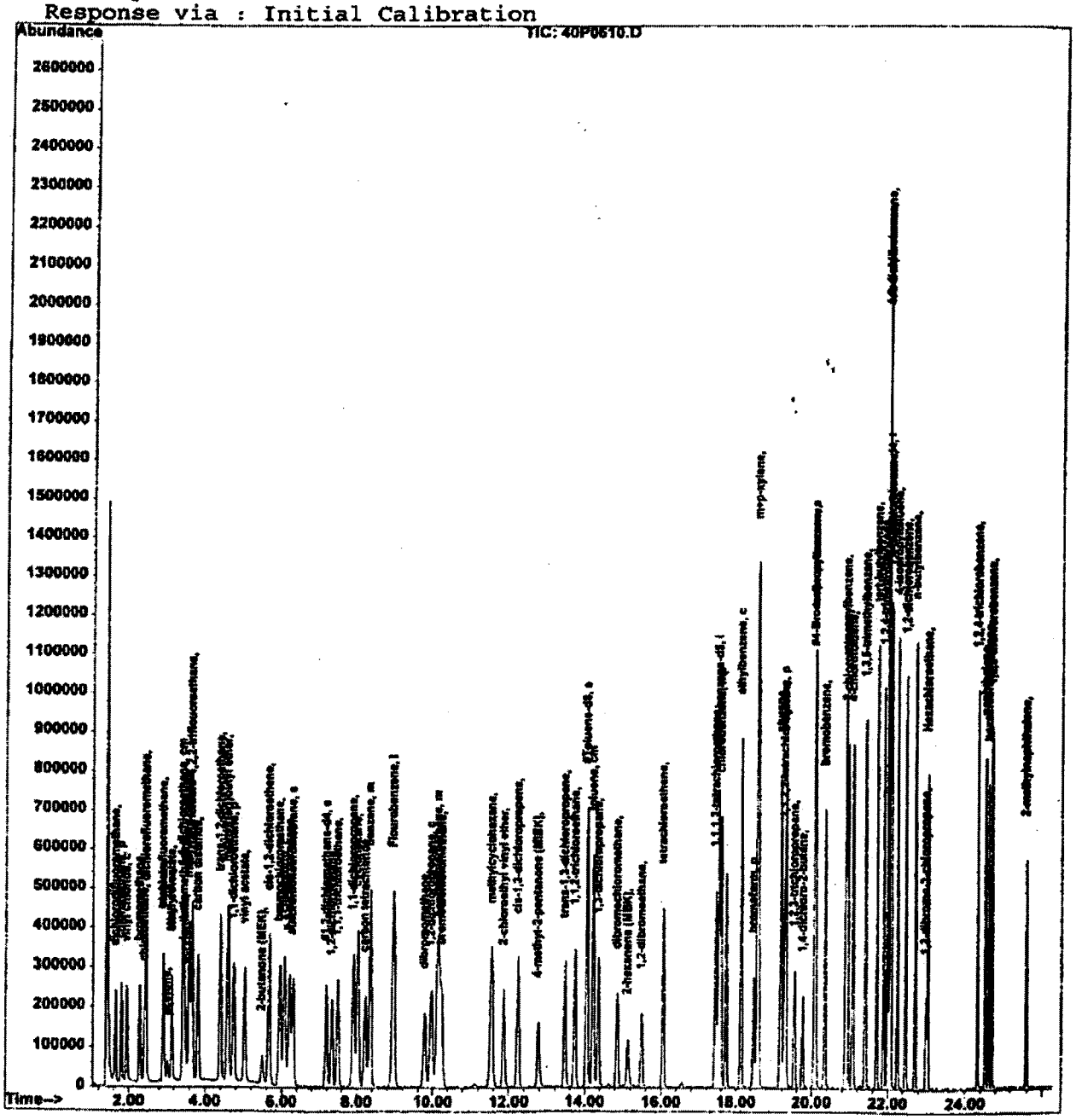
Attachment 23.9
Example Chromatogram, DB-624

```
Method        : C:\Sat132\QUANT\8260b49.m (RTE Integrator)
Title         : Saturn 132
Last Update   : Thu May 09 11:09:55 2002
Response via  : Initial Calibration
```



Attachment 23.10
Example Chromatogram, DB-VRX

```
Method        : C:\HPCHEM\2\METHODS\8260WT23.M (RTE Integrator)
Title         : EPA METHOD 624/8240
Last Update   : Thu Jun 06 15:21:31 2002
Response via  : Initial Calibration
```



TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Total Cyanide by Konelab UV Spectrophotometry

Standard Methods 4500-CN G
Standard Methods 4500-CN E
SW-846 Method 9014

APPROVALS:

Area Supervisor: Heather L. Brady Date: 9-15-09
Heather L. Brady

QA Officer: Date: 9-10-09
Tom C. Boocher

Operations Manager: Date: 9/14/09
Jeff P. Glaser

Procedure Number: GR-05-122
Revision Number: 0.4

Date Initiated: 10/28/03
Effective Date: 9/25/09

Date Revised: 9/10/09
Pages Revised: All

By: Jodi L. Blouw

Total Number of Pages: 18

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the determination of cyanide in drinking and surface waters, domestic and industrial wastes, and soil.

1.2 The applicable range for water samples is 0.005 mg/L to 0.20 mg/L.

1.3 The reporting limit for water samples is 0.005 mg/L. The reporting limit for soils is 0.2 mg/kg.

2.0 PRINCIPLE PROCEDURE REFERENCES

2.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-CN⁻ E, "Colorimetric Method"

2.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-CN⁻ G, "Cyanides Amenable to Chlorination after Distillation"

2.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 0, December, 1996, Method 9014, "Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide"

3.0 SUMMARY OF PROCEDURE

3.1 Cyanide in alkaline distillate is converted to cyanogen chloride (CNCl) by reaction with chloramine-T below pH 8.0, without hydrolyzing to CNO^{1-}.

3.2 CNCl then forms a red-blue color by reacting with pyridine-barbituric acid. The developed color is read at 575 nm on the Konelab spectrophotometer.

Note: The methods are modified by replacing 0.04N sodium hydroxide (NaOH) diluent with 0.25N NaOH to maintain the same NaOH concentration between manual and automated distillation.

4.0 PARAMETER OR COMPOUND LIST

4.1 Total Cyanide

4.2 Amenable Cyanide

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-18-106, *Cyanide Total, Preliminary Macro-Distillation from Soil, Sludge and Waste,* latest revision

5.2 TriMatrix SOP GR-18-109, *Cyanide, Preliminary Midi-Distillation*, latest revision

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5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

5.6 TriMatrix SOP GR-05-129, *Konelab 20 Operation*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Distillation removes most interferences. Refer to TriMatrix SOP GR-18-106 and GR-18-109 for distillation procedures.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

7.4 The total toxicity and/or carcinogenicity of reagents used in this procedure have not been precisely defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce exposure to the lowest possible level by adherence to established safety policies.

7.4.3 Material Safety Data Sheets are maintained on the laboratory intranet of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean waste-contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the attention of the Area Supervisor and/or Health and Safety Officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water and wastewater samples are collected in opaque brown plastic bottles. Samples are preserved with sodium hydroxide to greater than pH 12.

8.2 Wastes, sludge and soils are collected in glass jars without preservative.

8.3 Samples must be stored at 4 ±2° C until analysis.

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8.4 Homogenize water samples by shaking immediately before removing a sample aliquot. Wastes, sludge and soils must be homogenized using an appropriate mixing procedure, before distillation.

8.5 Distillation and analysis must be performed within 14 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Konelab Aqua 20 automated UV spectrophotometer, Type 968, at 575 nm. Refer to the Equipment List located on the laboratory intranet library for a full description of minimum and current instrument specifications.

9.2 Volumetric flasks, various sizes

9.3 Pipets:

9.3.1 Volumetric, glass, type A

9.3.2 Micropipette, calibrated and serialized

9.3.3 Macropipette, calibrated and serialized

9.4 Personal computer. Refer to the Information Technology (IT) department's Computer Inventory Database for minimum and current computer and software specifications associated with the analytical instrument.

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Refer to TriMatrix SOP GR-05-129 for a schedule of preventive instrument maintenance.

11.0 CHEMICALS AND REAGENTS

11.1 Indicator solution: Dissolve 0.020 g p-dimethylaminobenzal-rhodanine in 100 mL acetone. Expiration for this solution is six months from the date made.

11.2 Indicator solution: Potassium chromate (K_2CrO_4), 5% (w/v), Purchased commercially.

11.3 Sodium Chloride Standard: Dissolve 1.125 g NaCl (dried at 140° C) in reagent water. Dilute to 1000 mL with reagent water. Expiration for this solution is six months from the date made.

11.4 Silver nitrate titrant: Dissolve 3.27 g $AgNO_3$ in 1 L reagent water. Standardize against the NaCl standard, using the argentometric titration, with K_2CrO_4 indicator. Expiration for this solution is six months from the date made.

$$Cl^- + AgNO_3 \xrightarrow{K_2CrO_4} AgCl(s) + NO_3$$

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$$2Ag^{+} + CrO_4^{2-} \longrightarrow Ag_2CrO_4$$

11.4.1 $AgNO_3$ Standardization:

11.4.1.1 Place 10.0 mL of the NaCl standard and about 100 mL of reagent water, into a beaker.

11.4.1.2 Add 1 mL K2CrO4 indicator solution.

11.4.1.3 Titrate with $AgNO_3$ solution, to a pinkish yellow endpoint.

11.4.2 The $AgNO_3$ correction factor is calculated as follows:

$$\frac{A}{B} = C$$

Where:
A = NaCl standard volume, in mL
B = $AgNO_3$ titrant used, in mL
C = Correction factor

11.5 Laboratory Reagent Water: ASTM Type II, Milli-Q system

11.6 Chloramine-T solution: Dissolve 1.0 g chloramine T in 100 mL reagent water. Prepare weekly and store at 4 ±2° C in the refrigerator.

11.7 Sodium dihydrogen phosphate buffer, 1M: Dissolve 34.5 g $NaH_2PO_4 \cdot H_2O$ in 250 mL of laboratory reagent water. Expiration for this solution is six months from the date made. Store at 4 ±2° C in the refrigerator.

11.8 Pyridine-barbituric acid reagent:

11.8.1 Weigh 15.0 g barbituric acid in a 250 mL volumetric flask. Add just enough water to wash down the flask neck and wet the barbituric acid.

11.8.2 Add 75 mL pyridine. Cap and mix.

11.8.3 Add 15 mL concentrated hydrochloric acid (HCl). Mix and cool to room temperature. Dilute to the mark with reagent water, and mix again.

11.8.4 This reagent is stable for up to six months. Discard if a precipitate forms. Prepare in a hood. Store at 4 ±2° C in the refrigerator.

11.9 Sodium Hydroxide Diluent (0.25 N):

11.9.1 In a 2 L volumetric flask, dissolve 20.0 g sodium hydroxide in 1000 mL reagent water.

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11.9.2 Dilute to 2000 mL. Invert several times to mix.

11.9.3 The solution may be stored in an inert plastic bottle at room temperature, for up to six months.

11.10 All reagents must be recorded in the reagents log with a unique ID number. Refer to Attachment 23.1 for a reagents log example.

12.0 STANDARDS PREPARATION

12.1 Primary Cyanide Solution (1000 mg/L CN^-):

12.1.1 Cyanide solutions are prepared from ACS stock.

12.1.2 Dissolve 1.255 g potassium cyanide and 1.0 g potassium hydroxide in 400 mL of reagent water, in a 500 mL volumetric flask. Dilute to volume with reagent water. The concentration is 1000 mg/L cyanide. Expiration for this solution is six months from the date made.

12.1.3 This standard must be re-standardized by titration, weekly.

CAUTION: KCN is highly toxic. Avoid inhalation of cyanide dust, or contact with the solid or solutions.

12.2 Standardize the 1000 mg/L CN^- solution as follows:

12.2.1 Dilute 10 mL of the 1000 mg/L KCN to 100 mL, using 0.25N NaOH diluent.

12.2.2 Add 0.5 mL of rhodanine indicator.

12.2.3 Titrate with standardized $AgNO_3$ solution to the first color change from a canary yellow to a salmon hue.

12.2.4 Titrate a 0.25N NaOH blank.

12.2.5 Cyanide concentration in the standard is calculated as follows:

$$\frac{\text{mg CN -}}{\text{L}} = (A-B) \times 100 \times C$$

Where:
A = $AgNO_3$ titrant used for the standard, in mL
B = $AgNO_3$ titrant used for the blank, in mL
C = $AgNO_3$ titrant correction factor (from section 11.4.2)

12.3 Intermediate Cyanide Solution (100 mg/L CN^-):

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12.3.1 Prepare a 100 mg/L CN^- standard by measuring the appropriate calculated volume of 1000 mg/L CN^- solution into a 100 mL volumetric flask.

12.3.2 Dilute to volume with 0.25N NaOH.

12.3.3 Expiration for this solution is one week from the date made.

12.4 Tertiary Cyanide Solution (1.0 mg/L CN^-):

12.4.1 Prepare a 10 mg/L CN^- solution by measuring 10 mL of 100 mg/L CN^- into approximately 60 mL of 0.25N NaOH in a 100 mL volumetric flask.

12.4.2 Dilute to volume with 0.25N NaOH. The concentration is 10 mg/L. Prepare fresh daily.

12.4.3 Then, prepare a 1.0 mg/L CN^- standard by measuring 10 mL of 10 mg/L CN^- into approximately 60 mL of 0.25 N NaOH in a 100 mL volumetric flask.

12.4.4 Dilute to volume with 0.25N NaOH. The concentration is 1.0 mg/L CN^-. Prepare fresh daily.

12.4.5 Use to prepare the calibration.

12.5 Calibration Standards are prepared as follows (prepare fresh daily):

Concentration mg/L	**Dilution**
0.2	20 mL 1.0 mg/L CN^- to 100 mL, with 0.25 N NaOH
0.1	10 mL 1.0 mg/L CN^- to 100 mL, with 0.25 N NaOH
0.05	5.0 mL 1.0 mg/L CN^- to 100 mL, with 0.25 N NaOH
0.02	10 mL 0.2 mg/L CN^- to 100 mL, with 0.25 N NaOH
0.01	10 mL 0.1 mg/L CN^- to 100 mL, with 0.25 N NaOH
0.005	10 mL 0.05 mg/L CN^- to 100 mL, with 0.25 N NaOH

12.6 Prepare a second-source 1000 mg/L cyanide standard with KCN reagent purchased from an alternate vendor according to Section 12.1.

12.6.1 Standardize as in Section 12.2.

12.6.2 Prepare an intermediate dilution then 0.05 mg/L and 0.1 mg/L working standards (Sections 12.3 through 12.5).

12.6.3 Use these working standards as the second-source calibration verification (SCV).

12.6.4 Refer to Section 18.2.2 for SCV acceptance limits.

12.7 Record all standard preparations in the appropriate standards log in Element™ with a unique ID number. Refer to Attachment 23.2 for a standards log example.

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12.8 Primary and secondary cyanide standards must be labeled with the following information:

12.8.1 Initials of the preparer

12.8.2 Preparation date

12.8.3 Concentration, with units

12.8.4 Parameter name

12.8.5 Standards logbook number as it appears in the standards logbook

12.8.6 Diluted volume

12.9 Working standards must be labeled with the following information:

12.9.1 Initials of the preparer

12.9.2 Preparation date

12.9.3 Concentration, with units

12.9.4 Parameter name

12.9.5 Expiration date

12.9.6 Standards logbook number as it appears in the standards logbook

13.0 SAMPLE PREPARATION

13.1 All samples must be distilled before analysis.

13.2 Refer to TriMatrix SOP GR-18-106 and TriMatrix SOP GR-18-109 for distillation instructions.

14.0 CALIBRATION PROCEDURES

14.1 Analyze working calibration standards (Section 12.5) in increasing order. Analyze by Section 15.0 instructions to obtain absorbance readings.

14.2 The instrument will generate a calibration curve of absorbance vs concentration, with the best-fit linear regression line through the data. Do not force through the origin or set the instrument to use non-linear spline functions as cyanide response is a linear calibration.

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14.2.1 Linear regression curves are constructed by the instrument using concentration plotted against absorbance. The following equation is a linear regression calculation to determine sample concentrations.

$X_S = (Y_S - b)/m$

Where:
Ys = Absorbance in sample "s"
Xs = Concentration of cyanide in sample "s"
m = Slope "s" of the calibration curve
b = The y-intercept of the calibration curve

14.2.2 Refer to Attachment 23.3 for an example cyanide calibration curve.

15.0 ANALYTICAL PROCEDURE

15.1 Turn on the Konelab spectrophotometer.

15.2 Select "cyanide" as the run method

15.3 Fill sample cups with the appropriate standards and sample distillates. Place in the instrument.

15.4 For color development, the Konelab performs the following:

15.4.1 Takes 100 µL from the sample cup.

15.4.2 Adds 20 µL phosphate buffer.

15.4.3 Adds 4 µL chloramine-T solution, then incubates for 2 minutes at 37° C.

15.4.4 The instrument then takes a background reading before adding 10 µL of pyridine–barbituric acid solution.

15.5 Absorbance is measured by the spectrophotometer at 575 nm, 10 minutes after adding the pyridine - barbituric acid.

15.6 When analysis is complete, perform a Konelab shut-down and turn the instrument off.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The Konelab regresses all data against the calibration curve then prints out concentration and absorbance. The analyst need only account for dilutions. Refer to Attachment 23.3, for a Konelab calibration and run data example.

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17.0 DATA REPORTING AND DELIVERABLES

17.1 The following must be attached to laboratory benchsheets for each batch analyzed:

17.1.1 Spectrophotometer raw data hardcopy

17.1.2 Calibration curve printout

17.1.3 Batch detail report

17.2 The instrument logbook (Refer to Attachment 23.4) must be filled in for each batch analyzed with the following information:

17.2.1 Date analyzed

17.2.2 Analyst initials

17.2.3 Method name

17.2.4 Calibration standards used

17.2.5 Client name and sample numbers analyzed

17.3 The following data must be recorded in the batch detail report (Refer to Attachment 23.5) for each batch analyzed:

17.3.1 BS, MPB, SCV, BLK, CCV, CCB, SPK, MSD or DUP results. Sample concentration, with units.

17.3.2 Instrument number, date run, supervisor, stock standard number, wavelength, cell path.

17.3.3 Standard concentrations with units, observed standard concentrations, working standard ID numbers.

18.0 QUALITY ASSURANCE

18.1 Method and matrix quality control (QC) must be analyzed for each analysis batch.

18.2 Method QC consists of a Blank Spike (BS), two second-source calibration verifications (SCV), a Distillation Blank (BLK), an Instrument Blank (BLK), Continuing Calibration Blanks (CCB) and Continuing Calibration Verifications (CCV).

18.2.1 The BS is a known concentration of cyanide standard spiked into 0.25 N NaOH diluent. Concentration is 0.04 mg/L or 0.1 mg/L. Spike, then distill and analyze as a sample. Acceptance limits must be between 90 and 110% for drinking water samples and within limits listed in the laboratory information management system (Element™) for other matrices.

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18.2.2 The SCV is a known concentration of second-source cyanide standard spiked into 0.25 N NaOH and analyzed like a sample. Prepare two SCVs as described in Section 12.6. Percent recovery must be between 90 and 110% for drinking water samples and within limits listed in the laboratory information management system (Element™) for other matrices.

18.2.3 The distillation BLK is an aliquot of laboratory reagent water, distilled and analyzed as a sample. The result must be less than the reporting limit.

18.2.4 The instrument blank BLK is an aliquot of 0.25N NaOH analyzed as a sample but without distillation. The result must be less than the reporting limit.

18.2.5 The CCV is an aliquot of midrange standard analyzed immediately after the calibration curve has been confirmed, after every 10 samples and at the end of the analytical run.

18.2.5.1 CCV acceptance limits are 90 to 110% of the expected value.

18.2.5.2 Include in the ten-sample CCV count the BS, SCV, BLK, MS, MSD and DUP runs.

18.2.6 The CCB is an aliquot of 0.25N NaOH analyzed immediately after the CCV. The result must be less than the reporting limit.

18.3 Matrix QC consists of a matrix pikes (MS) and matrix spike duplicate (MSD).

18.2.1 Two sample aliquots are spiked with a known cyanide concentration then distilled and analyzed in the same way as the unspiked sample.

18.2.2 Acceptance limits for recovery are listed in Element™.

18.2.3 Calculate SPK1/SPK2 duplication as relative percent difference (RPD). The maximum RPD limit is listed on the cyanides benchsheet.

18.4 Refer to TriMatrix SOP GR-10-106 for corrective action on unacceptable quality control results.

19.0 ANALYST CERTIFICATION/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. While IDCs are not instrument dependent, one is required on each instrument to demonstrate the instrument's ability to generate acceptable accuracy and precision.

19.1.1 Initial Demonstration of Capability (IDC) Study

19.1.1.1 Prepare and analyze four aliquots of SCV following every step in the distillation and analysis.

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19.1.1.2 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation (%RSD).

19.1.1.3 Average recovery must be 90 - 110%. Relative Standard deviation must be less than or equal to 20%.

19.1.1.4 If either criterion is not met, locate and correct the source of the problem and repeat the study successfully.

19.1.1.5 Repeated failure will indicate a general problem with the procedure and/or techniques used. If this occurs, locate the problem, correct the procedure and/or techniques used then repeat the study successfully.

19.1.1.6 Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

19.1.1.7 Give a copy of the successful IDC spreadsheet and data to Quality Assurance for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC) Study

19.1.2.1 A CDC study must be performed annually by all analysts running this procedure.

19.1.2.2 The CDC may be accomplished by any one of the following approaches:

19.1.2.2.1 By repeating the IDC study

19.1.2.2.2 By using four consecutive SCV results run exclusively by the analyst obtained during the course of routine sample analysis

19.1.2.2.3 By exclusively running and successfully passing a blind cyanide PT sample during the course of routine sample analysis.

19.1.2.2.4 By using the last four of seven results obtained from an MDL study run exclusively by the analyst. ONLY the last four results may be used.

19.1.2.3 Give a copy of the successful CDC spreadsheet and data to Quality Assurance for training documentation.

19.2 A method detection limit study must be run annually in accordance with TriMatrix GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

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20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. These documents are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-CN⁻ E, "Colorimetric Method"

22.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, 4500-CN⁻ G, "Cyanides Amenable to Chlorination after Distillation"

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 0, December, 1996, Method 9014, "Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide"

23.0 ATTACHMENTS

23.1 Reagents Log Example

23.2 Standards Log Example

23.3 Spectrophotometer Calibration and Run Data Example

23.4 Instrument Logbook Example

23.5 Batch Detail Report Example

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Attachment 23.1
Reagent Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7010874

Description:	Potassium phosphate USP Dibasic	Expires:	Aug-16-11
Standard Type:	Reagent	Prepared:	Aug-16-06
Solvent:	Solvent Lot #	Prepared By:	** Vendor **
Final Volume (mls):	500	Department:	Inorganic - Wet Chemistry
Vials:	1	Last Edit:	Feb-01-07 16:07 by TCB

Fisher part#P290-500
Lot 064412
Received 1/29/07

Analyte	CAS Number	Concentration	Units
			NA

Reviewed By _____ Date _____

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Approved By: M 9-10-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.2
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7050855

Description:	Potassium Cyanide	Expires:	Feb-05-12
Standard Type:	Reagent	Prepared:	Feb-05-07
Solvent:	Solvent Lot #E04793	Prepared By	** Vendor **
Final Volume (mls):	125	Department:	Inorganic - Wet Chemistry
Vials:	1	Last Edit:	May-25-07 14:46 by TCB

JT Baker part# 3080-04
Lot: E04793
Received 5/24/07

Analyte	CAS Number	Concentration	Units
			NA

Reviewed By Date

Page 1 of 1

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Approved By: 9-10-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.3
Spectrophotometer Calibration and Run Data Example

Calibration results — Konelab 6.0.2 — Page: 1

Trimatrix
Analyst:

15.01.2004 11:11

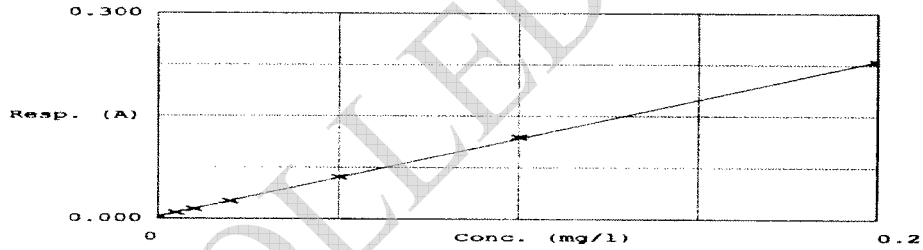
Test Cyanide

Accepted 15.01.2004 08:47

Factor 0.879
Bias 0.003

Coeff. of det. 0.999829

Errors



	Calibrator	Response	Calc. con.	Conc.	Errors
1	CN-0	0.003	-0.00052	0.00000	
2	CN-0.005	0.009	0.00473	0.00500	
3	CN-0.01	0.014	0.00957	0.01000	
4	CN-0.02	0.026	0.01975	0.02000	
5	CN-0.05	0.061	0.05066	0.05000	
6	CN-0.10	0.119	0.10184	0.10000	
7	CN-0.20	0.230	0.19897	0.20000	

Result Report — Konelab 6.0.2 — Page: 1

Trimatrix
Analyst:

Date : 2004-01-15
Time : 11.11

Test Cyanide
Unit mg/l

Sample ID:	Result	Date and Time	Man.dilut	Dilut
CNicv/ccv	0.0950	2004-01-15 09.05		
CNicb/ccb	0.0002	2004-01-15 09.05		
blk	0.0003	2004-01-15 09.05		
lcs 0.1	0.0999	2004-01-15 09.05		
lcs 0.05	0.0488	2004-01-15 09.05		
crdl	0.0050	2004-01-15 09.05		
~~mpb 114~~	0.0005	2004-01-15 09.05		
~~lfb 0.1 113~~	0.0905	2004-01-15 09.05		
~~lfb 0.04 113~~	0.0356	2004-01-15 09.05		
~~352684~~	0.0224	2004-01-15 09.05		
~~352683 mpk1~~	0.0953	2004-01-15 09.05		
~~352682 mpk2~~	0.0956	2004-01-15 09.05		
CNicv/ccv1	0.0934	2004-01-15 09.13		
CNicb/ccb1	<0.0000	2004-01-15 09.13		
~~352680~~	0.0011	2004-01-15 09.13		
mpb 114	0.0040	2004-01-15 09.13		
lfb 0.1 114	0.0985	2004-01-15 09.13		
352794	0.0043	2004-01-15 09.13		
352737	0.0165	2004-01-15 09.13		
352695 amen	0.0314	2004-01-15 09.13		
CNicv/ccv	0.0938	2004-01-15 09.21		
CNicb/ccb	<0.0000	2004-01-15 09.21		
352695 4 dup	0.0517	2004-01-15 09.21		
~~wa4 mdl1~~	0.0060	2004-01-15 09.21		
~~wa4 mdl2~~	0.0050	2004-01-15 09.21		
~~wa4 mdl3~~	0.0053	2004-01-15 09.21		
~~wa4 mdl4~~	0.0067	2004-01-15 09.21		
~~wa4 mdl5~~	0.0051	2004-01-15 09.21		
~~wa4 mdl6~~	0.0050	2004-01-15 09.21		
~~mdl wa4 7~~	0.0057	2004-01-15 09.21		
~~mpb wa4~~	0.0002	2004-01-15 09.21		
~~lfb 0.04 wa4~~	0.0991	2004-01-15 09.21		
CNicv/ccv 2	0.0949	2004-01-15 09.26		
CNicb/ccb 2	0.0007	2004-01-15 09.26		
~~Lfb 0.04 wa4~~	0.0442	2004-01-15 09.26		
CNicv/ccv	0.1049	2004-01-15 09.28		
CNicb/ccb	0.0011	2004-01-15 09.28		
~~352684~~ [redacted]	<0.19975	2004-01-15 09.33	1+99.0	1+1.0
CNicv/ccv	0.1017	2004-01-15 09.35		
CNicb/ccb	<0.0000	2004-01-15 09.35		
CNicv/ccv	0.0951	2004-01-15 09.53		
CNicb/ccb	<0.0000	2004-01-15 09.53		
0.2 mg/L	0.18856	2004-01-15 10.10		1+1.0
CNicv/ccv	0.0995	2004-01-15 10.12		
CNicb/ccb	0.0007	2004-01-15 10.12		
2 mg/L	1.82987	2004-01-15 10.16		1+19.0
CNicv/ccv	0.1037	2004-01-15 10.18		
CNicb/ccb	0.0005	2004-01-15 10.18		
mdl total mid: 6	0.0045	2004-01-15 10.27		
352682 free	<0.0000	2004-01-15 10.27		

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Approved By: D 9-10-09 QA Officer
Approved By: Heather L. Brady Area Supervisor

Attachment 23.4
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Instrument Number 298 Run Logbook

Date	Analyst	Method Name or Number	Concentration of Calibration Standards Used							Units	Client Names and Sample Numbers
			No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7		
10-2-03	JWB	Cr+6	0	0.001	0.0025	0.005	0.01	0.025		mg/L	QC .01, .02 - E tech EG .10
			Calibration Requirements		≥0.995		Calibration Results		0.999621		
10-3-03	JWB	CN⁻	0	.005	.01	.02	.05	.10	.20	mg/L	344458-461, 533, 543, 344535, 344649-650
			Calibration Requirements		≥0.995		Calibration Results		0.999673		344534, 537, 831, 833, 834, 538, 830, 835, 836 344840, 841, 834, 542, 544, 815, 837
10-6-03	JWB	CN	0	.005	.01	.02	.05	.10	.20	mg/L	344991 345003, 004
			Calibration Requirements		≥0.995		Calibration Results		0.999946		e.tech 344992 - 998, 345000 - 007
		Cl	0	1	5	10	20	60	100	mg/L	344583 - 586, 687 - 695, 669 - 671
			Calibration Requirements		≥0.995		Calibration Results		1.0000		344283 - 303
		SO4	0	5	10	15	20/25	30	40	mg/L	↓ ↓
			Calibration Requirements		≥0.995		Calibration Results		1.0000		
10-7-03	JWB	CN⁻	0	.005	.01	.02	.05	.10	.20	mg/L	344958, 985, 953, 955, 954, 545, 546
			Calibration Requirements		≥0.995		Calibration Results		0.999914		345116 344955 - 960 344954, 957, 948, 571, 947, 345078
		Cl	0	1	5	10	20	60	100	mg/L	344813
			Calibration Requirements		≥0.995		Calibration Results		1.0000		

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Approved By: SOA 9-10-09 QA Officer
Approved By: Heather L. Brady Area Supervisor

Attachment 23.5
Batch Detail Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0711704 Page 1 of 2 Printed: 10/18/2007 3:06:03PM

Inorganic - Wet Chemistry, Waste Water, Method-Specific Preparation
(No Surrogate)
Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0709675	Cyanide, Total 4500-CN E	0710014	Cyanide, Total 4500-CN E	0710045	Cyanide, Total 4500-CN E
0710058	Cyanide, Total 4500-CN E	0710059	Cyanide, Total 4500-CN E	0710085	Cyanide, Total 4500-CN E
0710075	Cyanide, Total 4500-CN E	0710129	Cyanide, Total 4500-CN E	0710186	Cyanide, Total 4500-CN E
0710218	Cyanide, Total 4500-CN E	0710248	Cyanide, Total 4500-CN E	0710261	Cyanide, Total 4500-CN E
0710265	Cyanide, Total 4500-CN E	0710288	Cyanide, Total 4500-CN E	0710321	Cyanide, Total 4500-CN E
0710349	Cyanide, Total 4500-CN E				

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0711704-BLK1		Oct-03-07 07:44	VAS	50	50					BLANK	
0711704-BS1		Oct-03-07 07:44	VAS	50	50			7100016	50	LCS	
0711704-BS2		Oct-03-07 07:44	VAS	50	50			7100016	35	LCS	
0709675-01	B	Oct-03-07 07:44	VAS	50	50					[redacted]	
0710014-01	B	Oct-03-07 07:44	VAS	50	50					[redacted]	
0710014-03	B	Oct-03-07 07:44	VAS	50	50					[redacted]	
0710014-05	B	Oct-03-07 07:44	VAS	50	50					[redacted]	
0710014-07	B	Oct-03-07 07:44	VAS	50	50					[redacted]	
0710045-01	B	Oct-03-07 07:44	VAS	50	50					[redacted]	0 9 07
0711704-BLK2		Oct-08-07 07:44	VAS	50	50					BLANK	
0711704-BS3		Oct-08-07 07:44	VAS	50	50			7100016	50	LCS	
0711704-BS4		Oct-08-07 07:44	VAS	50	50			7100016	20	LCS	
0711704-MS1		Oct-08-07 07:44	VAS	50	50		0710075-06	7100016	50	MATRIX SPIKE	
0711704-MSD1		Oct-08-07 07:44	VAS	50	50		0710075-06	7100016	50	MATRIX SPIKE DUP	
0710058-01	D	Oct-08-07 07:44	VAS	50	50					[redacted]	
0710069-01	D	Oct-08-07 07:44	VAS	50	50					[redacted]	
0710085-02	B	Oct-08-07 07:44	VAS	50	50					[redacted]	
0710075-06	J	Oct-08-07 07:44	VAS	50	50					[redacted]	4 17 07
0710129-04	H	Oct-08-07 07:44	VAS	50	50					[redacted]	4 13 07

Comments

Analyst Initials

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TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0711704 Page 2 of 2 Printed: 10/18/2007 3:06:03PM

Inorganic - Wet Chemistry, Waste Water, Method-Specific Preparation
(No Surrogate)
Batch Comments: (none)

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0711704-BLK3		Oct-15-07 07:45	LMA	50	50					BLANK	
0711704-BS5		Oct-15-07 07:45	LMA	50	50			7100016	50	LCS	
0711704-BS6		Oct-15-07 07:45	LMA	50	50			7100016	20	LCS	
0710186-02	A	Oct-15-07 07:45	LMA	50	50					[redacted]	
0710218-01	A	Oct-15-07 07:45	LMA	50	50					[redacted]	4 12 07
0710245-01	D	Oct-15-07 07:45	LMA	50	50					[redacted]	
0710261-01	A	Oct-15-07 07:45	LMA	50	50					[redacted]	
0710265-01	D	Oct-15-07 07:45	LMA	50	50					[redacted]	
0710266-01	D	Oct-15-07 07:45	LMA	50	50					[redacted]	
0710321-01	A	Oct-15-07 07:45	LMA	50	50					[redacted]	
0711704-BLK4		Oct-16-07 07:00	LMA	50	50					BLANK	
0711704-BS7		Oct-16-07 07:00	LMA	50	50			7100401	50	LCS	
0711704-BS8		Oct-16-07 07:00	LMA	50	50			7100401	20	LCS	
0710349-01	A	Oct-16-07 07:00	LMA	50	50					[redacted]	4 12 07

Comments

Analyst Initials

tch_TriMatrix.rpt

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Approved By: 9-10-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Closed System Purge and Trap Extraction for Volatile Organic Compounds

SW-846 Method 5035A

APPROVALS:

Area Supervisor: Diane VanMale Date: 1-23-09
Diane L. VanMale

QA Officer: Date: 1-22-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser Date: 1/26/09
Jeff P. Glaser

Procedure Number: GR-04-105
Revision Number: 1.2

Date Initiated: 4/30/98
Effective Date: 2/20/09

Date Revised: 1/22/09
Pages Revised: All

By: Diane L. VanMale
Total Number of Pages: 20

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the preparation of soils, sediments and solid waste for volatile organic compounds (VOC) analysis. The procedure includes preparation of low level, high level and oily waste.

1.2 The low level soil procedure is designed to use a hermetically-sealed 40 mL sample vial, preserved with sodium bisulfate which is sealed at the time of sample collection. Since the sample is never exposed to the atmosphere after sampling, VOC losses during shipment, storage and analysis are minimized. The low level soil procedure also details steps for collecting a sample in an appropriate storage device which can then be shipped to the laboratory where it is preserved upon receipt. The applicable concentration range for low level samples is 0.001 to 0.200 mg/kg.

1.3 The high level soil procedure is based upon preserving the sample with methanol at the time of collection or upon receipt at the laboratory. It is applicable to soil samples with VOC concentrations over 0.200 mg/kg. The closed system purge and trap employed for low concentration samples is NOT appropriate for samples preserved in the field with methanol. All methanol preserved samples must be analyzed by diluting an aliquot of the methanol into laboratory reagent water then analyzing in accordance with TriMatrix standard operating procedure (SOP) GR-04-104, GR-03-105 or GR-03-121.

1.4 This procedure can be used for most volatile organic compounds with boiling points below 200° C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, reporting limits are approximately ten times higher because of poor purging efficiency.

1.5 Refer to the appropriate analytical standard operating procedure for a target analyte list and the analysis procedure.

1.6 Low level bulk soil analysis is outside of the scope of this procedure and is no longer formally recognized by the EPA as an acceptable option for VOC analysis. However, since some states and clients still require bulk soil sample collection and analysis, the technique is included in Attachment 3.0 (Bulk Soil Collection and Preparation Technique).

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

3.0 SUMMARY OF PROCEDURE

3.1 Low concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations in the range of 0.001 to 0.2 mg/kg. The VOCs are determined by collecting approximately 5 g of sample and shipping it to the laboratory to be analyzed, or preserved and then analyzed. One of the following procedures must be used for sampling and preparation.

3.1.1 Collect approximately 5 g of sample by using a metal or plastic coring tool, weighed in the field at the time of collection. Place the coring in a pre-weighted 40 mL VOA vial with a septum-sealed screw cap that already contains a stir bar. The vial is then sealed and shipped to the laboratory. The sample must then be frozen within 48 hours of collection. Another option is to preserve the sample at the time of collection with sodium bisulfate preservative solution. The vial is then sealed and shipped to the laboratory where it is stored in the refrigerator until time of analysis. For analysis, the entire vial is allowed to come to room temperature, and is then placed unopened into the autosampler.

3.1.2 Alternatively, samples may be collected in the field using an appropriate coring device which can also serve as the storage device for shipping such as the EnCore™ sampler. Constructed of an inert polymer, the EnCore™ has a coring/storage chamber designed to collect a 5 g sample with a press-on cap that creates a vapor tight seal to prevent analyte loss. After collection, the sample is shipped to the laboratory where it is transferred to a new pre-cleaned and tared 40 mL VOA vial containing a stir bar. The vial is then sealed and weighed, and the weight recorded. At this point the entire vial may be placed in the freezer until time of analysis, or the appropriate volume of sodium bisulfate preservative solution (1:1 w/v) may be added, and the sample stored in the refrigerator until time of analysis. As in Section 3.1.1, the entire vial is placed unopened into the autosampler for analysis.

3.1.3 Regardless of collection technique and immediately before analysis, laboratory reagent water, surrogates and internal standards (if applicable) are automatically added without opening the sample vial. The vial is then heated to 45° C, magnetically stirred and volatiles are purged into an appropriate trap using helium gas. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped volatile analytes into a gas chromatograph (GC) for analysis by the appropriate detector.

3.2 High concentration soil procedure - Applicable to soil and other solid samples with VOC concentrations greater than 0.2 mg/kg. The low concentration technique is not applicable to all samples. Particularly those containing high concentrations of VOCs which may overload the trap or exceed the calibration range. In such instances, use one of the procedures described below.

3.2.1 The first option is to collect a bulk sample in a glass container with a PTFE-lined lid. A 5 g portion of the collected sample is removed from the container at the laboratory and added to 5.0 mL of methanol to extract volatile organic constituents. After mixing by shaking or agitating using an ultrasonic bath, a volume of the solvent extract is quantitatively diluted into laboratory reagent water and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: This is the least desirable option as the sample contacts the atmosphere which may cause some volatile constituent losses.

3.2.2 The second option is to collect approximately 10 g of sample in a pre-weighed vial with a septum-sealed screw cap that contains 10 mL of methanol for extraction. For analysis, a volume of the solvent extract is quantitatively diluted into laboratory reagent water as in Section 3.2.1 and placed in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if

applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

3.2.3 The third option is to use a coring/storage device, such as the EnCore™ sampler described in Section 3.1.2. The EnCore™ storage chamber size can be 5 g or 25 g. Immediately after a sample is collected, seal the device and ship to the laboratory. Upon receipt, the laboratory will extrude the sample into a pre-tared 60 mL jar, weigh the sample and add methanol to the container in a 1:1 ratio of solvent volume to sample mass. At analysis, dilute an aliquot of the methanolic extract into laboratory reagent water as in Section 3.2.1 and transfer directly to a purge and trap vessel. Surrogates and internal standards (if applicable) are added by the before purging and analysis in accordance with the appropriate analytical procedure.

3.3 High concentration oily waste procedure - Applicable to oily samples with VOC concentrations greater than 0.2 mg/kg that can be diluted in a water-miscible solvent (not necessarily methanol). After demonstrating that a test aliquot is soluble in the solvent of choice, dilute a separate aliquot into the solvent. At analysis, dilute an aliquot of the water-miscible solvent extract into laboratory reagent water as in Section 3.2.1 and place in a new pre-cleaned VOA vial for analysis. Surrogates and internal standards (if applicable) are added by the autosampler immediately before purging and analysis in accordance with the appropriate analytical procedure.

Note: Oily samples not soluble in a water-miscible solvent are beyond the scope of this procedure and must be analyzed by direct injection analysis after dilution in n-hexadecane.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to each analytical procedure for the appropriate parameters list.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-104, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography/Mass Spectrometry*, latest revision

5.2 TriMatrix SOP GR-03-105, *Volatile Organic Compounds By Purge And Trap Capillary Column Gas Chromatography With Photoionization And Electrolytic Conductivity Detectors in Series*, latest revision

5.3 TriMatrix SOP GR-03-121, *Method for the Determination of Gasoline Range Organics*, latest revision

5.4 TriMatrix SOP GR-03-124, *Volatile Organic Laboratory Corrective Actions*, latest revision

5.5 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.6 TriMatrix SOP GR-10-113, *Laboratory Balance Calibration and Verification*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Sample contamination can cause elevate reporting limits or give false positives and come from a variety of sources. Improper sampling can contaminate samples at the job site. During shipment and storage, volatile organics (particularly methylene chloride and fluorocarbons) can diffuse through the septum. A trip blank prepared from laboratory reagent water containing the sodium bisulfate preservative and carried through all sampling and handling serves as a check on contamination for the low level procedure. For the methanol preservation technique, a trip blank must be prepared using purge and trap grade methanol which must then be processed through all sample preparation and analysis steps.

6.2 To minimize contamination, the volatiles analysis laboratory must be free of solvents. The volatiles sample storage area must be isolated from atmospheric sources of methylene chloride and other solvents. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing must be stainless steel or copper. Clothing previously exposed to methylene chloride fumes during semi-volatiles extraction can contribute to sample contamination and must not be worn into the volatiles laboratory.

6.3 The sodium bisulfate preservative acts to lower sample pH which inhibits biological degradation of aromatic compounds. However, under acidic conditions, highly reactive compounds such as 2-Chloroethylvinyl ether are unstable and will be lost. Acid preservation can also degrade methyl-t-butyl ether (MtBE) to tert-butyl alcohol during purge and trap. Additional analytes affected include vinyl chloride, styrene and other fuel oxygenate ethers. If these analytes are to be reported, collect a second sample set without sodium bisulfate preservative. Acidification of certain soil types with sodium bisulfate can produce an acetone artifact which is typically between 0.025 and 0.100 mg/kg.

6.4 For the appropriate corrective actions when encountering contamination, refer to TriMatrix SOP GR-03-124.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used in this procedure has not been precisely defined. However, each chemical must be treated as a potential health hazard and exposure must be reduced to the lowest possible level by all means available.

7.3 The laboratory maintains a current reference file of material safety data sheets (MSDS) available to all personnel.

7.4 Many of the analytes tested have been classified as known or suspected carcinogens. Consequently, all neat standard materials and standard solutions must be opened only in a fume hood.

7.5 Appropriate personal protective equipment including a laboratory coat, approved safety glasses and disposable gloves must be worn in the laboratory when handling any chemical. The only exception for wearing personal protective equipment is when working at a computer station in the laboratory.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 The specific sample container required depends on the purge and trap system. TriMatrix Laboratories currently uses the needle sparge type purging device for all low level soils. This system requires a 40 mL borosilicate glass volatile organic analysis (VOA) vial.

8.2 The specific preparation for a sample vial depends on the expected concentration range with specific preparation techniques for low concentration soils, high concentration soils and solid waste. Sample vials must be prepared in the laboratory or other controlled environment then sealed and shipped to the collection site. Disposable gloves must be worn during the preparation steps which are as follows:

8.2.1 The following instructions apply to the preparation of vials used in the collection of low concentration soil samples that are to be preserved in the field.

8.2.1.1 Add a clean magnetic stirring bar to each clean vial.

8.2.1.2 Add 5.0 mL of 20% sodium bisulfate preservative solution to each vial. This should be sufficient to ensure a sample pH of <2 to prevent biodegradation.

8.2.1.3 Seal the vial with the screw-cap septum lid.

8.2.1.4 Affix a label to each vial. This eliminates the need to label vials in the field and assures that the vial tare weight is included on the label.

8.2.1.5 Weigh the prepared vial to the nearest 0.01 g, record the tare weight and write it on the label.

8.2.1.6 Because volatile organics will partition into the vial headspace from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes and internal standards (if applicable) must only be added after sample has been added. Surrogates, matrix spikes and internal standards (if applicable) must be injected automatically by the autosampler just prior to analysis.

8.2.2 When high concentration samples are collected without a preservative, a variety of sample containers may be employed including 40, 60 and 125 mL glass jar with a PTFE liner. This collection technique must only be used when sample solubility in methanol is questionable.

8.2.3 The following steps apply for high concentration soil samples collected and preserved in the field with methanol as described in Section 3.2.

8.2.3.1 Use pre-tared 40, 60 or 125 mL jars with screw cap lids and PFTE liners.

8.2.3.2 To obtain the pre-tared weighing, weigh the container with the label, lid and liner in place then record the tare weight on the label to the nearest 0.01 g. The mass of the ink is negligible.

8.2.3.3 Include a purchased 10 mL ampule of purge and trap methanol with each sample container.

8.2.4 When oily waste samples are known to be soluble in methanol, sample containers may be used as described in Section 8.2.3, preserving with methanol. However, when methanol solubility is unknown, collect the sample without preservative in a 40, 60 or 125 mL glass jar with a PTFE liner.

8.2.5 Preservation by freezing to less than -7° C may be used unless otherwise specified and is as follows:

8.2.5.1 If freezing at collection, add sample to an empty sample container and freeze to less than -7° C until analysis.

Note: When freezing, always place the sample container on its side to prevent the glass container from breaking.

8.2.5.2 Samples received by the laboratory unfrozen and at 4 ±2° C, and are within 48 hours of collection may be frozen to less than -7° C at the time of receipt.

8.2.5.3 Samples must be thawed for preparation and analysis within 14 days of collection.

8.2.5.4 Thawed samples must be prepared and analyzed within 24 hours of thawing.

8.3 Sample Collection

8.3.1 Collect samples according to the procedure outlined in the project sampling plan. In general, after a fresh surface of the solid material is exposed, collect a subsample in the least amount of time and with as little disruption as possible to minimize the loss of volatiles. Use a metal or rigid plastic coring tool to collect the subsample. These devices help maintain sample structure during collection and transfer to the sample container. When inserting the coring device into solid material, use care not to trap air behind the sample. If air is trapped, it can cause VOC loss by passing through the sample or cause the sample to be pushed from coring device before a transfer can be made to the sample container. The following devices have been designed to prevent headspace during collection and are approved for sampling:

8.3.1.1 EasyDraw Syringe™ with Powerstop Handle™

8.3.1.2 Terra Core™

8.3.1.3 EnCore™

8.3.2 Low level soil samples may be collected as follows:

8.3.2.1 Collect approximately 5 g of soil using a coring device as described in Section 8.3.1. After taking the sample, remove the filled corer and quickly wipe the barrel with a clean disposable wipe.

8.3.2.2 If a sample is to be preserved at collection, add to a sample jar containing sodium bisulfate preservative by holding the sample jar at an angle then extrude into the

container to minimize splashing. Quickly brush or wipe the vial threads to remove residual soil and immediately seal the vial tightly with the septum screw cap.

Note: An improper seal due to soil remaining on the sample jar threads and improper lid tightening are the primary factors in volatiles loss during sample collection. This can also cause problems during analysis by preventing the vial from pressurizing during the purge step, resulting in low or no recovery.

8.3.2.3 If a sample is shipped to the laboratory for preserving, cap the open end of the coring/storage device after ensuring all sealing surfaces are clean then store and transport at 4 ±2° C.

Note: An individual EnCore™ is needed for each sample aliquot.

8.3.2.4 If samples are expected to contain target analytes over a wide concentration range and high level samples are not being collected, collect an additional sample aliquot in a low concentration preserved VOA vial with 1 - 2 g rather than 5 g. If necessary, the low mass sample can be analyzed for analytes in the 5 g sample that exceed the calibration range.

8.3.2.5 Samples that contain carbonate minerals may effervesce upon contact with the sodium bisulfate preservative. If the gas volume generated is small, volatiles loss may be minimal. However, if large gas volumes are generated, volatiles are likely to be lost and the vial may rupture. If carbonate minerals are expected, collect a test sample to check for effervescence. If the sample effervesces when sodium bisulfate is added, discard the test sample and collect additional sample without preserving.

8.3.3 High level soil samples may be collected as follows:

8.3.3.1 Preserve in the field as follows:

8.3.3.1.1 Use a coring device to collect a 5 g or 25 g sample as soon as possible after solid surface material is exposed. Wipe the exterior of the collection device with a clean disposable wipe.

8.3.3.1.2 Quickly transfer to a 40 mL vial for 5 g, or to a 60 or 125 mL sample jar for a 25 g sample.

8.3.3.1.3 Add a volume of methanol in a 1:1 ratio of methanol volume to sample mass.

8.3.3.2 Preserved in the laboratory as follows:

8.3.3.2.1 If a sample is to be preserved in the laboratory, collect in an EnCore™ sampler, cap the open end after ensuring all sealing surfaces are clean and store at 4 ±2° C until transport.

8.3.3.2.2 Upon receipt at the laboratory, the sample must be extruded from the EnCore™ into a new sample jar then weighed and a 1:1 ratio of methanol volume to sample mass added. Refer to Section 13.0 for detailed instructions.

8.4 Sample Storage

8.4.1 Once in the laboratory, store samples at 4 ±2° C (or freeze to -7° C within 48 hours of collection) until analysis. The volatiles sample storage area must be free of organic solvent vapors.

8.4.2 All samples must be analyzed as soon as practical and within the designated holding time. Samples not analyzed within the holding time must be reported as exceeding the holding time. Results must be reported as estimated minimum values.

8.4.4 When low concentration samples are strongly alkaline or highly calcareous, the sodium bisulfate preservative solution may not be strong enough to reduce pH to below 2.

8.4.4.1 When low concentration soils are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required for preservation. Such steps include:

8.4.4.1.1 Addition of a larger sodium bisulfate preservative volume

8.4.4.1.2 Storage at below -10° C (taking care not to fill the vial it breaks from expansion)

8.4.4.1.3 Significantly reducing the maximum holding time.

8.4.4.2 The preservation used must be clearly described in the sampling and quality assurance (QA) project plan for distribution to both field and laboratory personnel.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Sample containers are as follows:

9.1.1 Vials, 20 or 40 mL with PTFE-lined septum-sealed screw cap lids

Note: Examine each vial prior to use to ensure the vial has a flat, uniform sealing surface.

9.1.2 Glass borosilicate jars, 60 or 125 mL with PTFE-lined screw cap lids

9.2 EnCore™ sampler (EnNovative Technologies, Inc.)

9.3 microSyringes, 100 µL and 1000 µL

9.4 Balance, top-loading, capable of accurately weighing to 0.01 g

9.5 Disposable Pasteur pipets

9.6 Magnetic stirring bars, PTFE of the appropriate sizes to fit sample containers.

Note: Stirring bars may be reused provided they are thoroughly cleaned between each use.

9.7 Field equipment is as follows:

9.7.1 Terra Core™ sampler, EnNovative Technologies, Inc.

9.7.2 EasyDraw Syringe™ with Powerstop Handle™, US Oil Company

9.7.3 Portable balance, capable of accurately weighing to 0.01g

9.8 Volumetric flask, 500 mL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no routine preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, organic-free

11.2 Methanol (CH_3OH), purge and trap grade

Note: Store away from other solvents

11.3 The low concentration sample preservative is prepared as follows:

11.3.1 Sodium bisulfate ($NaHSO_4$), certified

11.3.2 Weigh 100 g of $NaHSO_4$ into a 500 mL volumetric flask. Mix to dissolve the $NaHSO_4$ then bring to volume with laboratory reagent water (organic-free). Record the preparation in the laboratory information system (Element™) and/or in the reagent preparation logbook. Concentration is 20% (w/v) $NaHSO_4$.

11.3.3 Expiration is 6 months from the date prepared.

11.4 Refer to the appropriate analytical procedure for guidance on preparing internal standards and/or surrogates.

12.0 STANDARDS PREPARATION

12.1 There are no analytical standards directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Prepare low level soils collected with an Encore sampler (5 g) as follows:

13.1.1 Place a 40 mL vial on the top-loading balance and tare with the cap off.

13.1.2 Empty the 5 g Encore sample into the vial.

13.1.3 Weigh to the nearest 0.01 g and record on the vial label.

13.1.4 Add the equivalent volume of 20% $NaHSO_4$ to the vial to achieve a 1:1 ratio of sample mass to preservative volume.

13.1.5 Add a clean magnetic stir bar to the vial and seal the vial.

13.1.6 Remember to attach the label to the vial.

Note: Sodium bisulfate degrades 2-chloroethylvinyl ether. TriMatrix Laboratories has demonstrated that this preservative will not give acceptable quality control recovery so 2-chloroethylvinyl ether will not be reported when used.

13.2 Prepare high level soils collected with an Encore sampler (25 g) as follows:

13.2.1 Place 60 mL sample jar on the top-loading balance and tare with the cap off.

13.2.2 Empty the 25 g Encore sample into the jar

13.2.3 Weigh to the nearest 0.01 g and record on the jar label.

13.2.4 Add the equivalent volume of methanol to the vial to achieve a 1:1 ratio of sample mass to preservative volume then seal the jar.

13.2.6 Remember to attach the label to the jar.

13.2.7 Shake the sample jar for 2 minutes. Then sonicate the jar for 20 minutes.

13.2.8 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.2.9 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.3 Prepare high level soils pre-preserved in the field as follows:

13.3.1 Weigh the sample jar containing soil sample and methanol, and record on the label.

13.3.2 Enter the sample jar size and mass into the Volatile Soil Receipt Information spreadsheet located on the laboratory intranet library (Attachments 1 and 2).

13.3.4 The spreadsheet determines if additional methanol needs added and/or whether the sample can be used, as follows:

13.3.4.1 If the mass collected in ratio with the methanol added is less than a 1:1 ratio of sample to preservative, consult the project chemist. The sample must be reported with a low weight narration.

13.3.4.2 If the mass collected requires additional methanol that exceeds the sample jar volume, consult the project chemist. The sample may be rejected for analysis.

13.3.4.3 If the mass collected in ratio with the methanol added is greater than a 1:1 ratio of sample to preservative, add the appropriate methanol volume.

13.3.5 Shake the sample jar for 2 minutes. Then sonicate the sample jar for 20 minutes.

13.3.6 After shaking and sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.3.7 After settling, carefully decant enough methanol extract to fill a 4 mL vial without headspace and seal with the septum screw cap. Store at 4 ±2° C until analysis.

13.4 Prepare oily waste samples soluble in a water-miscible solvent as follows:

13.4.1 Place a 20 mL vial on the top-loading balance and tare with the cap off.

13.4.2 Weigh out 1 g of sample to the nearest 0.01 g.

13.4.3 Add 10 mL of water-miscible solvent and quickly cap the vial.

13.4.4 Shake for 2 minutes. After shaking or sonicating, place in a refrigerator at 4 ±2° C for one hour to let settle.

13.4.5 After settling, carefully decant enough of the mixture to fill a 4 mL vial without headspace and seal with the septum screw cap. A 1:50 dilution in the water-miscible solvent will be made unless the analyst suspects higher volatile compound concentrations.

Note: Oily samples that are not soluble in a water-miscible solvent are beyond the scope of this procedure.

14.0 CALIBRATION PROCEDURES

14.1 There are no calibration procedures directly associated with this procedure.

15.0 ANALYTICAL PROCEDURE

15.1 Analyze a low level soil as follows:

15.1.1 Remove the sample vial from storage and bring to room temperature. Shake the vial gently, to ensure the contents move freely and that stirring will be effective. Place the sample vial in the autosampler.

15.1.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.1.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.1.4 If any target analyte concentration exceeds the calibration range, reanalyze by the high concentration procedure. Reanalysis need only address analytes exceeding the calibration range.

15.1.5 Alternatively, analyze the 1-2 g sample aliquot if collected (Refer to Section 8.2.2.7). Reanalysis need only address analytes exceeding the calibration range in the 5 g sample analysis.

15.2 Analyze a high level soil or waste as follows:

15.2.1 Pipet 1.0 mL of solvent extract or diluant to a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free). Dilute to volume with laboratory reagent water organic-free) and invert 3 times to mix. Transfer to a 40 mL vial and place on the autosampler for analysis.

15.2.2 For matrix spiked samples, add the matrix spiking solution described in the appropriate analytical procedure. The spiking solution concentration and the volume injected will vary.

15.2.3 Perform the qualitative and quantitative analysis in accordance with every step in the analytical procedure.

15.2.4 If any target analyte concentration exceeds the calibration range, reanalyze by diluting a lesser amount of the solvent extract into a 50 ml volumetric flask. Reanalysis need only address analytes exceeding the calibration range.

16.0 CALCULATIONS AND DATA HANDLING

16.1 There are no analytical calculations directly associated with this procedure.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Volatiles Soil Receipt Information Spreadsheets are to be archived as raw data.

17.2 Refer to the analytical procedure for complete data reporting and deliverable requirements.

18.0 QUALITY ASSURANCE

18.1 Refer to the analytical procedure for complete quality control/quality assurance requirements.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Refer to the analytical procedure for demonstration of capability study and instrument validation requirements.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, New Methods*, Method 5035A, "Closed system Purge and trap and Extraction for Volatile Organics in Soil and Waste Samples", Draft Revision 1, July 2002

23.0 ATTACHMENTS

23.1 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

23.2 Volatile Soil Sample Receipt Information Spreadsheet, Page 1

Approved By: PD 1-22-09 **Approved By:** DW 1-23-09

QA Officer **Area Supervisor**

23.3 Bulk Soil Collection and Preparation

Attachment 23.1
Volatile Soil Sample Receipt Information Spreadsheet, Page 1

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Low Level Soils	Yes	No	N/A
Were Samples Received in 40 mL VOA Vials Containing a Stir Bar and Pre-Preserved with Sodium Bisulfate?			
Were Samples Received Non-Preserved in Encore Samplers?			
If Received in Encore Samplers, Was Sample Received Within 48 Hours?			
Volatile Lab Informed That Samples Must Be Preserved AND Analyzed Within 48 Hours?			

High Level Soils	Yes	No	N/A
Extra Sample Containers Received for Dry Weight Determination?			
MeOH Trip Blank Received (if MeOH not added from sealed ampules)?			
Samples Collected in Which of the Following Ways?			
Tared 40, 60, or 120 mL Containers:			
Were Containers Supplied by TriMatrix?			
Were Samples Received Within 4 Days After Collection, and Were They MeOH Preserved?			
Packed With No Headspace into Brass Tubes:			
Were Samples Received Within 4 Days After Collection, and Preserved Within 2 Hours of Sample Receipt?			
Packed With No Headspace in En Core Samplers:			
Were Samples Received Within 40 Hours After Collection and Preserved Within 48 Hours of Collection?			

Follow-Up	Yes	No	N/A
Has Soil Weight Table Been Completed?			
Has Project Chemist Been Informed of any Out-of-Compliance Samples?			

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Approved By: DM 1-22-09 QA Officer

Approved By: DW 1-23-09 Area Supervisor

Attachment 23.2
Volatile Soil Sample Receipt Information Spreadsheet, Page 2

TriMatrix Laboratories, Inc.

Volatile Soil Sample Receipt Information Sheet

Client: _____ Date Received: 08/29/2003 Sheet Completed By: JDM

Project-Submittal: _____ Date Form Completed: 09/03/2003 Sheet Reviewed By: _____

Client Sample Identification	TriMatrix Sample Number	Vial Size: 40, 60, or 120 mL	Empty Weight of Bottle (g)	MeOH Added: 10, 25, or 50 mL	Full Weight of Bottle (g)	Date Sample Weighed	Time Sample Weighed	Weight of Soil (g)	Low Wt. Flag Required?	Additional MeOH Required?	Accept/ Reject Sample	MeOH to Add (mL)	mL MeOH Added: By, Date, Time
1	341527	40	25.5	10	43.6			10.2			Accept Sample		
2	341528	40	25.6	10	43.6			10.1			Accept Sample		
3	341543	40	25.6	10	43.4			9.9			Accept Sample		
4	341544	40	25.3	10	43.0			9.8			Accept Sample		
5	341546	40	25.6	10	43.7			10.2			Accept Sample		
6	341547	40	25.3	10	43.3			10.1			Accept Sample		
7	341548	40	25.3	10	43.3			10.1			Accept Sample		
8	341549	40	25.3	10	43.4			10.2			Accept Sample		
9	341550	40	25.4	10	43.9			10.6			Accept Sample		

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Approved By: PD 1-22-09 QA Officer

Approved By: DW 1-23-09 Area Supervisor

Attachment 23.3
Bulk Soil Collection and Preparation

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the collection and preparation of bulk soils and sediments for volatile organic compounds (VOC) analysis. Low level bulk soil analysis is no longer formally recognized by the EPA as an acceptable option for VOCs. However, some states and/or clients still request bulk soil sample collection and analysis.

2.0 SUMMARY OF PROCEDURE

2.1 Bulk soil samples are collected with minimal headspace in either 60 or 125 mL glass jars. After collection, samples must be stored at 4 ±°2 C until transport to the laboratory.

2.2 When received by the laboratory, samples remain stored at 4 ±°2 C until the analyst transfers a 1-5 g subsample to a new 40 mL vial containing a stir bar for preparation.

2.3 The vial is then loaded onto the autosampler for analysis.

3.0 INTERFERENCES AND CORRECTIVE PROCEDURES

3.1 Bulk soil sampling is inaccurate and produces results that are biased low. Bulk samples can lose more than 90% VOC content prior to analytical measurement.

3.2 Reasons for such losses include the following:

3.2.1 Volatilization from exposure of the solid surface near the time of collection

3.2.2 Volatilization from intermediate storage containers

3.2.3. Volatilization from the disaggregation of the solid material during collection

3.2.4 Volatilization from failed seals on sample jar lids

3.2.5 Volatilization during laboratory subsampling

3.2.6 Biodegradation (primarily aromatic compounds) during storage

3.2.7 Chemical reactions during storage

4.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

4.1 Solid samples are collected using a stainless steel spatula-type device to completely fill a 40, 60 or 125 mL glass jar.

4.2 The sample container is then closed using PTFE-lined caps and stored at 4 ±2° C with ice throughout the collection event, during transport to the laboratory and during storage at the laboratory until just prior to analysis.

4.3 The holding time for samples is 14 days from the time of collection.

4.4 If samples require high level preparation/analysis based on low level results or the sample matrix, a subsample must be extracted using methanol.

4.5 The holding time for a methanol extract is also 14 days from the time of collection.

5.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

5.1 Refer to Section 9.0 in the main body of the procedure.

5.0 SAMPLE PREPARATION

6.1 Prepare a low level sample as follows:

6.1.1 From the original sample container, remove a representative subsample and transfer to a tared 40 mL vial containing a PTFE stir bar. Record the weight to the nearest 0.01 g. Use a 5 g sample unless higher level concentrations are expected and a smaller aliquot is needed.

Note: Do not use less than a 1 g sample aliquot.

6.1.2 Quickly wipe any residual soil from the vial threads and seal then load onto the instrument autosampler. The autosampler will add 10 mL of laboratory reagent water (organic-free) and applicable internal standards and/or surrogates immediately prior to the purge cycle.

6.2 Prepare a high level sample as follows:

6.2.1 If low level analysis results are outside the instrument calibration range or if the sample matrix indicates an unsuitability for low level analysis, the sample requires high level preparation and analysis.

6.2.2 From the original sample jar, remove a representative subsample and transfer to a tared 20 mL sample vial. Weigh out 5 g to the nearest 0.1 g and record on the label. Remove any residual

Attachment 23.3 (continued)
Bulk Soil Collection and Preparation

sample from the vial threads and pipet in 5.0 mL of methanol. Cap and shake the container by hand for 2 minutes. Place in the refrigerator at 4 ±2° C to settle out.

Note: Some samples may require letting stand overnight for sufficient phase separation.

6.2.3 Once the phases separate, decant the methanol phase into a new 4 mL vial for storage. Store at 4 ±2° C until analysis.

Note: The methanol must be withdrawn from the sample within 24 hours of extracting the sample to keep the extraction times constant.

7.0 CHEMICALS AND REAGENTS

7.1 Refer to Section 11.0 in the main body of the procedure.

8.0 ANALYTICAL PROCEDURE

8.1 Once prepared, low level soils can be directly loaded onto the purge and trap autosampler.

8.2 The autosampler will add laboratory reagent water (organic-free), internal standards and surrogates.

8.3 If the results of the analysis are outside the calibration range, reanalyze as a high level sample as follows:

8.2.1.1 Pipet 1.0 mL of extract into a 50 mL volumetric flask approximately ¾ full of laboratory reagent water (organic-free).

8.2.1.2 Dilute to volume with laboratory reagent water (organic-free).

8.2.1.3 Transfer to a 40 mL vial and place vial on autosampler for analysis.

8.4 If analysis of the high level preparation exceeds the calibration range, reanalyze by using an appropriate smaller extract volume to dilute into the 50 mL volumetric flask or perform a serial dilution until response is with the calibration range.

8.5 Follow the main body of the procedure to complete analysis and data reduction.

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Organic Carbon
Walkley-Black

Agronomy, Methods of Soil Analysis 29-3.5.2

APPROVALS:

Area Supervisor: Heather L. Brady — Date: 2-12-09
Heather L. Brady

QA Officer: _____ — Date: 2-9-09
Tom C. Boocher

Operations Manager: _____ — Date: 12/12/09
Jeff P. Glaser

Procedure Number: GR-06-105
Revision Number: 2.4

Date Initiated: 7/29/97
Effective Date: 3/9/09

Date Revised: 2/9/09
Pages Revised: All

By: Heather L. Brady
Total Number of Pages: 14

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to approximating organic carbon in non-carbonized soils and sediments, with a zero average organic carbon oxidation state.

1.2 The minimum reporting limit is 0.1% (dry weight) as organic carbon according to the following method requirements:

$$\text{Minimum Reporting Limit, \% } C^0 = \frac{10 \text{ mg } C^0}{10\text{g Soil}} \Bigg| \; 100\%$$

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Methods of Soil Analysis Part 2, Chemical and Microbiological Properties, 2nd Edition,* Number 9 (Part 2), Agronomy, 1982, Section 29-3.5.2, "Walkley-Black Procedure"

3.0 SUMMARY OF PROCEDURE

3.1 Organic carbon is determined by an oxidation-reduction reaction in which potassium dichromate is added to a sample, followed by addition of concentrated sulfuric acid. Dichromate ($Cr_2O_7^{2-}$) oxidizes organic carbon to CO_2 in an acidic medium as follows:

$$2Cr_2O_7^{2-} + 3C^0 + 16H^+ \rightarrow 4Cr^{3+} + 3CO_2 + 8H_2O$$

3.2 The reduced dichromate is quantitatively related to oxidized organic carbon. Any remaining $Cr_2O_7^{2-}$ is reduced by Fe^{2+} from the ferrous sulfate titrant. The endpoint at which all $Cr_2O_7^{2-}$ has been reduced is indicated by the maroon color of an o-phenanthroline ferroin indicator.

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

4.0 PARAMETER OR COMPOUND LIST

4.1 Organic Carbon

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-109, *Laboratory Waste Disposal*, latest revision

5.2 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Significant chloride (Cl^-) concentration in soil will give a positive error.

6.2 Fe^{2+} in soil will also give a positive error. Oven drying soils at 103 to 105° C oxidizes Fe^{2+} to Fe^{3+} and a more accurate organic carbon result can be determined. To minimize interference from Fe^{0}, samples must not be ground or sieved with ferrous equipment.

6.3 Manganese oxide (MnO_2) may give a negative error. However, only a small percentage of the total MnO_2 actually takes part in the redox reaction. This reaction error should not be serious enough to need correcting for most matrices.

6.4 Analysis of carbonized materials such as charcoal, graphite, coal, and soot containing large quantities of elemental carbon is beyond the scope of this procedure. Soil analysis must only be performed on soil or sediment presumed to have an average carbon oxidation state of zero.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Extreme caution must be exercised when handling potassium dichromate. All spent potassium dichromate must be bottled and labeled as hazardous waste.

7.3 A fume hood must be used when handling or dispensing concentrated H_2SO_4 (refer to section 15.3).

7.4 Personal protective equipment including disposable gloves, approved safety glasses, and laboratory coats must be worn in the laboratory when handling samples, reagents, acids or other materials used in this procedure.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING PROCEDURES

8.1 Soils must be collected in inert bottles (plastic or glass), with no preservative added.

8.2 Analysis must be performed within 28 days of sample collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Burette, 50 mL, type A

9.2 Erlenmeyer flasks, 500 mL

9.3 Type A volumetric pipet, 10 mL

9.4 Sieve (non-ferrous), 0.5 mm

9.5 Mortar & pestle, porcelain or other non-ferrous material

9.6 Volumetric flasks, Type A

9.7 Macropipette

9.8 Bottle top dispenser for concentrated H_2SO_4, capable of dispensing 20 mL

9.9 Stir bars

9.10 Stir plate, unheated

9.11 Balance, top loading, capable of weighing to at least 0.01 g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q system

11.2 Potassium dichromate ($K_2Cr_2O_7$), ACS, 1N

11.2.1 In a 1000 mL volumetric flask, dissolve 49.04 g of ACS grade $K_2Cr_2O_7$ (dried at 103 - 105° C)

11.2.2 Dissolve the dichromate by mixing or swirling, then dilute to volume with laboratory reagent water.

11.2.3 Expiration is six months from the date made.

11.3 Sulfuric acid (H_2SO_4), concentrated (18M), ACS

11.4 Ferroin indicator (0.025 M Phenanthroline Ferrous Sulfate Complex), purchased commercially

11.5 Ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$) ACS grade solution (0.5N)

11.5.1 In a 1000 mL volumetric flask, dissolve 140 g $FeSO_4 \cdot 7H_2O$ in laboratory reagent water.

11.5.2 Add 15 mL of concentrated H_2SO_4.

11.5.3 Cool and dilute to volume with laboratory reagent water.

11.5.4 Expiration is three months from the date made unless the normality check of Section 14.2 is unacceptable.

11.6 Potassium hydrogen phthalate or KHP (HOOC-C_6H_4-COOK), acidimetric standard ACS, oven-dried

11.6.1 Place approximately 25 g KHP in a glass beaker and dry at 103-105° C for 2 hours.

11.6.2 Remove from the oven and immediately place in a desiccator. Cool to room temperature in the desiccator then transfer to a permanent storage bottle.

11.6.3 Dried KHP will not need re-dried if stored in the desiccator and is only opened to remove material.

11.6.4 Dispose of and replace dried KHP annually.

11.7 Ottawa sand – dried @ 550°C for 1 hour.

12.0 STANDARDS PREPARATION

12.1 Laboratory control sample (10,000 mg/L C^0): Dissolve 2.128 g oven-dried KHP in a 100 mL volumetric flask with laboratory reagent water. Dilute to volume with reagent water and store at 4 ±2° C in an amber bottle. Expiration is six months from the date made.

12.2 Record all standard preparations in the LIMS system (Element™) and/or the paginated standards logbook, each with a unique ID number. Refer to Attachment 23.1 for an example of the standards logbook.

13.0 SAMPLE PREPARATION

13.1 Samples must be dried at 103-105° C until sufficiently dry to be ground into powder and sieved. Typically, the drying time is over-night. Record drying time data on the raw data spreadsheet (refer to Attachment 23.2), including the following:

13.1.1 Balance used

13.1.2 Date and time oven drying begins

13.1.3 Date and time oven-drying ends

13.1.4 Dish mass, in g

13.1.5 Pre-dried soil and dish mass, in g

13.1.6 Post-dried soil and dish mass, in g

13.2 Grind the dried soil in a porcelain mortal and pestle.

13.3 After grinding, pass through a 0.5 mm non-ferrous sieve.

13.4 Subsample the sieved material for analysis.

14.0 CALIBRATION PROCEDURES

14.1 Perform a titration blank (without soil or Ottawa sand) before processing samples to standardize the ferrous sulfate normality as follows:

14.1.1 Add 10.0 mL of 1N $K_2Cr_2O_7$ to a 500 mL Erlenmeyer flask.

14.1.2 Carefully add 20 mL of concentrated H_2SO_4, directing the stream into the solution. Immediately, swirl gently until the reagents mix. This must be done inside a fume hood.

14.1.3 Let the flask sit for approximately 30 minutes then add 200 mL of laboratory reagent water.

14.1.4 Add 5-10 drops of ferroin indicator and titrate with 0.5N $FeSO_4$. The solution will turn bluish-green when approaching the endpoint. When the solution turns dark green, add titrant dropwise until the color changes sharply to maroon, which is the endpoint.

14.1.5 Read and record the titrant volume used to at least the nearest 0.05 mL.

14.2 If titrant normality is not 0.500 ±0.05N, clean the burette and remake the titrant solution and/or the 1N $K_2Cr_2O_7$.

14.3 Calculate ferrous sulfate normality based on the blank (refer to section 16.2).

15.0 ANALYTICAL PROCEDURE

15.1 Transfer up to 10.00 g of sieved material containing 10 to 25 mg organic carbon to a 500 mL Erlenmeyer flask.

15.2 Pipette 10.0 mL of 1N $K_2Cr_2O_7$ into the flask and gently swirl to disperse the soil into a suspension.

15.3 Carefully add 20 mL of concentrated H_2SO_4, directing the stream into the suspension. Immediately, swirl gently until the soil and reagents mix. Place the flask on a magnetic plate, add a stir bar, and stir for one minute. This must be done inside a fume hood.

15.4 Let the flask sit for approximately 30 minutes.

15.5 After 30 minutes add 200 mL of laboratory reagent water. At this point filter the suspension if the endpoint will not be clearly discernable.

15.6 Add 5-10 drops of ferroin indicator and titrate with 0.5N $FeSO_4$. The solution will turn bluish-green when approaching the endpoint. When the solution turns dark green, add titrant dropwise until the color changes sharply to maroon.

15.7 Repeat a titration with less sample if less than 4.0 mL of titrant is used to reach the endpoint.

15.8 All results must be recorded on the raw data spreadsheet (Refer to Attachment 23.2). After titration is complete, empty the flask contents into the waste bottle labeled "TOC soil waste", to be disposed of as dichromate waste. Make sure waste bottles are labeled as dichromate waste, with the date filled on the label as well.

16.0 CALCULATIONS AND DATA HANDLING

16.1 All measurements and reagents used must be recorded on the raw data spreadsheet (refer to Attachment 23.2). Data must then be entered into a spreadsheet (refer to Attachment 23.3) to calculate the final concentration. The spreadsheet duplicates the logbook format for ease of data entry.

16.2 Normality of the $FeSO_4$ titrant is determined from the standardization as follows:

$$\text{normality of } FeSO_4 \text{ titrant} = \frac{10}{\text{mL of titrant}}$$

16.3 Percent organic carbon is calculated by the spreadsheet, as follows:

$$\text{Organic C, \%} = \frac{[10 - (\text{Normality of } FeSO_4 \times \text{mL of } FeSO_4)](0.003)(100)}{\text{g dried soil}} \times 1.3$$

17.0 DATA REPORTING AND DELIVERABLES

17.1 The following must be recorded on the preparation batch report or elsewhere for each batch analyzed:

17.1.1 The LCS, BLK, DUP and sample final concentration with units.

17.1.2 Work order, date prepared, analyst, initials of the peer reviewer, stock standard numbers.

17.1.3 Any batch comments that need made.

17.2 Refer to Attachment 23.4 for a preparation batch report example.

18.0 QUALITY ASSURANCE

18.1 Method quality control (QC) must be analyzed with each analysis batch of up to 20 samples. Matrix QC must be analyzed every 10 samples or with each analysis batch of less than 10 samples, whichever is more frequent.

18.1.1 Method QC consists of the laboratory control sample (LCS) and a method blank (BLK).

18.1.1.1 The approved LCS is 1.0 mL of 10,000 mg/L C^0 (refer to Section 12.1) pipetted into a 500 mL Erlenmeyer flask containing 1.0 g of dried Ottawa sand and analyzed as a sample. The expected value is 1.0 %. Acceptance limits are the control limits listed in Element. The expected value is calculated as follows:

$$\% C^0 = \frac{2.128 \text{ g KHP}}{100 \text{ mL}} \left| \frac{\text{mole KHP}}{204.23 \text{ g KHP}} \right| \frac{8 \text{ mole } C^0}{\text{mole KHP}} \left| \frac{12.011 \text{ g } C^0}{\text{mole } C^0} \right| \frac{1.0 \text{ mL}}{1.0 \text{ g Sand}} \left| 100\% \right.$$

18.1.1.2 A BLK is 10 g dried Ottawa sand analyzed as a sample. Blank results must be less than the reporting limit. If a blank is not less than the reporting limit, address the contamination problem then perform an acceptable blank.

18.1.2 Matrix QC consists of a digestion/titration duplicate (DUP).

18.1.2.1 A DUP is a second aliquot of dried sample analyzed the same as the first.

18.1.2.2 Acceptance limits for DUP percent difference are the control limits listed in Element.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate an ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) is required annually.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare and analyze four LCS spikes as samples (section 18.1.1.1). Process the four spikes following every step in the procedure. Calculate average percent recovery and relative standard deviation of the average. Average percent recovery must be between within the LCS acceptability window listed in Element. Relative standard deviation must be less than 20%.

19.1.1.2 If either criterion is not met, locate and correct the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If this occurs, correct the procedure or techniques and repeat the study successfully.

19.1.1.3 Samples may not be analyzed by any analyst until an initial demonstration of capability study has been successfully completed. Copies of successful IDC spreadsheets must be submitted to the Quality Assurance Department for training documentation.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A CDC study must be performed annually by all analysts using this procedure.

19.1.2.2 The CDC study may be completed by repeating the IDC, by processing four consecutive LCS results obtained from routine sample analysis, by using the last four results from the annual MDL study or by successfully analyzing a performance testing study result.

19.1.2.3 Input CDC data to the same demonstration of capability spreadsheet and submit to the Quality Assurance Department for training documentation.

19.2 A Method Detection Limit (MDL) Study is required annually in accordance with TriMatrix SOP GR-10-125.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are located on the laboratory intranet library.

21.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

21.3 Follow all instructions outlined in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Methods of Soil Analysis Part 2, Chemical and Microbiological Properties, 2nd Edition,* Number 9 (Part 2), Agronomy, 1982, Section 29-3.5.2, "Walkley-Black Procedure"

23.0 ATTACHMENTS

23.1 Stock Standards Logbook Example

23.2 Raw Data Example

23.3 Calculation Spreadsheet Example

23.4 Preparation Batch Report Example

Attachment 23.1
Stock Standards Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used/ Lot #	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By	Date Verified
1	IN5.18 -1	80H	IN5.17.17			5ppm	20ml	H_2O	200ml	0.5ppm	JMF	1/17/02	2/8/02		
2	IN5.18 -2	↓	IN5.17.17			5ppm	10ml	H_2O	↓	0.25ppm	↓	↓	↓		
3	IN5.18 -3	↓	IN5.17.18			1ppm	20ml	↓	↓	0.1	↓	↓	↓		
4	IN5.18 -4	↓	IN5.18.2			0.25ppm	20ml	↓	↓	0.025	↓	↓	↓		
5	IN5.18 -5	↓	IN5.18.3			0.1ppm	20ml	↓	↓	0.01	↓	↓	↓		
6	IN5.18 -6	80H	IN5.18.1			0.5ppm	2ml	H_2O	200ml	0.005ppm	JMF	1/17/02	2/8/02		
7	IN5.18 -7	TOC		Baker Lot#613112		Potassium Biphthalate	2.128g	H_2O	100mlo	10,000ppm	NLB	1/30/02	1/30/02		
8	IN5.18 -8	Sulfide	2206							810ppm	NLB	1/28/02	1/28/02		
9	IN5.18 -9	Sulfide (LCS)	2206							1580ppm	NLB	1/28/02	1/28/02		
10	IN5.18 -10	TOC	IN5.17.10			10ppm	5ml	H_2O	100ml	0.5ppm	JMF	1/24/02	1/28/02		
11	IN5.18 -11	Fluoride (LCS)	206C			Orychem.	0.5526g	H_2O	250ml	1000ppm	SAJ	1/28/02	2/28/02		
12	IN5.18 -12	Chloride (LCS)	264 J			Orychem.	0.4121g	H_2O	250ml	1000ppm	SAJ	1/28/02	2/28/02		
13	IN5.18 -13	Sulfate (LCS)	1690			Orychem.	0.4535g	H_2O	250ml	1000ppm	SAJ	1/28/02	2/28/02		
14	IN5.18 -14	Fluoride (LCS)	IN5.18-11			100ppm	10ml	H_2O	100ml	10ppm	SAJ	1/28/02	2/4/02		
15	IN5.18 -15	80H	IN5.3.5			100ppm	25ml	H_2O	100ml	25ppm	JMF	1/28/02	2/11/02		
16	IN5.18 -16	↓	↓			↓	10ml	↓	↓	10ppm	↓	↓	↓		
17	IN5.18 -17	↓	↓			↓	5ml	↓	↓	5ppm	↓	↓	↓		
18	IN5.18 -18	80H	IN5.3.5			100ppm	2ml	H_2O	100ml	2ppm	JMF	1/28/02	2/11/02		

file: standardlogs.xlsx page: 18 of 100 revision: 1.1

Approved By: 2-9-09 QA Officer

Approved By: Heather L. Brady Area Supervisor

Attachment 23.2
Raw Data Example

TriMatrix Laboratories, Inc.

Walkley-Black Total Organic Carbon in Soil

Analyst: NLB
Date/Time/Temperature Oven Drying Began: 11/21/08 19:00 103°C
Date/Time/Temperature Oven Drying Ended: 11/22/08 10:15 104°C
Balance Used: 205

Date Batch Opened: 11-26-08
Supervisor: JG
Instrument Number: 176

mL Titrant Used: 20.3
Ferrous Sulfate Normality =

$$\text{Normality } Fe^{2+} = \frac{10 \text{ mL } Cr_2O_7^{2-}}{x \text{ mL } Fe^{2+}} \times 1N\ Cr_2O_7^{2-}$$

Sample Number	Dish (g)	g of Soil + Dish Before Drying	g of Soil + Dish After Drying	Calculated Percent Solids	g of Oven Dried Sample Used	mL of Titrant Used	Reported Concentration (%)
0811328-01	11.37	66.69	60.10		1.00	17.00	
-07	11.57	78.97	64.81		0.74	17.40	
-11	11.35	103.45	84.20		0.99	17.90	
0811331-05	11.38	60.54	49.15		0.36	17.55	
-06	11.35	59.02	53.62		0.85	15.70	
-07	11.15	55.39	50.96		0.63	16.50	
0811331-05dup	11.38	60.54	49.15		0.36	17.60	
BLK					10	20.25	
BS					1	14.10	

Reagent and Standard Numbers

Ferroin Indicator: INR1.50-13
Potassium Dichromate: INR5.61-14
Ferrous Sulfate: INR9.18-2
H_2SO_4 Lot #: 085201
Spike Standard: IN24.69-15

file: TOC Soil_walkley-black page: 1 of 1 revision 09/13/07

Approved By: 2-9-09 QA Officer
Approved By: Heather L. Brady Area Supervisor

Attachment 23.3
Calculation Spreadsheet Example

TriMatrix Laboratories, Inc.

Walkley-Black Total Organic Carbon in Soil

Analyst: _____ Date Batch Opened: _____
Date/Time/Temperature Oven Drying Began: _____ Supervisor: _____
Date/Time/Temperature Oven Drying Ended: _____ Instrument Number: _____
Balance Used: _____
mL Titrant Used: _____

$$\text{Normality } Fe^{2+} = \frac{10 \text{ mL } Cr_2O_7^{2-}}{x \text{ mL } Fe^{2+}} \times 1N\ Cr_2O_7^{2-}$$

Ferrous Sulfate Normality = _____

Sample Number	Dish (g)	g of Soil + Dish Before Drying	g of Soil + Dish After Drying	Calculated Percent Solids	g of Oven Dried Sample Used	mL of Titrant Used	Reported Concentration (%)

Reagent and Standard Numbers

Ferroin Indicator: _____ Potassium Dichromate: _____
Ferrous Sulfate: _____ H_2SO_4 Lot #: _____ Spike Standard: _____

file: TOC Soil_walkley-black.xls page: 1 of 1 revision 09/13/07

Approved By: 2-9-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.4
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0606877 Page 1 of 1

LVY

Printed: 6/17/2006 2:27:45PM

Inorganic - Wet Chemistry, Soil, Method-Specific Preparation

Due 6-19-06

(No Surrogate)

Batch Comments: (none)

DLG 6-19-06

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0606082	TOC MSA 29-3.5.2				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0606877-BLK1		Jun-16-06 08:00	HLB	10	10					BLANK	
0606877-DUP1		Jun-16-06 08:00	HLB	0.98	0.98		0606082-02			DUPLICATE	
0606877-BS1		Jun-16-06 08:00	HLB	0.5	0.5			A602089	500	LCS	
0606877-MS1		Jun-16-06 08:00	HLB	0.97	0.97		0606082-02	A602089	500	MATRIX SPIKE	
0606082-01	A	Jun-16-06 08:00	HLB	1.01	1.01					[redacted]	
0606082-02	A	Jun-16-06 08:00	HLB	0.97	0.97					[redacted]	
0606082-03	A	Jun-16-06 08:00	HLB	0.98	0.98					[redacted]	
0606082-04	A	Jun-16-06 08:00	HLB	1.13	1.13					[redacted]	
0606082-05	A	Jun-16-06 08:00	HLB	0.3	0.3					[redacted]	
0606082-06	A	Jun-16-06 08:00	HLB	0.86	0.86					[redacted]	
0606082-07	A	Jun-16-06 08:00	HLB	0.91	0.91					[redacted]	
0606082-08	A	Jun-16-06 08:00	HLB	1.14	1.14					[redacted]	
0606082-09	A	Jun-16-06 08:00	HLB	0.34	0.34					[redacted]	
0606082-10	A	Jun-16-06 08:00	HLB	0.87	0.87					[redacted]	
0606082-11	A	Jun-16-06 08:00	HLB	0.47	0.47					[redacted]	
0606082-12	A	Jun-16-06 08:00	HLB	0.38	0.38					[redacted]	
0606082-13	A	Jun-16-06 08:00	HLB	1.04	1.04					[redacted]	
0606082-14	A	Jun-16-06 08:00	HLB	0.85	0.85					[redacted]	
0606082-15	A	Jun-16-06 08:00	HLB	0.74	0.74					[redacted]	
0606082-16	A	Jun-16-06 08:00	HLB	0.85	0.85					[redacted]	
0606082-17	A	Jun-16-06 08:00	HLB	0.74	0.74					[redacted]	
0606082-18	A	Jun-16-06 08:00	HLB	0.68	0.68					[redacted]	
0606082-19	A	Jun-16-06 08:00	HLB	0.99	0.99					[redacted]	
0606082-20	A	Jun-16-06 08:00	HLB	1.1	1.1					[redacted]	

Comments:

Analyst Initials:

bch_TriMatrix.rpt

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

TITRIMETRIC PROCEDURE FOR ACID-SOLUBLE AND REACTIVE SULFIDES

SW-846 Method 9034 and Chapter 7, Section 7.7

SOP has undergone an annual review. No revisions were required.
Date Reviewed: 9-12-06
Reviewed By: JNB
Review Expires: 9-12-07

APPROVALS:

Inorganic Supervisor: Jodi L. Blouw — Date: 11/5/01
Jodi L. Blouw

QA Manager: Rick D. Wilburn — Date: 11/5/01
Rick D. Wilburn

Laboratory Manager: Douglas E. Kriscunas — Date: 11/5/01
Douglas E. Kriscunas

Procedure Number: GR-06-108
Revision Number: 2.0

Date Initiated: 9/1/95
Effective Date: 11/7/01

Date Revised: 11/5/01
Pages Revised: All

By: Heather Brady

Total Number of Pages: 10

1.0 SCOPE AND APPLICATION

1.1 This procedure is used as a determinative step for the quantitation of acid-soluble sulfides following distillation of the sample by SW-846 Method 9030. It is applicable to aqueous, solid waste, or effluent samples.

1.2 The sensitivity of the method is based on the reaction of an iodine solution with the zinc sulfide contained in the sample distillate. The sulfide is oxidized to sulfur using a known excess amount of the iodine solution. The excess amount of iodine is then determined by titration with phenyl arsine oxide or sodium thiosulfate. Knowing that each 1 mL of iodine solution oxidizes 0.4 mg of the zinc sulfide to sulfur, and based on the ability to read a titrant volume change of 0.25 mL, it is possible to quantitate down to 0.1 mg of sulfide. As 100 mL of sample is used in the distillation, a minimum aqueous reporting limit of 1 mg/L is possible. The minimum reporting limit achievable for soil samples is 10 mg/kg, based on the distillation of a 10 g sample.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 0, December 1996, Method 9034, "Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides".

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 3, December 1996, Chapter 7, Section 7.7, "Interim Guidance for Reactive Sulfide".

3.0 SUMMARY OF PROCEDURE

3.1 Method 9034 oxidizes the zinc sulfide obtained from the method 9030B acid-soluble sulfide distillation to sulfur, using a known excess amount of iodine. Due to oxidative degradation, the use of sulfide standards for quantitation is not possible; therefore, the sulfide quantitation is based on titrating the excess iodine with a standard solution of phenylarsine oxide (PAO) or sodium thiosulfate, until the blue iodine starch complex disappears.

4.0 PARAMETER OR COMPOUND LIST

4.1 Sulfide, acid-soluble

5.0 REFERENCED SOPs

5.1 TriMatrix SOP number GR-06-103, *Distillation Procedure for Acid-Soluble Sulfides*, most current version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 The determinative method for acid-soluble sulfides suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and hydrosulfite. These reduced sulfur compounds decompose in

acid, and may form sulfur dioxide. This gas may be carried over to the zinc acetate gas scrubbing bottles used during the pre-treatment process. The sulfur dioxide will subsequently react with the iodine solution used during the titration, which will yield false high values. The addition of formaldehyde into the zinc acetate gas scrubbing bottles will remove this interference. Sulfur also interferes because it may be reduced to sulfide by tin (II) chloride in this procedure. Various organic chemicals can also interfere with the titration.

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all Standard Operating Procedures for Health and Safety as outlined in the TriMatrix Laboratories, Inc. Safety Manual.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 All aqueous samples are to be preserved using zinc acetate. Add four drops of 2N zinc acetate per 100 mL of container volume. In an amber glass container, collect enough sample to completely fill the container with no headspace. Cap and gently mix the sample. Slowly pour out a small amount of the sample. Adjust the pH of the sample in the container to greater than 9 using 6N NaOH. With a minimum of aeration carefully add more sample until the bottle is completely filled, with no headspace.

8.2 It is recommended that solid samples have the surface of the sample filled with 2N zinc acetate until moistened.

8.3 Store all samples with no headspace, cooled to 4° C.

8.4 All distillations must be performed within 7 days of the sampling date.

8.5 Distillates that are not analyzed immediately must be stored in a sealed flask at 4° C.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 500 mL flasks

9.2 Hot plate stirrer

9.3 10 and 25 mL burettes

9.4 Volumetric pipets

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Not applicable

11.0 CHEMICALS AND REAGENTS

11.1 Reagent water

11.2 Starch solution – Use either an aqueous solution or soluble starch powder mixtures. The aqueous solution is prepared by dissolving 2 g soluble starch and 2 g salicylic acid ($C_7H_6O_3$) as a preservative, in 100 mL hot reagent water.

11.3 Hydrochloric acid (6N), HCl. Place 51 mL of reagent water in a 100 mL Class A volumetric flask. Slowly add concentrated HCl to bring the total volume to 100 mL.

11.4 Standard phenylarsine oxide solution (PAO) (0.025N), C_6H_5AsO, purchased commercially prepared. Caution: PAO is toxic.

11.5 Standard sodium thiosulfate solution (0.025N), $Na_2S_2O_3 \bullet 5H_2O$. Dissolve 6.205 ± 0.005g $Na_2S_2O_3 \bullet 5H_2O$ in 500 mL reagent water. Add 9 mL 1N NaOH and dilute to 1 liter.

11.6 Iodine solution (approximately 0.025N) – Dissolve 20 – 25 g of potassium iodine, KI, in 150 mL of reagent water in a 1-liter volumetric flask. Add 3.2 g iodine, I_2. Allow the chemicals to dissolve and dilute to 1 liter with reagent water. The solution must now be standardized to calculate the exact normality. This is accomplished using either the phenylarsine oxide solution (PAO), or sodium thiosulfate. The below standardization procedure must be performed on this solution daily.

11.6.1 Standardization

11.6.1.1 Dissolve approximately 2 g of KI in 150 mL of reagent water. Add exactly 20 mL of the iodine solution made above and dilute to 300 mL with reagent water.

11.6.1.2 Titrate the iodine solution with the 0.025N PAO solution, or the sodium thiosulfate solution until the amber color fades to yellow. Add a few drops of the starch indicator solution. Continue titration drop by drop until the blue color disappears.

11.6.2 Calculate the normality of the iodine solution using the following equation:

$$\text{Normality } (I_2) = \frac{\text{mL of Titrant x Normality of Titrant}}{\text{Amount of Iodine in mL}}$$

11.7 All reagents must be logged into the Reagent Logbook (Attachment 23.1). Unless otherwise indicated, all reagents have an expiration date of 1 year from the date of preparation.

12.0 STANDARDS PREPARATION

12.1 No standards are used in the analytical portion of this method. Standards used in the method and matrix spikes are discussed in the pre-treatment SOP, TriMatrix SOP number GR-06-103.

13.0 SAMPLE PREPARATION

13.1 Samples are pre-treated following method 9030B, SOP #GR-06-103.

14.0 CALIBRATION PROCEDURES

14.1 Not applicable

15.0 ANALYTICAL PROCEDURE

15.1 The following iodometric titration procedure is used to quantify the sulfide concentration in the distillate generated by Method 9030.

15.2 Pipet a known amount of standardized 0.025N iodine solution (section 11.6) into a 500 mL flask. The volume of standardized iodine solution required is approximately 65 mL for each 50 mg of sulfide. Pipet an amount in excess of what you think will be required. Add enough reagent water to bring the volume to 100 mL.

15.2.1 Acid-Soluble Sulfide

15.2.1.1 If the iodine solution is to be used for the analysis of acid-soluble sulfides, add 2 mL of 6N HCl. If the iodine solution is to be used for the analysis of reactive sulfides, do not perform this step.

15.2.2 Reactive Sulfide

15.2.2.1 The distillate from the pre-treatment method 9030 must be brought to a pH of 2 when the analysis is for reactive sulfides. Take a small amount of the distillate and using 6N HCl, titrate to a pH of 2.

15.2.2.2 Based on the volume titrated, calculate the amount of acid needed to bring the remainder of the sample distillate to a pH of 2.

15.2.2.3 Add the determined amount of acid to the remaining distillate, then re-combine the small acidified distillate portion with the remainder of the now acidified distillate.

15.3 Using a pipet, transfer the distillate obtained in Method 9030 (acidified according to section 15.2.2 if analyzing for reactive sulfide) into the 500 mL flask, keeping the end of the pipet used for transferring the distillate below the surface of the iodine solution.

15.4 Prepare a rinse solution containing a known amount of the standardized iodine solution, 1 mL of 6N HCl, and reagent water. Using the same pipet as above, transfer this solution into the gas scrubbing bottle that contained the distillate. Swirl to rinse any remaining white precipitate (zinc sulfide) from the sides of the bottle. There should be no visible traces of precipitate left after rinsing. Transfer the rinsate into the 500 mL flask. If there is any solution left in the impinger tube, transfer it to the flask.

15.5 Using reagent water, rinse any remaining traces of iodine from the gas scrubbing bottles into the 500 mL flask.

15.6 If at any point in transferring the distillate or rinsate into the 500 mL flask the amber color of the iodine disappears or fades to yellow, an additional, known amount of the standardized iodine solution must be added.

15.7 Total the amount of standardized iodine solution used (initially, during the rinse step, and any supplemental necessary due to faded, yellow, or loss of color).

15.8 Using the 25 mL burette, titrate the solution in the flask with the 0.025N phenylarsine oxide or 0.025N sodium thiosulfate solution until the amber color fades to yellow. Add enough starch indicator for the solution to turn dark blue (usually 2-5 drops) and continue the titration until the blue disappears. If the contents of the 25 mL burette are consumed, continue titrating with the 10 mL burette. Record the volume of titrant used to the nearest 0.25 mL.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Calculate the concentration of sulfide using the following equation:

$$\frac{(\text{mL } I_2 \text{ x N } I_2) - (\text{mL Titrant x N Titrant}) \text{ x } (16000)}{\text{Sample Weight (g) or Sample Volume (mL) Originally Distilled}} = \text{Sulfide (mg / kg or mg / L)}$$

17.0 DATA REPORTING AND DELIVERABLES

17.1 The following data must be included on every benchsheet (Attachment 23.2):

- Owner, date, area manager
- Reagent numbers
- Spike Standard numbers
- Analyst, run date, sample volume, mL iodine, mL PAO, normality of PAO and iodine solutions, reported concentration

17.2 All logbooks must be filled in completely and correctly. All corrections are to be made in indelible ink. **Corrections are to be made with a single lineout, which is then dated and initialed. Write-overs are not acceptable.** The erroneous result must remain legible. The new result is placed near the incorrect result. Blank lines in the logbook should be Z'd out.

17.3 If internal chain of custody is required it is very important that the COC sheet is filled out correctly.

18.0 QUALITY ASSURANCE

18.1 Method QC and matrix QC must be analyzed with each batch.

18.2 Method QC consists of the Laboratory Fortified Blank (LFB), and the Method Preparation Blank (MPB).

18.2.1 The LFB and MPB are prepared as instructed in the method 9030 pre-treatment SOP, TriMatrix SOP number GR-06-103, and analyzed as instructed above. The LFB acceptance limits are the laboratory control limits listed on the benchsheet. The MPB result must be less than the reporting limit listed on the benchsheet.

18.3 Matrix QC consists of spike (SPK) and a duplicate (DUP).

18.3.1 The SPK is a sample aliquot spiked with a known amount of analyte, distilled and analyzed as a sample. See the pretreated method (Method 9030) for preparation and distillation of the spikes. The acceptance laboratory limits for the SPK are listed on the benchsheets.

18.3.2 The DUP is a second aliquot of sample analyzed as a sample. See the pretreated method (Method 9030) for preparation and distillation of the DUP. The acceptance limits for the percent difference for the DUP are the laboratory control limits listed on the benchsheet.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare four LFBs as described in pre-treatment SOP number GR-06-103. Analyze the four samples following this SOP. Calculate the average percent recovery and the standard deviation of the 4 recoveries. The average percent recovery must be within the LFB acceptability windows listed on the benchsheet. The standard deviation of the 4 recoveries must be ≤20%.

19.1.1.2 If either criteria are not met, locate and correct the source of the problem and repeat the test. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validations spreadsheet and raw data should be given to the Quality Assurance Manager.

19.1.2 Continuing Demonstration of Capability

19.1.2.1 Annually a Continuing Demonstration of Capability (CDC) is required by each analyst. The CDC can be accomplished by repeating the IDC or processing four consecutive LFBs as an IDC. Process these samples using the same spreadsheet

used for the IDC study. Alternatively, the successful completion of a Performance Testing sample from that year may be used for a CDC.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable.

20.4 Comply with all environmental laws associated with the disposal of chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 0, December 1996, Method 9034, "Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides".

22.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 3, December 1996, Chapter 7, Section 7.7, "Interim Guidance for Reactive Sulfide".

22.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 9030B, "Acid-Soluble and Acid-Insoluble Sulfides: Distillation".

23.0 ATTACHMENTS

23.1 Reagent Logbook Example

23.2 Benchsheet Examples

Attachment 23.1
Reagent Logbook Example

TriMatrix Laboratories, Inc.

Reagent Preparation Logbook

Reagent Name: Iodine Titrant (Sulfide) **Stock Reagent Number:** ***INR1.61***
Prep Instructions: 20.25g KI + 3.3g Iodine / 1L DI H_2O
Concentrations:

Working Reagent Number	Date Made	Expiration Date	Made By	Reagent(s)/Lot Number(s) Used	Solvent(s)/Lot Number(s) Used
INR1.61-1	4/5/00	4/5/01	CEM	KI 10E6C, Iodine 126398, 292A (0E406MF)	
INR1.61-2	4/20/00	4/20/01	CEM	↓	
INR1.61-3	5/12/00	5/12/01	CEM	126398/126398	
INR1.61-4	5/30/00	5/30/01	CEM	↓	
INR1.61-5	7/7/00	7/7/01	CEM	↓	
INR1.61-6	7/14/00	7/14/01	CEM	↓	
INR1.61-7	8/9/01	8/9/02	HLB	↓	
INR1.61-8	10/10/01	10/10/02	HLB	↓	
INR1.61-9	10/10/01	10/10/02	HLB	↓	
INR1.61-10					
INR1.61-11					
INR1.61-12					
INR1.61-13					
INR1.61-14					
INR1.61-15					
INR1.61-16					
INR1.61-17					
INR1.61-18					

file: intreagent page: 61 of 100 revision: 0.0

Approved By: 11/5/01
QA Manager

Approved By: 11/5/01
Area Manager

Attachment 23.2
Benchsheet Examples

06-AUG-01 INORGANIC BENCHSHEET -- SULFIDE / SULFIDE REACTIVITY PAGE 1

Test #: 412. 1- 312.03 Instrument #: 176 Est anal hrs: 1 Benchsheet ID: 272638
Parameter: SULFIDE Act anal hrs: Owner: NLB 8-7-01
Method: H2S/ACID/UTR ODL: 1.0 Samples in batch: 1
Ref. cit.: USEPA-9034 Unit: mg/L Stock std #: Supervisor: JLB

Batch: 175101 Sand
Comments: 8/14/01
9.8/10 [illegible] 8/14
PAO(mls)/Iodine(mls) * 0.025 = Normality of Iodine Solution = 0.0245
Normality of PAO Titrant = 0.025

Client Submittal Location	Sample	COC	QC	Anal	Run Date	gm Sample	ml of Iodine	ml of PAO	% Solid	Rep. Conc. (mg/kg)	ODL	QH
29543- 123	284621 *		LV1			100	10.	9.6		<1.0		
Injection												

04-OCT-01 INORGANIC BENCHSHEET -- SULFIDE / SULFIDE REACTIVITY ** QC ** PAGE 1

Test #: 412. 1- 287.02 Benchsheet ID: 276570
Parameter: SULFIDE Owner: NLB
Method: H2S/TITRI/WW ODL: 1.0 Date: 10-5-01
Ref. cit.: USEPA-376.1 Unit: mg/L Supervisor: JLB

Comments:

	Anl.	Run Date	mls/gm Sample	ml of Iodine	ml of Titrant	% Solids	Rep. Conc. (mg/kg)	% Solids	Spike Qty	% rec/dif	ODL	QH
BLK:			100	10.	9.8		<1.0		XXXXXXX XXXXXXX	0		
LCS: Stk IN4.46.18			100	10.	5.0		19.2		19.6	98		
SPK1: Stk IN4.43.14 Smp 289485			100	10.	6.8		12.0		12.2	98		
SPK2: Stk Smp												
MSD: Smp		XXXXXXXXXX XXXXXXXXXX	XXXXXXX XXXXXXX	XXXXXX XXXXXX	XXXXXXX XXXXXXX	XXXXXXXX XXXXXXXX	XXXXXXXXXXX XXXXXXXXXXX	XXXXXXXX XXXXXXXX	XXXXXXX XXXXXXX			
DUP: Smp 289485			100	10.	9.8		<1.0		XXXXXXX XXXXXXX	0		
SPK1: Stk Smp												
SPK2: Stk Smp												
MSD: Smp		XXXXXXXXXX XXXXXXXXXX	XXXXXXX XXXXXXX	XXXXXX XXXXXX	XXXXXXX XXXXXXX	XXXXXXXX XXXXXXXX	XXXXXXXXXXX XXXXXXXXXXX	XXXXXXXX XXXXXXXX	XXXXXXX XXXXXXX			
DUP: Smp									XXXXXXX XXXXXXX XXXXXXX XXXXXXX			
MPB: QCB#												
LFB: Stk QCB#												
CRDL: Stk												

QC Type	Range	Lower	Upper
DUP	1		20
DUP	2		20
LCS (wtr)	1	97	103
LFB (wtr)	1	80	120
MSD	1		20
MSD	2		20
SPK	1	77	107

LCS
mls PAO → 4.9
mls Iz → 10
1960ppm
IN4.46.18

10-5-01 NLB
ACSPK
mls PAO → 8.7
mls Iz → 12
1224ppm
IN4.43.14

TRIMATRIX LABORATORIES, INC.

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Potentiometric pH

SW-846 9040C
Standard Methods 4500-H^+ B

APPROVALS:

Area Supervisor: Heather L. Brady Date: 5-14-09
Heather L. Brady

QA Officer: Date: 5-14-09
Tom C. Boocher

Operations Manager: Date: 5/14/09
Jeff P. Glaser

Procedure Number: GR-07-100
Revision Number: 3.4

Date Initiated: 10/5/95
Effective Date: 5/25/09

Date Revised: 5/14/09
Pages Revised: All

By: Tom C. Boocher
Total Number of Pages: 15

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to drinking water, ground water, wastewater and wastes with greater than a 20% water phase by volume.

1.2 The effective measurement range is 2.0 to 12.0 pH units.

1.3 The minimum reporting limit is 2.0 pH units.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, November, 2004, Method 9040C, "pH Electrometric Measurement"

2.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 4500-H^+ B, "pH Value, Electrometric Method"

3.0 SUMMARY OF PROCEDURE

3.1 Sample pH is measured potentiometrically using a buffer-calibrated combination reference/pH electrode and pH meter.

4.0 PARAMETER OR COMPOUND LIST

4.1 Potentiometric pH

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-05-102, *Waste Disposal,* latest revision

5.2 TriMatrix SOP GR-07-113, *pH Potentiometric Method Soil and Waste (Non-Aqueous Liquids),* latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-10-114, *Thermometer Calibration and Verification*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 High sodium samples with very low or high pH will give incorrect meter readings. For pH results greater than 10.0, pH will be biased low unless using a "low sodium error" electrode. For pH less than 2.0,

measurements will be biased high and must be reported as "pH <2.0". The electrode used has very little sodium error at low-sodium concentrations and this is not a factor with most environmental samples analyzed. However, report pH results greater than 12.0 as "pH >12.0".

6.2 Temperature variation between calibration buffers and samples is a source of measurement error. Buffers and samples must be measured at the same temperature.

Note: The temperature sensor of the meter must be verified as accurate, at least annually, in accordance with TriMatrix SOP GR-10-114.

6.3 Soaps, oily matter, suspended solids or precipitates may coat the glass pH electrode and cause a sluggish response. Clean the electrode as follows:

6.3.1 For general cleaning, soak the pH electrode in 0.1M hydrochloric acid (HCl) or 0.1 nitric acid (HNO_3) for 15 minutes. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.2 For grease and oil deposits, rinse with a mild detergent or methanol solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.3 For inorganic deposits, rinse with 0.1M tetrasodium EDTA solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.4 For protein deposits, digest with 1% pepsin in 0.1M HCl. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all health and safety instructions as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used in this procedure has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as possible.

7.3 A laboratory coat, disposable gloves and safety glasses are required when working in the wet chemistry laboratory and when handling chemicals.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Collect samples in a plastic or glass bottle large enough to hold at least 100 mL. Fill the container to capacity when bringing to the laboratory for analysis.

8.2 Do not add preservative.

8.3 Analyze as soon as possible after receipt by the laboratory.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Potentiometric pH meter.

9.2 Sample cups, disposable, 50 mL

9.3 Magnetic stir plate, not heated

9.4 Stir bars, PTFE-coated

9.5 pH electrode, glass, with sodium error of ±0.01 pH units at pH 12.0 in 0.5N sodium chloride solution

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.2 The pH electrode must be inspected frequently and refilled with fluid if necessary.

10.1 When necessary, clean the pH electrode in accordance with Section 6.3.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q system

11.2 Calibration buffers, commercially purchased

11.3 Quality control buffers, commercially purchased

11.4 Hydrochloric acid, reagent grade or better

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Samples must be brought to ambient temperature before analysis. If sample temperature differs from calibration buffers by more than ±2° C, a temperature correction must be made (manually or automatically).

13.2 Further sample preparation is not required if samples have a greater than 20% aqueous phase. Analysis of pH samples with less than 20% (v/v) aqueous phase is beyond the scope of this procedure. Such samples need re-logged in for analysis by TriMatrix SOP GR-07-113 by the project chemist.

13.3 The aqueous phase in a sample can be determined by visually observing the sample jar and estimating whether approximately 20% (v/v) is a water phase by volume. If there is obviously at least 20% (v/v) free water in the sample then determine pH on the water phase only. If there is obviously less than 20% (v/v) water or no water then proceed in accordance with Section 13.2. For less ambiguity, determine the water phase volume before shaking the sample.

14.0 CALIBRATION PROCEDURES

14.1 Calibrate the pH meter to bracket expected pH values with three buffers, three pH units or more apart. Refer to Attachment 23.5 for a pH calibration flowchart.

Note: When field sample pH is between 10.0 and 12.0, control the temperature to 25 ±1° C.

14.2 Use only fresh buffer solution for the calibration. Do not save to use on another meter. Discard immediately after the calibration is complete.

14.3 To calibrate the pH meter for normal-range samples, use buffers 4.0, 7.0 and 10.0.

14.3.1 If a sample reads less than pH 4.0, recalibrate using buffers 2.0, 4.0, and 7.0 then reanalyze.

14.3.2 If a sample reads greater than pH 10.0, recalibrate using buffers 7.0, 10.0 and 12.0 then reanalyze.

Note: Always begin the calibration with the pH 7.00 buffer solution.

14.4 When calibrating, wait for a stable reading for each buffer solution.

14.5 During calibration and analysis, gently stir each solution at a constant rate great enough to minimize drift (<0.1 pH unit) but not enough to generate bubbles and/or aeration that might introduce carbon dioxide.

14.6 Record the displayed calibration slope on the benchsheet and in the run logbook. The slope needs to be 92 – 102%. If not, clean the electrode.

14.7 Once the meter is calibrated, read each buffer as the initial calibration verification (CCV). Each must read correct to within ±0.05 pH units. If not, repeat the calibration until all buffers read within ±0.05 pH units.

14.8 After calibrating and verifying the calibration, the meter is ready for sample analysis.

15.0 ANALYTICAL PROCEDURE

15.1 For all analyses, adjust the probe clamp so immersion will be just deep enough into the solution to establish good electrical contact through the electrode fiber-capillary hole. Rinse probes thoroughly with reagent water before transferring to another solution. Discard the rinseate.

Note: Standard and sample temperatures must be within ±2° C of the calibration buffer temperature. Also, do not let samples sit open on the bench for any length of time or over-stir when taking a reading as exposure to the air and/or excessive stirring will introduce carbon dioxide and lower the pH.

15.2 If not already done, verify the calibration by checking each calibration buffer. Record these checks as CCV_1, CCV_2 and CCV_3. Gently stir at the same rate used during calibration. Wait for the meter to beep and display "stable" then record the pH to the second decimal place. Each value obtained must be within ±0.05 pH units of the certified value for acceptability. If acceptable, the meter is ready for sample analysis. Remember to rinse the probe thoroughly with reagent water between solutions.

15.3 Repeat with second-source verification (SRM_1, SRM_2) buffers as specified in Attachment 23.5. These readings must also be within ±0.05 pH units of the certified value for acceptability. Sample analysis may not begin until CCV and SRM results are acceptable.

15.4 After acceptable CCV and SRM results are obtained, measure the pH of up to 10 samples (this count includes the SRM). If necessary, repeat sample analysis on multiple volumes of sample until the difference in readings is less than 0.1 pH units. Two or three volumes are usually sufficient. Report the value obtained.

15.5 After 10 analyses and at the end of each batch, analyze a continuing calibration verification (CCV) as specified in Attachment 23.5. The CCV will be the same three buffers used to calibration the instrument. Measured results must be within ±0.05 pH units of the certified value for acceptability.

15.6 Provided the three CCV buffers pass, sample analysis may continue with the three CCV buffers being analyzed after every 10 analyses. Also, close the analytical batch with an acceptable CCV check of the three buffers.

15.7 If a CCV fails, the previous 10 samples must be reanalyzed after cleaning the electrode in accordance with Section 6.3, and recalibrating.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The pH meter measures all data against the calibration and displays a direct pH reading. No manual calculations are necessary.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts are responsible for data quality and for correctly filling in all documentation. This is required for quality control and to provide clients with fully defensible data.

17.2 Record the following data and all other data specified throughout the procedure on the benchsheet and/or in the logbook for each batch handed in:

17.2.1 The laboratory information management system (LIMS/Element™) standard identification of all calibration buffers used (logbook)

17.2.2 Sample temperature (benchsheet)

17.2.3 The pH result (benchsheet)

17.2.4 Observed values of each buffer used for calibration (benchsheet)

17.2.5 The slope (benchsheet)

17.2.6 Method name and number (benchsheet)

17.2.7 Refer to Attachment 23.1 for a benchsheet example.

17.3 Instrument logbooks must be filled in with the following information.

17.3.1 Date analyzed

17.3.2 Analyst's initials

17.3.3 Calibration standards used

17.3.4 Client name and sample numbers analyzed

Note: Refer to Attachment 23.3 for an instrument logbook example.

17.4 All logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single dated/initialed lineout. Write-overs are not acceptable.** An incorrectly entered result must remain legible even after the correction is made. A new result must be placed near the incorrect result with the date and analyst's initials written next to it. Blank lines in logbooks must be Z'd out.

17.5 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in correctly and completely.

18.0 QUALITY ASSURANCE

18.1 Method and matrix quality control must be analyzed with each batch of up to 10 samples.

18.1.1 Quality control consists of two secondary calibration verification (SRM) buffers, an initial calibration verification (as the three CCV buffers) and continuing calibration verifications (the same three CCV buffers) as specified in Attachment 23.5. Acceptance limits are ±0.05 pH units of the certified value.

18.1.1.1 Acceptance limits are ±0.05 pH units of the certified value.

18.1.1.2 Include quality control samples in the frequency count.

pH	Method ±limit	Lower Control Limit as pH (LCL)	Upper Control Limit as pH (UCL)	LIMS LCL (%)	LIMS UCL (%)
2	0.05	1.95	2.05	97.5	102.5
4	0.05	3.95	4.05	98.8	101.3
6	0.05	5.95	6.05	99.2	100.8
7	0.05	6.95	7.05	99.3	100.7
8	0.05	7.95	8.05	99.4	100.6
10	0.05	9.95	10.05	99.5	100.5
12	0.05	11.95	12.05	99.6	100.4

18.1.2 Matrix quality control consists of a sample duplicate (DUP) every 10 samples. A DUP is an analysis replicate analyzed in the same way as the original. Acceptance limits in relative percent difference (RPD) are listed in the laboratory information management system (Element™).

18.2 Refer to TriMatrix GR-10-106 for the appropriate corrective action needed when an out-of-control condition exists.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability study (IDC). While IDC studies are not instrument dependent, one is required on each pH meter used to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Prepare four aliquots of pH 6.00 or 8.00 buffer. Process the four aliquots following every step in this procedure including calibration. Calculate average percent recovery and standard deviation of the average using the IDC spreadsheet located on the laboratory intranet library. The average value must be within ±0.1 pH units of the certified value. The standard deviation must be ≤ 3%.

19.1.1.2 If these criteria are not met, locate and correct the source of the problem and repeat the study. Repeated failure will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the procedure or inappropriate technique, then repeat the study successfully.

19.1.1.3 Samples may not be analyzed by any analyst on any pH meter until a demonstration of capability study has been successfully completed. Give a copy of successful studies, including the IDC spreadsheet and raw data to the quality assurance department for training documentation and review.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A demonstration of capability study must be performed annually by all analysts. This may be accomplished as follows:

19.1.2.1.1 By repeating the initial demonstration of capability study.

19.1.2.1.2 By processing four consecutive pH 6.00 or 8.00 results (4 of the same pH) exclusively obtained by the analyst during routine sample analysis.

19.1.2.1.3 By exclusively obtaining an acceptable blind performance evaluation study result obtained during routine sample analysis.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate material safety data sheet (MSDS) when disposing of chemicals. Material safety data sheets are maintained on the laboratory intranet library.

21.2 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal.

Approved By: [signature] **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, November, 2004, Method 9040C, "pH Electrometric Measurement"

22.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998,* 4500-H^+ B, "pH Value, Electrometric Method"

23.0 ATTACHMENTS

23.1 Sample Benchsheet Example

23.2 Data Review Report Example

23.3 Instrument Logbook Example

23.4 Standards Log Example

23.5 pH Calibration Flowchart

Attachment 23.1
Sample Benchsheet Example

due 7/31

TriMatrix Laboratories, Inc. **ANALYSIS SEQUENCE** 6071943 Page 1 of 1 Printed: 7/19/2006 2:57:05PM

Inorganic - Wet Chemistry, Water, Jul-19-06

Instrument = 164, Calibration = UNASSIGNED

Sequence Analyses:
pH 4500-H B

Lab Number	*Analysis*	*Contain*	pH ~~STD ID~~	temp. ~~ISTD ID~~	*Client / QC Type*	pH ~~Extraction Comments~~	temp
6071943-ICV1	QC		6.99	22.4	INITIAL CAL CHECK		
0608164-SRM1	QC		6.02	22.6	REFERENCE		
0608164-SRM2	QC		7.41	22.4	REFERENCE		
6071943-CCV1	QC		7.02	22.5	CALIBRATION CHECK		
0607223-01	pH 4500-H B	A	9.34	22.3	Environmental Resource Associates	pH 9.34	22.1
0608164-DUP1	QC 0607223-01		9.34	22.1	DUPLICATE	9.34	21.9
6071943-CCV2	QC		7.00	22.6	CALIBRATION CHECK		

ICV (6.99 ÷ 7.00) (100%) = 99.9%

SRM1 (6.02 ÷ 6.00) (100%) = 100.3%

SRM2 (7.41 ÷ 7.40) (100) = 100.1%

CCV1 (7.02 ÷ 7.00) (100) = 100.3%

CC2 (7.00 ÷ 7.00) (100) = 100%

Comments:	Analyst Initials:

seq_TriMatrix.rpt

Approved By: TD 5/14/09 QA Officer

Approved By: Heather L. Brady Area Supervisor

Attachment 23.2
Data Review Report Example

TriMatrix Laboratories, Inc.

Data Review Report -- Wet Chem Lab

Sequence = 6071943 Page 1 of 1 on 1/28/2008 at 5:14

SampleID	Analysis	IResult	Diln	FResult	FMRL	Qualifier	Recovery	RPD	Analyzed	Analyst	Batch
6071943-ICV1	pH 4500-H B	6.99	1	6.99			100		7/19/2006 2:56	MSM	6071943
0608164-SRM1	pH 4500-H B	6.02	1	6.02	1.0		100		7/19/2006 2:56	MSM	0608164
0608164-SRM2	pH 4500-H B	7.41	1	7.41	1.0		100		7/19/2006 2:56	MSM	0608164
6071943-CCV1	pH 4500-H B	7.02	1	7.02			100		7/19/2006 2:56	MSM	6071943
0607223-01	pH 4500-H B	9.34	1	9.3	1.0	WC010			7/19/2006 2:56	MSM	0608164
0608164-DUP1	pH 4500-H B	9.34	1	9.34	1.0			0	7/19/2006 2:56	MSM	0608164
6071943-CCV2	pH 4500-H B	7	1	7.00			100		7/19/2006 2:56	MSM	6071943

Approved By: 5-14-07 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.3
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Date	Analyst	Buffer Solution pH Concentrations and Lot Numbers (fill in Lot Numbers only for Buffers Used)					Calibration Verification Level and Lot Number		CV Results	Acceptable CV (±0.05 TV)	Client and Sample Numbers
		2	4	7	10	12	TV	Lot Number			
6/17/15	JMD	IN14.79-16	IN14.42-12	IN12.99-10			6.00	IN14.78-4	5.98	yes / no	[redacted]
										yes / no	
6/20/15	JMD		IN14.42-12	IN12.99-10	IN14.42-13		6.00	IN14.78-4	6.00	yes / no	[redacted]
							7.40	IN14.78-3	7.38	yes / no	
6/23/15	JMD	IN14.79-16	IN14.42-12	IN12.99-10			6.00	IN13.78-4	6.02	yes / no	[redacted] 372185, 372051
										yes / no	[redacted] 371854
6/23/15	JMD		↓	↓	IN14.42-13		6.00	IN13.78-4	5.96	yes / no	[redacted] 3506135-05 [redacted] 3506152-06,96 3506178-07,96
							7.40	IN13.78-3	7.36	yes / no	[redacted] 12100-2 [redacted] 372187, 192, 196, 200, 204, 208, 212
6/28/05	AOG		In14.42.12	In12.99.10	In14.42.13		6.00	In13.78.4	6.02	yes / no	05 06 199-01
							7.40	In13.78.3	7.40	yes / no	
6/29/05	AOG	In14.79.16	In14.42.12	In12.99.10			6.00	In13.78.4	6.03	yes / no	[redacted] 392438
							7.40	In13.78.3	7.42	yes / no	
6/30/05	AOG		In14.42.12	In12.99.10	In14.42.13		6.00	In13.78.4	6.04	yes / no	[redacted]
							7.40	In13.78.3	7.40	yes / no	
6/30/05	AOG	In14.79.16	In14.42.12	In12.99.10			6.00	In13.78.4	6.02	yes / no	[redacted]
							7.40	In13.78.3	7.38	yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	

file: phlog1 page: 21 of 50 revision: 1.0

Approved By: 5-14-09 QA Officer Approved By: Heather L. Brady Area Supervisor

Attachment 23.4
Standards Log Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

A512818

Description:	pH buffer 7.00 IN16.50-13	Expires:	Dec-30-06
Standard Type:	Calibration Star	Prepared:	Jan-06-06
Solvent:	Solvent Lot #	Prepared By:	** Vendor **
Final Volume (mls):	20000	Department:	Expired
Vials:	1	Last Edit:	Aug-10-06 10:10 by MSM

Fisher catalog # SB107-20
Lot:054717-24

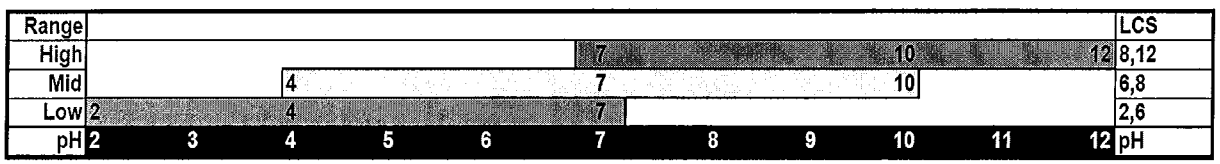
Analyte	CAS Number	Concentration	Units
pH		7	pH Units
Color (Apparent)		7	pH Units
Carbon Dioxide	124-38-9	7	pH Units
Alkalinty, Phenolphthalein		7	pH Units
Alkalinity, Total		7	pH Units
Alkalinity, Hydroxide		7	pH Units
Alkalinity, Carbonate		7	pH Units
Alkalinity, Bicarbonate		7	pH Units
Acidity as CaCO3		7	pH Units

Approved By: 5-14-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.5
pH Calibration Flowchart

TriMatrix Laboratories, Inc.

pH Calibration and Quality Control



1. Run three point curve, ALWAYS starting with 7: Slope of the curve must be between 92 - 102%.
   High: 7, 10, 12
   Mid: 7, 4, 10
   Low: 7, 2, 4

2. Re-run each point of the curve as a sample as ICVs. Response must be ±0.05 pH units of true value.

3. Run the LCSs appropriate to the curve: Response must be ±0.05 pH units of true value. The LCSs are second source buffers.
   High: 8, 12
   Mid: 6, 8
   Low: 2, 6

4. Every 10 samples and at end of batch run CCVs: Response must be ±0.05 pH units of true value. The CCVs are the same buffers as the curve.
   High: 10, 12
   Mid: 4, 10
   Low: 2, 4

Approved By: TD 5-14-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Potentiometric pH in Soil and Waste

SW-846 Method 9045C

APPROVALS:

Area Supervisor: Heather L. Brady — Date: 2-5-09
Heather L Brady

QA Officer: — Date: 2-5-09
Tom C. Boocher

Operations Manager: Jeff P. Glaser — Date: 2/6/09
Jeff P. Glaser

Procedure Number: GR-07-113
Revision Number: 0.3

Date Initiated: 10/15/99
Effective Date: 3/5/09

Date Revised: 2/5/09
Pages Revised: All

By: Heather L. Brady
Total Number of Pages: 15

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to solids, sludges, waste and non-aqueous liquids with less than a 20% (v/v) water phase.

1.2 The effective measurement range is 2.0 to 12.0 pH units.

1.3 The minimum reporting limit is 2.0 pH units.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 9045C, "Soil and Waste pH", Revision 3, January, 1995

3.0 SUMMARY OF PROCEDURE

3.1 Samples are mixed with water and pH of the water phase is measured potentiometrically using a buffer-calibrated combination reference/pH electrode and pH meter.

4.0 PARAMETER OR COMPOUND LIST

4.1 Potentiometric pH

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.2 TriMatrix SOP GR-07-100, *Potentiometric pH*, latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 High sodium samples with very low or high pH will give incorrect meter readings. For pH results greater than 10.0, pH will be biased low unless using a "low sodium error" electrode. For pH less than 2.0, measurements will be biased high and must be reported as "pH <2.0". The electrode used has very little sodium error at low-sodium concentrations and this is not a factor with most of the environmental samples analyzed. However, pH results greater than 12.0 must be reported as "pH >12.0".

6.2 Temperature variation between calibration buffers and samples is a source of measurement error. Buffers and samples must be measured at the same temperature as specified in the procedure.

6.3 Soaps, oily matter, suspended solids, or precipitates may coat the glass pH electrode and cause a sluggish response. The electrode must be kept clean as follows:

6.3.1 For general cleaning, soak the pH electrode in 0.1M hydrochloric acid (HCl) or 0.1 nitric acid (HNO_3) for 15 minutes. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.2 For grease and oil deposits, rinse with a mild detergent of methanol solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.3 For inorganic deposits, rinse with 0.1M tetrasodium EDTA solution. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

6.3.4 For protein deposits, digest with 1% pepsin in 0.1M HCl. Rinse with laboratory reagent water then soak the electrode in pH electrode storage solution for at least 30 minutes before using.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all health and safety instructions as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The toxicity or carcinogenicity of reagents used in this procedure has not been fully established. Each chemical must be regarded as a potential health hazard and exposure must be as low as possible.

7.3 A laboratory coat, disposable gloves and safety glasses are required when working in the inorganics lab and when handling chemicals.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples must be collected in a plastic or glass bottle large enough to hold at least 100 mL.

8.2 No preservative is added or required.

8.3 Samples not analyzed for pH in the field must be analyzed as soon as possible after receipt by the laboratory.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Potentiometric pH meter

9.2 Sample cups, disposable, 50 mL

9.3 Magnetic stir plate, not heated

9.4 Magnetic stir bars, PTFE-coated

9.5 The pH electrode used is Thermo Orion, model 9156APWP, serial 250984-A01, with sodium error of 0.01 pH units at pH 12.0, in 0.5N sodium chloride solution

9.6 Balance, top-loading, capable of measurement to 0.1 g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 The pH electrode must be inspected frequently and refilled with fluid if necessary.

10.2 When necessary, clean the pH electrode in accordance with Section 6.3.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water, ASTM Type II, Milli-Q

11.2 Calibration buffers, commercially purchased

11.3 Quality control buffers, commercially purchased

11.4 Hydrochloric acid, reagent grade or better

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 SAMPLE PREPARATION

13.1 Samples must be brought to ambient temperature before analysis. If sample temperature differs from calibration buffers by more than ±2° C, a temperature correction cannot be made by the instrument.

13.2 Further sample preparation is required if samples have less than 20% (v/v) of aqueous phase. Analysis of pH samples with greater than 20% (v/v) aqueous phase is beyond the scope of this procedure. Such samples need re-logged in for analysis by TriMatrix SOP GR-07-100, by the project chemist.

13.3 The aqueous phase in a sample can be determined by visually observing the sample jar and estimating whether approximately 20% (v/v) is a water phase by volume. If there is obviously at least 20% (v/v) free

water in the sample then determine pH on the water phase only. If there is obviously less than 20% (v/v) water or no water then proceed in accordance with Section 15.0. For less ambiguity, determine the water phase volume before shaking up the sample.

13.4 Prepare samples with less than 20% (v/v) water phase as follows:

13.4.1 Weigh out 20 g of sample into a 50 mL sample cup then add 20 mL of laboratory reagent water. Cover and continuously stir for five minutes without generating bubbles or effervescence. If working with a water-absorbing matrix, use 20 g of sample and 40 mL of laboratory reagent water.

13.4.2 Let the suspension stand for one hour (15 minutes for waste samples) to let suspended particles settle out. If a waste is multiphasic, separate and measure only the aqueous phase. Electrodes must be cleaned (Section 6.3) if coated with nonaqueous material.

13.4.3 After the suspension has settled and the water phase has been isolated (if necessary), check the pH.

Note: All standard and sample temperatures must be within ±2° C of the calibration buffer temperature.

CALIBRATION PROCEDURES

14.1 The pH meter must be calibrated to bracket expected pH values with three buffers, three pH units or more apart. Refer to Attachment 23.5 for a pH calibration flowchart.

Note: When field sample pH is between 10.0 and 12.0, control sample temperature to 25 ±1° C.

14.2 To calibrate the pH meter for normal-range samples, use buffers 4.0, 7.0 and 10.0.

14.2.1 If a sample reads less than pH 4.0, recalibrate using buffers 2.0, 4.0, and 7.0 then reanalyze.

14.2.2 If a sample reads greater than pH 10.0, recalibrate using buffers 7.0, 10.0 and 12.0 then reanalyze.

Note: Always begin the calibration with pH 7.00 buffer solution.

14.3 Start calibration by pressing the mode key until the pH mode indicator is displayed. Place the two probes into the first buffer.

14.4 Press 2nd cal. ("calibrate", time and date of the last calibration will be displayed). After a few seconds P1 will be displayed in the lower field, indicating the meter is ready for the first buffer.

14.5 Wait for a stable reading. Manually enter the value using the scroll keys then press "yes". To enter a value, press the ^ or v key. The first digit will flash. Continue pressing the scroll key until the desired value is

displayed. Press "yes" to accept. Continue for each digit. When the correct buffer value is displayed, press "yes" to enter.

14.6 The P2 prompt is then displayed for the second buffer.

14.7 Rinse the probes and place in the second buffer. Wait for a stable reading and enter the correct buffer value, then press "yes". The P3 prompt for the third buffer will then be displayed.

14.8 Rinse the probes and place in the third buffer. Wait for a stable reading and the correct buffer value, then press "yes".

14.9 When the P4 prompt is displayed, press "measure". Record the displayed calibration slope on the benchsheet and in the run logbook. The slope needs to be 90 – 102%. If not, clean the electrode. Read each buffer as a sample. (each must read correct to within ±0.05 pH units). If not, repeat the calibration until all buffers read within ±0.05 pH units. When the meter is calibrated, it is ready for sample analysis.

14.10 All buffers must be used fresh. Do not save to use on another meter. Discard immediately after the calibration is complete.

14.11 During calibration and analysis, all buffer solutions and samples must be gently stirred at a constant rate great enough to minimize drift (<0.1 pH unit) but not enough to generate bubbles and/or aeration that might introduce carbon dioxide into solution from the air.

15.0 ANALYTICAL PROCEDURE

15.1 Before checking the pH, adjust the probe clamp, so immersion will be just deep enough into the solution to establish good electrical contact through the electrode fiber-capillary hole. Rinse probes thoroughly with laboratory reagent water before transferring to another solution. Discard the rinseate.

NOTE: Standard and sample temperatures must be within ±2° C of the calibration buffer temperature. Also, do not let samples sit open on the bench or over-stir when taking a reading as exposure to the air and/or excessive stirring will introduce carbon dioxide and lower the pH.

15.2 Place the probe in the Initial Calibration Verification (ICV) buffer. An ICV is the midpoint buffer of the calibration (midpoints are 4.00, 7.00 or 10.00). Gently stir at the same rate used during calibration. Wait for the meter to beep and display "ready" then record the pH result to the second decimal place. The value obtained must be within ±0.05 pH units of the certified value for acceptability. Rinse the probe thoroughly with reagent water.

15.3 Repeat with Laboratory Control Samples (LCS) as specified in Attachment 23.5. These readings must also be within ±0.05 pH units of the certified value for acceptability. Sample analysis may not begin until ICV and LCS results are acceptable.

15.4 After acceptable ICV and LCS results are obtained, measure the pH of up to 10 samples (this count includes the LCS). Repeat sample analysis on multiple volumes until the difference in readings is less than 0.1 pH units. Two or three volumes are usually sufficient. Report the value obtained.

15.5 After 10 analyses, analyze a Continuing Calibration Verifications (CCV) as specified in Attachment 23.5. The CCV will be the same buffers used to calibration the instrument. The measured result must be within ±0.05 pH units of the certified value.

15.6 Provided the CCV buffers pass, sample analysis may continue with a CCV being analyzed after every 10 analyses. The analytical batch must also be closed with an acceptable CCV.

15.7 If a CCV fails, the previous 10 samples must be reanalyzed. Before reanalyzing, clean the electrode in accordance with Section 6.3 then recalibrate.

16.0 CALCULATIONS AND DATA HANDLING

16.1 The pH meter measures all data against the calibration and displays a direct pH reading. No manual calculations are necessary.

16.2 Rules for significant figure reporting:

16.2.1 Report values to 0.1 pH units for samples (example: report 7.6, not 7.59).

16.2.2 Report values to 0.01 pH units for quality control (example: report 7.63, not 7.6).

16.2.2 Report pH of less than 2.0 as "pH <2.0". Do not report pH values less than 2.0.

16.2.3 Report pH of greater than 12.0 as "pH>12.0". Do not report pH values greater than 12.0.

17.0 DATA REPORTING AND DELIVERABLES

17.1 Analysts processing sample batches are responsible for data quality and for correctly filling in all documentation. This is required for quality control and to provide clients with fully defensible data.

17.2 The following data and all other data specified throughout this procedure must be recorded on the Element™ printout and/or in the logbook for each batch handed in:

17.2.1 The standards logbook number of calibration buffers used (logbook)

17.2.2 Sample temperature (benchsheet)

17.2.3 The pH result (benchsheet)

17.2.4 Observed values of each buffer used for calibration (benchsheet)

17.2.5 The slope (benchsheet)

17.2.6 Method name and number (benchsheet)

17.2.7 Refer to Attachment 23.1 for an analysis sequence report example.

17.3 Instrument logbooks must be filled in for each batch handed in with the following information.

17.3.1 Date analyzed

17.3.2 Analyst's initials

17.3.3 Calibration standards used

17.3.4 Client name and sample numbers analyzed

Note: Refer to Attachment 23.3 for an instrument logbook example.

17.4 All logbooks must be filled in completely and correctly. All corrections must be made in indelible ink. **Corrections must be made with a single dated/initialed lineout. Write-overs are not acceptable.** An incorrectly entered result must remain legible even after the correction is made. A new result must be placed near the incorrect result with the date and analyst's initials written next to it. Blank lines in logbooks must be Z'd out.

17.5 If internal chain-of-custody (COC) is required, it is very important that the COC form be filled in correctly and completely.

18.0 QUALITY ASSURANCE

18.1 Method and matrix quality control must be analyzed with each batch.

18.1.1 Quality control consists of two laboratory control samples (LCS), an initial calibration Verification (ICV) and continuing calibration verifications (CCV) as specified in Attachment 23.5. Acceptance limits are ±0.05 pH units of the certified value.

18.1.1.1 Acceptance limits are ±0.05 pH units of the certified value.

18.1.1.2 Quality control samples need included in the frequency count.

18.1.2 Matrix quality control consists of a sample duplicate (DUP) every 10 analyses. A DUP is an analysis replicate analyzed in the same way as the original. Acceptance limits in relative percent difference (RPD) are listed in the laboratory information management system (Element™).

18.2 Refer to TriMatrix GR-10-106 for the appropriate corrective action needed when an out-of-control condition exists.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before performing sample analysis, each analyst must first demonstrate the ability to generate acceptable results by running an initial demonstration of capability study. Also, while a demonstration of capability study is not instrument dependent, it is required for each pH meter as well. Analyst and pH meter capability studies must be repeated annually.

19.2 Prepare four aliquots of a soil sample following every step in the procedure. Have an experienced analyst independently prepare four aliquots of the same sample for comparison.

19.3 Calibrate the pH meter and analyze the four aliquots. Have the experienced analyst do the same with the comparison set.

19.4 Calculate average recovery (x) and standard deviation of the average (s) in pH units by entering the eight results into the Smith-Satterthwaite spreadsheet for determining demonstration of capability acceptance. The spreadsheet is located on the laboratory intranet library.

19.4.1 The spreadsheet will calculate a statistical comparison between the two sets of data. If the spreadsheet determines the two analyst averages are statistically indistinguishable, the analyst and pH meter are authorized to run samples.

19.4.2 If the spreadsheet indicates the two data sets are not indistinguishable, the analyst must proceed according to Section 19.5.

19.5 Locate and correct the source of the problem and repeat the test. Repeated failure however, will confirm a general problem with the procedure or technique used. If this occurs, locate and correct the procedure or technique, then repeat the test beginning with section 19.2. Samples may not be analyzed by any analyst or on any pH meter until a demonstration of capability study has been successfully completed. Copies of successful studies, spreadsheets and raw data must be given to the Quality Assurance department.

19.6 A pH demonstration of capability study must be performed annually by all analysts. This may be accomplished by repeating the initial test or by submitting an acceptable performance testing study result on the appropriate paperwork.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve the use of chemicals where applicable

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate material safety data sheet (MSDS) when disposing of chemicals. This information is located on the laboratory intranet library in the MSDS folder.

21.2 Follow all applicable instructions in GR-15-102 for laboratory waste disposal.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 9045C, "Soil and Waste pH", Revision 3, January, 1995

23.0 ATTACHMENTS

23.1 Analysis Sequence Report Example

23.2 Preparation Batch Report Example

23.3 Instrument Logbook Example

23.4 Standards Log Report Example

23.5 pH Calibration Flowchart

Attachment 23.1
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE **6062935** Page 1 of 1 Printed: 5/30/2007 2:06:02PM

Inorganic - Wet Chemistry, Waste, Jun-29-06

Instrument = 164, Calibration = UNASSIGNED

Sequence Analyses:
pH 9045C

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
6062935-ICV1	QC		A512818		INITIAL CAL CHECK	
0607475-SRM1	QC				REFERENCE	
0607475-SRM2	QC				REFERENCE	
6062935-CCV1	QC		A512818		CALIBRATION CHECK	
0606497-01	pH 9045C	A			[redacted]	
0607475-DUP1	QC				DUPLICATE	
6062935-CCV2	QC		A512818		CALIBRATION CHECK	

Comments:	Analyst Initials:

seq_TriMatrix rpt

Approved By: 2-5-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 23.2
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0703937 Page 1 of 1

Printed: 5/30/2007 1:49:20PM

Inorganic - Wet Chemistry, Waste, General Inorganic Prep

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0704160	pH 9045C				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client/QC Type	Extraction Comments
0703937-DUP1		Apr-16-07 11:14	CLB	20	20		0704160-01			DUPLICATE	
0703937-SRM1		Apr-16-07 11:14	CLB	20	20			A608776	20000	REFERENCE	
0703937-SRM2		Apr-16-07 11:14	CLB	20	20			7030694	20000	REFERENCE	
0704160-01	A	Apr-16-07 11:14	CLB	20	20					[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: 2-5-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

Attachment 23.3
Instrument Logbook Example

TriMatrix Laboratories, Inc.

Date	Analyst	Buffer Solution pH Concentrations and Lot Numbers (fill in Lot Numbers only for Buffers Used)					Calibration Verification Level and Lot Number		CV Results	Acceptable CV (±0.05 TV)	Client and Sample Numbers
		2	4	7	10	12	TV	Lot Number			
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	
										yes / no	

Approved By: 2-5-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

Attachment 23.4
Standards Log Report Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8030282

Description:	pH buffer 8.00	Expires:	Aug-31-09
Standard Type:	Analyte Spike	Prepared:	Aug-31-07
Solvent:	Solvent Lot #1708575	Prepared By:	** Vendor **
Final Volume (mls):	500	Department:	Inorganic - Wet Chemistry
Vials:	2	Last Edit:	Jun-13-08 15:29 by BAT

VWR part# BDH5060-500ml
Lot: 1708575
Received 3/07/08

Analyte	CAS Number	Concentration	Units
pH		8	pH Units
Carbon Dioxide, Free		8	pH Units
Carbon Dioxide	124-38-9	8	pH Units
Acidity as CaCO3		8	pH Units
Acidity		8	pH Units

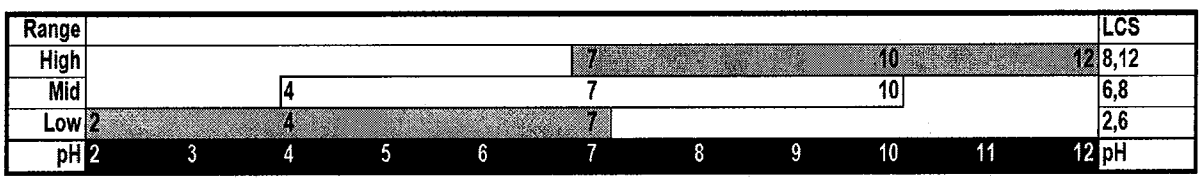
Approved By: m 2-5-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.5
pH Calibration Flowchart

TriMatrix Laboratories, Inc.

pH Calibration and Quality Control



1. Run three point curve, ALWAYS starting with 7: Slope of the curve must be between 90 - 102%.
   High: 7, 10, 12
   Mid: 7, 4, 10
   Low: 7, 2, 4

2. Re-run each point of the curve as a sample as ICVs. Response must be ±0.05 pH units of true value.

3. Run the LCSs appropriate to the curve: Response must be ±0.05 pH units of true value. The LCSs are second source buffers.
   High: 8, 12
   Mid: 6, 8
   Low: 2, 6

4. Every 10 samples and at end of batch run CCVs: Response must be ±0.05 pH units of true value. The CCVs are the same buffers as the curve.
   High: 10, 12
   Mid: 4, 10
   Low: 2, 4

Approved By: m 2-5-09
QA Officer

Approved By: Heather L. Brady
Area Supervisor

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Extraction of Base Neutrals and Acids from Water

EPA Method 625
SW-846 Method 3510C

APPROVALS:

Area Supervisor: _____ Date: 2/12/09
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By: Dan J. Mierendorf
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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure describes extraction of insoluble and slightly soluble semi-volatile organic base-neutral and acid compounds from aqueous samples and TCLP leachates. Also described are concentration techniques preparatory to GC/MS analysis.

1.2 The procedure is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by performance of a satisfactory demonstration of capability study.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.* Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"

2.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

3.0 SUMMARY OF PROCEDURE

3.1 A measured sample volume, usually 1 L, is serially extracted with methylene chloride at acidic pH, then again at basic pH using separatory funnels.

3.2 Extracts are dried and concentrated to a final volume of 1.0 mL or 10 mL before GCMS analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to Table 1 in TriMatrix SOP GR-04-103 for a compound list associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-04-103, *Semi-Volatile Laboratory Ion Trap Mass Spectrometry Analysis of Base/Neutral/Acid Compounds*, latest revision

5.2 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-16-100, *Organic Glassware Cleaning,* latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware. Contamination leads to discrete artifacts and elevated baselines. A method performance blank (MPB) must be performed routinely, to demonstrate that equipment and reagents are not contaminated.

6.2 Always use high purity reagents, solvents and clean glassware, cleaned in accordance with TriMatrix SOP GR-16-100.

6.3 Matrix interference is co-extracted with analyte from samples. Extraction interferences will vary considerably from matrix to matrix.

6.4 Phthalate esters co-elute during analysis as extraneous chromatographic peaks. Do not use any type of flexible plastic during extraction. Contact with extraction solvents will leach phthalate esters into sample extracts, presenting interference in compound identification and quantitation.

6.5 Matrix interference can also affect analyte recovery. Re-extraction may be necessary if surrogates or other spiked compounds fail established laboratory recovery limits.

7.0 SAFETY PRECAUTIONS

7.1 Extraction personnel must wear lab coats and approved safety glasses while in the prep lab area. In addition, disposable elastic gloves must be worn whenever samples, solvents or reagents are handled.

7.2 For extractions and surrogate/spike/reagent solution preparations, follow all safety instructions outlined in the latest revision of the Chemical Hygiene Plan and Laboratory Safety Manual.

7.3 For proper spill response, chemical storage and waste disposal refer to the latest revision of TriMatrix SOP GR-15-102.

7.4 Toxicity and carcinogenicity of materials used in this procedure have not been completely and precisely defined.

7.4.1 All chemicals must be treated as a potential health hazard.

7.4.2 Exposure can be reduced to the lowest possible level by adherence to established safety practices.

7.4.3 Material Safety Data Sheets are maintained within the laboratory (in the intranet library) of all chemicals used in this procedure.

7.5 Safety issues must be brought to the immediate attention of the area supervisor.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

8.2 All aqueous samples must be extracted within 7 days of the collection date. TCLP leachates must be extracted within 7 days of complete leachate generation. Extract analysis must occur within 40 days of the extraction.

8.3 When not in use, samples must be stored in the walk-in cooler at 4° C ± 2° C, or as specified by the project.

8.4 Analysts must use care when handling sample containers to avoid loss or injury due to breakage.

8.5 After extraction, extracts must be stored in the GC/MS refrigerator at 4° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Graduated cylinders: 10 mL, 100 mL, 1000 mL

9.2 Separatory funnels with PTFE stopcocks: 2 L

9.3 Erlenmeyer flasks: 250 mL, 500 mL

9.4 Kuderna-Danish (K-D) concentrator glassware

9.4.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.4.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.4.3 Concentrator tube clips

9.4.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.4.5 Snyder columns: Two-ball micro

9.5 pH indicator strips: pH range 0-14

9.6 Filter funnels: 100 mm

9.7 Filter funnels: 250 mL, ribbed

9.8 Filter paper: Fisher P8 qualitative or equivalent

9.9 Glass wool

9.10 Pasteur pipettes: Glass disposable, 2 mL

9.11 Water bath: Capable of variable temperature control to ±5° C

9.12 Hot plate: Capable of variable temperature control to ±5° C

9.13 Beakers, glass: 100 mL, 400 mL and 800 mL

9.14 Boiling chips: PTFE, rinsed with methylene chloride

9.15 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.16 Vials with PTFE-lined screw-cap lids: Amber, 40 mL

9.17 Syringes, micro gastight: 100 uL and 1000 uL

9.18 Centrifuge tubes: PTFE, 40 mL

9.19 Centrifuge: Variable speed

9.20 Vials: Glass auto-sampler with PTFE-lined screw-cap lids, 1.7 mL calibrated to 1.0 mL (as outlined in SOP number GR-09-106)

9.21 Vials: Glass PTFE-lined screw-cap lids, 14 mL calibrated to 10 mL (refer to TriMatrix SOP GR-09-106)

9.22 Balance: Analytical, capable of accurate measurement to the nearest 0.0001 g

9.23 Sodium sulfate rinsing equipment

9.23.1 Buchner funnel: 20 cm

9.23.2 Flask: 4 L Vacuum

9.23.3 Drying pan: 13 x 9 inch, metal

9.23.4 Vacuum pump

9.23.5 Drying oven

9.23.6 Stopper: PTFE, with hole for the Buchner funnel

9.23.7 Clamps: supporting

9.23.8 Filter paper: qualitative fast, 20 cm

9.23.9 Squirt bottle: PTFE only

9.24 Phase separation paper (JT Baker 8002-15)

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Glass syringes must be cleaned or replaced when needles appear bent or plugged.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above reporting limits.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride, to minimize interference from phthalate ester contamination.

11.1.2.1 To a 1000 mL separatory funnel, add 1000 mL of reagent water.

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

11.2 Methylene chloride: pesticide grade or equivalent

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed

11.3.1 Sodium sulfate must be solvent-rinsed before use. Assemble the rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 Kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing the insides down with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour the methylene chloride in the container over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride, if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from a PTFE squirt bottle. Maintain vacuum until the solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

Note: The oven must be vented to the outside to prevent methylene chloride fumes from entering the laboratory.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.8 After cooling, return to the original container using a clean glass funnel.

11.3.9 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.

11.4 Sodium hydroxide solution, 10N: (prepared from ACS grade pellets)

11.4.1 Carefully add 400 g sodium hydroxide to 1 L of laboratory reagent water that has not been methylene chloride extracted.

11.4.2 Stir on a magnetic stir plate until dissolved then store in a closed and labeled glass container.

Caution: Adding sodium hydroxide to water will generate significant heat. Add pellets slowly and cool to room temperature before using.

11.5 Sulfuric acid solution, 1:1 (v/v): Reagent ACS

11.5.1 Slowly add 500 mL of concentrated sulfuric acid to 500 mL of laboratory reagent water that has not been extracted with methylene chloride.

11.5.2 Stir carefully when cool then store in a closed, labeled glass container.

Caution: Adding sulfuric acid will generate significant heat. Add slowly to avoid splattering and cool before using. **ALWAYS ADD SULFURIC ACID TO WATER (NEVER ADD WATER TO SULFURIC ACID).**

11.6 Methanol: pesticide grade or equivalent

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions are made by the following rules:

12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.2 Use the appropriate solvent for dilution.

12.1.3 Label all glassware and vials holding surrogate/spiking solutions.

12.1.4 Use minimal headspace in vials.

12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date prepared

12.2.4 Analyst initials

12.2.5 Solvent used

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Surrogate/spike data are recorded in an extraction standards logbook. Solutions are labeled with a two-letter prefix followed by a number depending upon logbook page and line. Surrogate/spiking solutions are stored in the prep lab refrigerator at 4° ±2° C. A dilution of spiking solution must be analyzed by the GC/MS laboratory to check concentration before use by the extraction lab. Concentrations must be within established laboratory acceptance limits of 80-120%.

12.4 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If neat compound purity is below 95%, concentration must be accounted for in analytical calculations. Locate neat chemicals within the laboratory by use of the chemical inventory list. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered.

12.4.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Over-sonicating will generate excessive heat. Dilute to volume and mix well by inverting at least three times. Discard the volume in the neck of the flask and transfer to pre-labeled vials. Store the vials at 4° ±2° C.

12.4.2 For neat liquids, weigh into a tared volumetric flask half-full of appropriate solvent. Add drop-wise using a Pasteur pipette directly into the solvent, being careful not to touch the sides of the flask. Record the mass, swirl then dilute to volume. Mix well and discard the volume in the neck of the flask. Transfer to pre-labeled vials and store at 4° ±2° C.

12.5 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however, if a manufacturer's expiration date occurs within that time for neat materials used. Laboratory-assigned expiration dates cannot exceed the manufacturer's expiration date. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.6 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration.

$$10000\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 100\ \text{mg/L}$$

$$100\ mg/L \times \frac{1\ mL\ stock\ standard}{100\ mL\ final\ volume} = 1.0\ mg/L$$

12.7 When making a dilution, all data must be entered into Element™.

12.7.1 If a dilution of a dilution is made then the resultant solution would be given another number.

12.7.2 For solution preparation, use only pesticide-grade or better solvents.

12.7.3 Before using a solution, calculations performed when calculating concentration must be verified by a second analyst. Once calculations have been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a solution cannot be used. Acceptance limits are based on 80% to 120% of calculated values.

12.8 At the end of a shift, vials containing 5 mL or less must be disposed of to minimize contamination and concentration in the solution.

12.9 Acid and base compound surrogate solutions are made separately, from neat materials.

12.9.1 Add 0.5 g of each base-neutral surrogate compound to a 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.

12.9.2 Add 1.0 g of each acid surrogate compound to another 250 mL volumetric flask half-filled with methanol. Swirl, shake or sonicate to dissolve then dilute to volume with methanol.

12.9.3 Add 100 mL of each solution to a 2 L volumetric half-filled with methanol. Swirl to mix then dilute to volume with methanol. Have this solution checked by GC/MS. Once approved, the prep lab and the mass spec lab must coordinate to be sure the same surrogate mixture is used for calibration and spiking.

12.9.4 Transfer the entire 2 L solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Surrogate	**Mass (g)**	**Dilution Volume (mL)**	**Stock Concentration (ug/mL)**	**Secondary Dilution (mL:mL)**	**Final working Concentration (ug/mL)**
Decafluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
o-terphenyl	0.5	250 in methanol	2000	100:2000	100
Nitrobenzene-d_5	0.5	250 in methanol	2000	100:2000	100
2-Fluorobiphenyl	0.5	250 in methanol	2000	100:2000	100
2,4,6-Tribromophenol	1.0	250 in methanol	4000	100:2000	200
Phenol-d_6	1.0	250 in methanol	4000	100:2000	200
2-Fluorophenol	1.0	250 in methanol	4000	100:2000	200

12.10 Spike solutions with reference to EPA Method 8270C are purchased in individual base/neutral and acid ampules except for naphthalene, which is diluted in methylene chloride from the neat compound.

12.10.1 Weigh 1.0 g of naphthalene into a 100 mL volumetric flask. Add approximately half-volume of methylene chloride to the flask and swirl, shake or sonicate to dissolve then dilute to volume with methylene chloride. Concentration of this solution is 10,000 ug/mL naphthalene.

12.10.2 Pipette 1.0 mL of acid ampule #1 into a 50 mL volumetric flask half-filled with methanol.

12.10.3 Into the same 50 mL volumetric flask, pipette 1.0 mL of base/neutral ampule #2.

12.10.4 Pipette 0.5 mL of the naphthalene solution into the flask then dilute to volume with methanol.

12.10.5 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.10.6 Transfer the entire solution to 40 mL amber vials with PTFE-lined screw-cap lids and store in the prep.laboratory refrigerator.

Compound	Mass	Dilution Volume (mL)	Stock/Ampule Concentration (ug/mL)	Secondary Dilution (mL:mL)	Final working Concentration (ug/mL)
Naphthalene	1.0 g	100($MeCl_2$)	10000	2.0:200	100
4-Chloro-3-methylphenol (#1)			7500	5.3:200	200
2-Chlorophenol (#1)			7500	5.3:200	200
4-Nitrophenol (#1)			7500	5.3:200	200
Pentachlorophenol (#1)			7500	5.3:200	200
Phenol (#1)			7500	5.3:200	200
Acenapthene (#2)		5000	4.0:200	100	
1,4-Dichlorobenzene (#2)			5000	4.0:200	100
2,4-Dinitrotoluene (#2)			5000	4.0:200	100
N-Nitrosodi-n-propylamine (#2)			5000	4.0:200	100
Pyrene (#2)			5000	4.0:200	100
1,2,4-Trichlorobenzene (#2)			5000	4.0:200	100

12.11 Spike solutions with reference to EPA Method 625 are purchased from NSI (Catalog # C-397) at 100 ug/mL. These solutions are used as received and expiration is 6 months from date received. No dilution is required.

12.12 Spike solution with compounds for reference to SW-846 Method 1311 is purchased from Ultra Scientific (Catalog # TCLP-512).

12.12.1 Pipette 1250 µL of 2000 ug/mL purchased solution into a 25 mL volumetric flask half-filled with methanol and dilute to volume.

12.12.2 Working spike solutions are checked by GC/MS before using. If all spike compounds are within 80 to 120% of calculated values then the solution is approved for use.

12.12.3 Transfer the entire solution to a 40 mL amber vial with PTFE-lined screw-cap lids and store in the prep laboratory refrigerator.

Compound	Ampule Concentration (mg/L)	Secondary Dilution (mL:mL)	Final working Concentration (mg/L)
o-cresol	2000	1.25:25	100
m-cresol	2000	1.25:25	100
p-cresol	2000	1.25:25	100
1,4-dichlorobenzene	2000	1.25:25	100
2,4-dinitrotoluene	2000	1.25:25	100
hexachlorobenzene	2000	1.25:25	100
hexachlorobutadiene	2000	1.25:25	100
hexachloroethane	2000	1.25:25	100
nitrobenzene	2000	1.25:25	100
pentachlorophenol	2000	1.25:25	100
pyridine	2000	1.25:25	100
2,4,5-trichlorophenol	2000	1.25:25	100
2,4,6-trichlorophenol	2000	1.25:25	100

12.13 Additional spiking solutions for client-specific analytes will be prepared as needed.

13.0 ANALYTICAL PROCEDURE

13.1 Rinse all glassware with methylene chloride before use.

13.2 Label each Erlenmeyer flask with the following information:

13.2.1 Sample number

13.2.2 "S" for surrogate

13.2.3 "A" for acid when appropriate

13.2.4 "B" for base when appropriate

13.2.5 "L" for spike when appropriate

13.2.6 Method

13.2.7 Final volume

13.3 Label Kuderna-Danish (K-D) flasks and concentrator tubes:

13.3.1 Sample number

13.3.2 Parameter (BNA, TCLP, as appropriate)

13.3.3 Final volume

13.4 Determine sample volume as outlined below.

13.4.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark then measure with a graduated cylinder to attain the sample volume.

13.4.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE. After sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.

13.4.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting. Extracting less than 500 mL will result in elevated reporting limits, which must be client approved.

Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.

13.5 Add 100 µL of surrogate solution to each separatory funnel with a 100 µL syringe, to extracts taken to 1.0 mL final volume. Add 1.0 mL of surrogate to each separatory funnel with a 1000 µL syringe, to extracts taken to 10.0 mL final volume.

13.5.1 Syringes used must be dedicated to BNA surrogate additions.

13.5.2 Surrogate is added to all extractions including samples, Extraction Blanks (BLK), Blank Spikes (BS) and Matrix Spikes.

13.5.3 Do not submerge the syringe into a liquid when injecting surrogate. Slowly inject approximately one inch above the liquid's surface.

13.5.4 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.

13.5.5 Cross out the "S" on each corresponding Erlenmeyer flask as surrogate is added.

13.6 Add 100 µL of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 1.0 mL. Add 1.0 mL of spiking solution to Matrix Spikes, Matrix Spike Duplicates and Blank Spikes, if final volume will be 10.0 mL. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.7 Acidify samples by adding approximately 2 mL of 1:1 sulfuric acid solution to each separatory funnel using a Pasteur pipette. Mix well by swirling and/or shaking.

13.8 Rinse the sample jar and/or graduated cylinder with methylene chloride:

13.8.1 Add approximately 100 mL methylene chloride to each sample jar. Replace the lid (very important).

13.8.2 Shake and vent as necessary. If a graduated cylinder was used to measure the sample volume, transfer the methylene chloride from the sample container into the graduated cylinder. Swirl the methylene chloride to rinse the cylinder sides.

13.8.3 Transfer the methylene chloride into the separatory funnel containing sample.

13.9 Acid Extraction:

13.9.1 Stopper the separatory funnel and invert once. Immediately open the stopcock to release solvent pressure.

13.9.2 Close the stopcock, shake and vent again.

13.9.3 Repeat until there is no evidence of pressure.

13.9.4 Shake for one to two minutes.

13.9.5 Let the separatory funnel sit for at least ten minutes for phase separation.

13.9.5.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into a separatory funnel to take a reading.

13.9.5.2 The pH should be less than 2. If not, add additional 2 mL aliquots of acid until a pH of less than 2 is attained then cross out the "A" on the corresponding Erlenmeyer flask. Add no more acid than one percent of the initial sample volume (10 mL for a 1000 mL initial volume) as larger additions constitute a significant extraction volume change.

13.9.5.3 Consult with the project chemist if a sample cannot be adjusted to a pH less than 2 with the maximum acid volume.

13.9.6 If a heavy emulsion has formed, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.

13.9.6.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.

13.9.6.2 If separation does not occur, drain the entire volume from the 125 mL funnel into a PTFE centrifuge tube (this may require multiple tubes). Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.

13.9.7 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the corresponding 500 mL Erlenmeyer flask, passing it through phase separation paper. Rinse the paper thoroughly.

13.9.8 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.

13.10 Repeat the extraction twice more, each time using approximately 60 mL of methylene chloride.

13.10.1 Repeat the process of rinsing the sample container and (if used) graduated cylinder with methylene chloride, then transfer to the separatory funnel for the second extraction.

13.10.2 Add the methylene chloride directly to the separatory funnel for the third extraction.

13.10.3 Approximately 200 mL of acid solvent extract will be collected.

13.11 After the acid extraction is complete, adjust pH to greater than 11 by adding approximately 7 mL of 10N sodium hydroxide solution to each separatory funnel using a graduated cylinder. Mix well by swirling and/or shaking.

13.12 Base-Neutral Extraction

13.12.1 Add 60 mL of methylene chloride.

13.12.2 Stopper the separatory funnel and invert. Immediately open the stopcock to release pressure.

13.12.3 Close the stopcock, shake and vent again.

13.12.4 Repeat until there is no evidence of pressure venting.

13.12.5 Shake for an additional two minutes.

13.12.6 Let the separatory funnel sit for at least ten minutes, for phase separation.

13.12.6.1 Check the pH of each solution using a pH indicator strip by removing a few drops from the separatory funnel with another clean Pasteur pipette. Do not dip the pH strip directly into separatory funnel to take a reading.

13.12.6.2 The pH should be greater than 11. If not, add additional 1 mL aliquots of sodium hydroxide until a pH of greater than 11 is attained, then cross out the "B" on the corresponding Erlenmeyer flask. Add no more than one percent of the initial extraction volume (10 mL for a 1000 mL initial volume) as larger additions constitute sample dilution.

13.12.6.3 Consult with the project chemist if a sample cannot be adjusted to a pH greater than 11 with the maximum base volume.

13.12.7 If a heavy emulsion forms during extraction, drain the emulsion into a 125 mL separatory funnel and shake vigorously. Let sit for at least ten minutes.

13.12.7.1 If separation occurs in the 125 mL funnel, drain the solvent phase into the Erlenmeyer flask. Drain the water back into the 2 L funnel.

13.12.7.2 If separation does not occur, drain the entire contents of the 125 mL funnel into a PTFE centrifuge tube. Centrifuge for five minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom phase into the Erlenmeyer flask.

13.12.8 If a light emulsion has formed, drain the methylene chloride (bottom layer) into the 500 mL Erlenmeyer flask containing acid compound extract, passing it through phase separation paper. Rinse the paper thoroughly.

13.12.9 If there is no emulsion, drain the methylene chloride directly into the Erlenmeyer flask.

13.13 Repeat the base-neutral extraction twice more, each time using approximately 60 mL of methylene chloride. A total of approximately 400 mL of BNA extract will be collected for concentration.

13.14 When extractions are complete, if a graduated cylinder was not used to determine sample volume, determine the volume by filling the sample container with tap water up to the water level mark. Transfer to a graduated cylinder and record the volume.

13.15 Remove all sample identification tags from the empty sample jar and discard both.

13.16 Transfer extract to a K-D concentrator:

13.16.1 Quantitatively transfer each extract from the 500 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into Kuderna-Danish concentrators, with an attached 10 mL graduated concentrator tube.

13.16.2 Rinse the flask and funnel with several 20-30 mL aliquots of methylene chloride to complete the transfer.

13.17 Concentrate the extracts

13.17.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.17.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

13.17.3 Place the concentrators in a water bath (70-80° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.17.4 At the proper rate of distillation, the column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder columns periodically.

13.17.5 When the apparent volume of liquid reaches 3-4 mL (in ten to fifteen minutes), remove the K-D and let drain for at least 10 minutes.

13.17.6 Remove the Snyder column, rinsing the lower joint into the concentrator tube with 2 to 3 mL of methylene chloride.

13.18 Micro-Snyder column technique is used to adjust final extract volumes to 1.0 mL. Blowdown is not used when adjusting final volume to 1.0 mL, to minimize analyte loss from concentration.

13.18.1 Micro-Snyder Column Technique:

13.18.1.1 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.18.1.2 Pre-wet the column with 0.5 mL methylene chloride.

13.18.1.3 Place concentrator tubes in a water bath, partially immersed. Water temperature should be 60-70° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.

13.18.1.4 When the apparent volume reaches 0.5 mL, remove from the bath. Cool for at least ten minutes.

13.18.1.5 Rinse the ground-glass joints with 0.2 mL methylene chloride after removing Snyder columns.

13.18.1.6 Transfer extracts to a 1.0 mL calibrated vial using a clean disposable Pasteur pipette.

13.18.1.7 Using approximately 0.2 mL of methylene chloride, rinse each concentrator tube, then transfer to the vial.

13.18.1.8 Adjust the final volume to 1.0 mL with methylene chloride then cap tightly.

13.18.1.9 Extracts are ready for analysis and must be stored in the GC/MS sample refrigerator at 4 ±2° C.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

15.1 Extraction analysts must extract an Extraction Blank and Blank Spike daily, once per shift or every 20 samples (whichever is more frequent) to demonstrate that extraction interferences are under control.

15.2 Matrix Spikes and Matrix Spike Duplicates (provided enough sample volume is received) are extracted at a minimum of once every twenty samples or once a week, depending upon the frequency of sample receipt.

15.2.1 Performance records are maintained to document data quality generated.

15.2.2 Matrix spiked samples are prepared for analysis by first adding spike solution and surrogate before extraction.

15.3 Blank Spikes are prepared by extracting the addition of BNA spike solution and surrogate solution to 1 L of pre-extracted laboratory reagent water.

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation and re-extraction may be necessary if surrogate or spiked compounds fail to pass laboratory established acceptance limits.

15.5 Surrogate must be added to all extractions.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting a demonstration of capability study.

16.2 Prepare a spiking solution that will give final extract concentrations, mid-range to the GC/MS calibration. This solution must be prepared independent of GC/MS calibration standards. Prepare four (4) 1000 mL aliquots of pre-extracted water. Inject 100 µL of spiking solution into each. Add surrogate, then extract as samples, going through all extraction steps in this procedure, to a final volume of 1.0 mL.

16.3 After extraction, have the four (4) extracts analyzed for all spiked compounds in accordance with all GC/MS procedure requirements.

16.4 Input results to the IDC spreadsheet to calculate average percent recovery (x) and percent relative standard deviation of recovery (s), for each analyte, using the four results.

16.4.1 For each analyte, (x) must be within historical statistical recovery limits and (s) must be less than or equal to 20%. If (s) and (x) for all analytes meet acceptance, the demonstration of capability study is acceptable. The analyst is authorized to extract samples by the procedure.

16.4.2 If one or more analytes fail at least one acceptance limit, the analyst must proceed through the following steps.

16.4.2.1 Locate and correct any source of error then repeat the demonstration of capability only for analytes not meeting acceptance. If the failed analytes are acceptable on the second attempt, the study is complete and acceptable.

16.4.2.2 Repeated failure will indicate general problems with the extraction procedure or analyst technique. If this occurs, correct and revise any faulty steps in the procedure or technique.

16.4.2.3 Once corrective action has been taken, re-extract the demonstration of capability study for all analytes. If all analytes are acceptable, the study is complete and acceptable.

16.5 Samples may not be prepared by any analyst until an acceptable demonstration of capability study has been successfully completed.

16.6 A successful continuing demonstration of capability (CDC) study must be completed annually by all analysts by one of the following approaches:

16.6.1 By repeating the IDC study.

16.6.2 By using four consecutive blank spike results obtained during the course of routine sample extraction and when extracted exclusively by the analyst.

16.6.3 By extracting an acceptable blind performance testing sample.

16.6.4 By exclusively extracting a method detection limit (MDL) study. The last four results of the study may be used.

16.7 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor chemical use.

17.2 Reference any specific written disposal instructions before disposing of laboratory chemicals.

17.3 Conserve the use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material Safety Data Sheets are located on the laboratory intranet library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.

18.3 Follow all instructions in TriMatrix Laboratory SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.* Final Update III, Revision 3, December 1996, Method 3510C, "Separatory Funnel Liquid-Liquid Extraction"

19.2 *40 Code of Federal Regulations, most current edition*, Pt. 136, App. A, Method 625-Base/Neutrals and Acids

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Standards Log Report Example

20.3 Water Extraction Logbook Example

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0801114 Page 1 of 1 Printed: 11/7/2008 10:14:54AM

Semivolatiles MS, Waste Water, 625 Liquid/Liquid Extraction

Batch comments:

(none)

Lab Number	Container	Prepared	By	Initial (mL)	Final (mL)	Client Source ID	Client Spike ID	Client uL Spike	Comments	
0801114-BLK1		Feb-01-08 08:23	KB9	1000	5					
0801114-BLK2		Feb-05-08 08:23	ASC	1000	1				same as batch 0801235	
0801114-BS1		Feb-01-08 08:23	KB9	1000	5		8010650	500		
0801114-BS2		Feb-05-08 08:23	ASC	1000	1		8010650	100	same as batch 0801235	
0801114-BSD1		Feb-01-08 08:23	KB9	1000	5		8010650	500		
0801114-DUP1		Feb-01-08 08:23	KB9	1000	5	0801501-19				
0801114-DUP2		Feb-01-08 08:23	KB9	1000	5	0801501-20				
0801114-DUP3		Feb-01-08 08:23	KB9	1000	5	0801501-25				
0801114-DUP4		Feb-01-08 08:23	KB9	1000	5	0801501-26				
0801114-DUP5		Feb-05-08 08:23	ASC	1000	1	0801501-27				
0801501-19	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)	*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custo*							
0801501-20	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)	*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custo*							
0801501-25	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			acids, base neutrals	acids, base neutrals
625 SVOCs (master list)	*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custo*							
0801501-26	A	Feb-01-08 08:23	KB9	1000	5	[redacted]			acids, base neutrals	acids, base neutrals
625 SVOCs (master list)	*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custo*							
0801501-27	A	Feb-05-08 08:23	ASC	1000	1	[redacted]			Added for BatchQC in: 0801114	BatchQC
625 SVOCs (master list)	*625 SVOCs (TTOs)*		*625 SVOCs (TTOs) custo*							

Comments:	Analyst Initials:

bch_TM_byAnalysis.rpt

Approved By: 2-12-09 **QA Officer** **Approved By:** BJL 2/12/09 **Area Supervisor**

Attachment 20.2
Standards Log Report Example

Analytical Standard Record

TriMatrix Laboratories, Inc.

8060471

Description:	BNA Surrogate	Expires:	Nov-10-08
Standard Type:	Surrogate Spike	Prepared:	Jun-16-08
Solvent:	MeOH Lot#076070	Prepared By:	Andrea S. Colborn
Final Volume (mls):	2000	Department:	Semivolatiles MS
Vials:	1	Last Edit:	Jun-16-08 17:19 by ASC

Analyte	CAS Number	Concentration	Units
Phenol-d6	13127-88-3	200	ug/mL
o-Terphenyl	84-15-1	100	ug/mL
Nitrobenzene-d5	4165-60-0	104	ug/mL
Decafluorobiphenyl	434-90-2	100	ug/mL
2-Fluorophenol	367-12-4	202	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	201	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
7120351	Base Surrogate Stock	Dec-14-07	Brian J. Hall	Dec-14-08	Dec-14-07 11:52 by ASC	100
7120371	Acid Surrogate Stock	Dec-18-07	Andrea S. Colborn	Nov-10-08	Dec-18-07 08:40 by ASC	100

Approved By: MS 2-12-09 **Approved By:** BSH 2/12/09
QA Officer **Area Supervisor**

Attachment 20.3
Water Extraction Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Water Extraction Logbook

Date: 12/29/03

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
RMT	36416-13	352226 - 233	211038	8270BNA 3510	PL4.2-7	100µL	PL3.12-9	100µL
Rose Tap	29928-597	352327 - 328	"	8270BN 3510	"	"		
RMT	36416-13	352228 - 230	210615	8015 DRO 3510			GC2.41-16	500µL
				DJM 12/29/03				

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
352226	82	990	1.0mL	MBS					
352227	↓	1040	↓	↓					
352228	↓	1040	↓	↓					
352231	↓	1060	↓	↓					
352233	↓	1060	↓	↓					
MPB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
352327	82	1030	1.0mL	MBS					
352328	↓	1040	↓	↓					
352228	82	1020	1.0mL	MBS					
352230	↓	255	↓	↓					
MPB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
LFB129	↓	1000	↓	↓					
		DJM 12/29/03					DJM 12/29/03		

Reagent Information

CH_2Cl_2 Lot #:	030986	1:1 H_2SO_4 Reagent #:	PLR1.42-13
Hexane Lot #:	030702	1:1 HCl Reagent #:	NA
Na_2SO_4 Reagent #:	PLR1.42-16	10N NaOH Reagent #:	PLR1.44-11
		NaCl Reagent #:	NA

file: Prep Lab Water Logbook page: 3 of 30 revision: 0.0

Approved By: DD 2-12-09 QA Officer

Approved By: BJL+ 2/12/09 Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides, PCBs and Chlorinated Hydrocarbons from Water

SW-846 Method 3510C
EPA Method 608
EPA Method 612

APPROVALS:

Area Supervisor: _____ Date: 8/10/09
Brian J. Hall

QA Officer: _____ Date: 8-10-09
Tom C. Boocher

Operations Manager: _____ Date: 8/10/09
Jeff P. Glaser

Procedure Number: GR-09-107
Revision Number: 5.2

Date Initiated: 3/30/94
Effective Date: 8/12/09

Date Revised: 7/27/09
Pages Revised: All

By: Andrea S. Colborn
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure has detailed instructions for extracting organochlorine pesticides and polychlorinated biphenyls and chlorinated hydrocarbons from aqueous samples using methylene chloride as the extraction solvent. The procedure also describes the concentration technique suitable for preparing extracts for analysis.

1.2 This procedure is restricted to use by or under the supervision of a trained analyst. Each analyst must demonstrate the ability to generate acceptable results completion of a demonstration of capability study before actual sample extractions may be performed.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

2.2 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

2.3 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

3.0 SUMMARY OF PROCEDURE

3.1 A measured volume of sample, usually one liter, is serially extracted at neutral pH with methylene chloride using a 2 L glass separatory funnel.

3.2 Extracts are dried with sodium sulfate and concentrated with Kuderna-Danish concentration glassware.

3.3 Solvent exchange to hexane is performed and the extract is again concentrated to a final volume of 2.0 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 and SOP GR-03-128 for parameters applicable to this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

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5.4 TriMatrix SOP number GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-120, *Florisil/Silica Gel Column Cleanup of PCB's, Toxaphene, and Chlordane*, latest revision

5.6 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-16-100, *Glassware Cleaning and Preparation for the Organics Extraction Laboratory*, latest revision

5.9 TriMatrix SOP GR-09-111, *Florisil Column Cleanup,* latest revision

5.10 TriMatrix SOP GR-04-101, *semi-Volatile Organic Laboratory Corrective Actions*, latest revision

5.11 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware leading to discrete artifacts or elevated baselines. All materials must routinely demonstrate freedom from interferences by analysis of laboratory reagent blanks.

6.1.1 Glassware must be scrupulously cleaned in accordance with TriMatrix SOP GR-16-100.

6.1.2 Use only high purity reagents and pesticides-grade solvents to minimize interferences.

6.2 Matrix interference may be caused by contaminants co-extracted from samples. Matrix interference will vary considerably from matrix to matrix. Such interference can affect analyte recovery and re-extraction or other corrective action may be necessary if surrogates and/or other spiked compounds fail established laboratory recovery limits.

6.3 Phthalate esters cause a direct interference with analyte extractions. Avoid using flexible plastics in contact with solvent to minimize this type of interference.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the organic extractions laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

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7.4 The total toxicity and/or carcinogenicity of reagents used in this procedure have not been precisely defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce expose to the lowest possible level by adherence to established safety policies.

7.4.3 Material Safety Data Sheets are maintained on the laboratory intranet of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the attention of the Area Supervisor and/or Health and Safety Officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

8.2 All samples must be extracted within seven days of the collection date, and analyzed within 40 days of extraction except for method 608 PCB wastewater samples which have a year to extract.

8.3 When not in use, samples must be stored in the walk-in cooler, at 4 ±2° C.

8.4 Analysts must use care when handling sample containers to avoid loss due to breakage.

8.5 After extraction and/or cleanup, samples must be stored in the GC laboratory refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Erlenmeyer flasks: 300 mL

9.2 Separatory funnels: 2 L glass with PTFE stopcock and PTFE stopper

9.3 Filtering funnels: 100 mm

9.4 Filter paper: Fisher P8, coarse or equivalent

9.5 Kuderna-Danish (K-D) concentrator glassware

9.5.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.5.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

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9.5.3 Concentrator tube clips

9.5.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.5.5 Snyder columns: Two-ball micro

9.6 Auto-sampler vials: 4.0 mL with PTFE-lined screw cap lids, pre-calibrated to 2.0 mL as outlined in TriMatrix SOP GR-09-106.

9.7 Boiling chips: PTFE, methylene chloride rinsed

9.8 pH paper: wide-range (0-14)

9.9 Phase separation paper

9.10 N-EVAP nitrogen evaporation bath

9.11 Pasteur pipettes: 2 mL disposable, glass

9.12 Water bath: variable temperature, capable of maintaining temperature to ±5° C

9.13 Syringes: 100 µL, 500 µL, 1000 µL

9.14 Graduated cylinders: 1 L

9.15 Analytical Balance, capable of accurately weighing 0.0001 g

9.16 Hot Plate: capable of variable temperature control to ±5° C

9.17 Glass wool

9.18 Volumetric flasks: 10 mL, 50 mL, 100 mL

9.19 Vials: 40 mL amber, with PTFE-lined screw cap lids

9.20 Centrifuge: variable speed

9.21 Centrifuge tubes: 40 mL, PTFE

9.22 Sodium sulfate rinsing equipment:

9.22.1 Buchner funnel: 20 cm

9.22.2 Flask: 4 L Vacuum

9.22.3 Drying pan: 13 x 9 inch, metal

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9.22.4 Vacuum pump

9.22.5 Drying oven

9.22.6 Stopper: PTFE with hole for the Buchner funnel

9.22.7 Clamps: supporting

9.22.8 Filter paper: qualitative fast, 20 cm

9.22.9 Squirt bottle: PTFE

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above any reporting limit.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride to minimize interference from phthalate ester contamination:

11.1.2.1 Add 1000 mL of laboratory grade water to a 1000 mL separatory funnel

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

11.2 Methylene chloride: Pesticide grade or equivalent

11.3 Hexane: Pesticide grade or equivalent

11.4 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.4.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

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11.4.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.4.3 Add 1 L of methylene chloride to the glass sodium sulfate container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.4.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.4.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.4.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.4.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.4.8 After cooling, return to the original container using a glass funnel.

11.4.9 Label the container "Rinsed" with the organic preparation laboratory reagent identification (ID) number then place it in reagent storage.

11.5 Sodium Hydroxide solution (10N): Carefully, dissolve 400 g sodium hydroxide pellets in 1 L of reagent water.

CAUTION: This solution generates significant heat. Add pellets slowly and cool the solution before using.

11.6 Sulfuric acid solution (1:1 v/v): Carefully, add 500 mL concentrated sulfuric acid to 500 mL reagent water in a glass container.

CAUTION: This solution generates significant heat. Add acid slowly to the water and cool before using. NEVER add water to acid.

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions are made by the following rules:

12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.2 Use the appropriate solvent for dilution.

12.1.3 Label all glassware and vials holding surrogate/spiking solutions.

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12.1.4 Use minimal headspace in vials.

12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Using a microsyringe, inject the stock standard into a volumetric flask approximately half full of the appropriate solvent. For all standards, use only pesticide grade or better solvents. Make sure the syringe tip is below the solvent surface when injecting. Invert enough times to mix thoroughly. All surrogate and spiking standards are stored in 40 mL amber jars with PTFE-lined lids.

12.4 Surrogate/spike standards data is recorded in the extraction standards log. Surrogate/spiking solutions are stored in the organic extractions laboratory refrigerator at 4 ±2°C. A dilution of the spiking solution must be analyzed by the analytical laboratory to verify the concentration prior to use. The concentration recovered must be within 80 – 120%.

12.5 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If purity of a neat standard is below 95%, adjust the concentration in all subsequent calculations. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered before it runs out.

12.5.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Do not over-sonicate as excessive heat will be generated. Only after complete dissolution, dilute to volume and mix well by inverting at least three times. Discard the volume in the flask neck and transfer the rest to pre-labeled vials with minimal headspace. Store the vials at 4 ±2° C.

12.5.2 For neat liquids, weigh into a tared volumetric flask half-full of the appropriate solvent. Add the neat liquid drop-wise using a Pasteur pipette directly into the solvent, being careful not to touch the inside of the flask neck. Record the mass, swirl to mix then dilute to volume. Mix well and discard the volume in the flask neck. Transfer the rest to pre-labeled vials with minimal headspace and store at 4 ±2° C.

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12.6 The shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Dispose of any solution sooner if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor the expiration date of all chemicals and solutions. To avoid costly rush shipping, it is important to promptly order more chemical once consumed.

12.7 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

$$10000 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 100 \text{ mg/L}$$

$$100 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 1.0 \text{ mg/L}$$

12.8 When making a dilution, all data must be entered into the standards log in Element™ which is the laboratory information management system (LIMS) .

12.8.1 Refer to Attachment 20.2 for an example standards log entry.

12.8.2 Record each serial dilution made from a solution with a full explanation of what was done.

12.8.3 If a dilution of a dilution is made then the resultant solution would be given another Element™ number. This nomenclature is continued for each subsequent dilution.

12.8.4 For solution preparations, use only pesticide-grade or better solvents.

12.8.5 Before using a solution, the calculation performed when calculating concentrations must be verified by a second analyst. Once the calculation has been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within 80 – 120%, the solution may not be used.

12.9 Surrogate solution concentration:

12.9.1 Surrogates are made from purchased solutions containing both surrogates and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Concentration	Stock Dilution	Working Concentration
2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl	Absolute Standards (#20023)	Acetone	200 mg/L	100 µL:100 mL	0.20 mg/L

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12.10 Pesticide analysis spike solution concentration:

12.10.1 Spikes are made from purchased solutions and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration as surrogates since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
TCL Pesticides	Purchased	Methanol	1000 mg/L	80 µL:100 mL	0.80 mg/L

12.11 PCB analysis spike solution concentration:

12.11.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts a concentration of 0.50 mg/L.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
PCBs 1016, 1221, 1232, 1242 1248, 1254, 1260	NSI	Acetone	1000 mg/L	1:1000	1.0 mg/L

12.12 Chlorinated Hydrocarbon Spike Solution is made from purchased solution at vary concentrations:

Compound(s)	Source	Stock Conc.	Stock Dilution	Working Conc.
2-Chloronaphthalene	O_2SI	2500	20 µL:25 mL	2 mg/L
1,2,3-Trichlorobenzene	O_2SI	100	21 µL:25 mL	0.08 mg/L
1,2,4,5-Tetrachlorobenzene	O_2SI	100	22 µL:25 mL	0.08 mg/L
1,2,4-Trichlorobenzene	O_2SI	100	23 µL:25 mL	0.08 mg/L
1,2-Dichlorobenzene	O_2SI	2500	24 µL:25 mL	2 mg/L
1,3,5-Trichlorobenzene	O_2SI	100	25 µL:25 mL	0.08 mg/L
1,2,3,4-Tetrachlorobenzene	O_2SI	100	26 µL:25 mL	0.08 mg/L
1,4-Dichlorobenzene	O_2SI	2500	27 µL:25 mL	2 mg/L
Pentachloronitrobenzene	O_2SI	100	28 µL:25 mL	0.08 mg/L
Hexabromobenzene	O_2SI	100	29 µL:25 mL	0.08 mg/L
Hexachlorobenzene	O_2SI	100	30 µL:25 mL	0.08 mg/L
Hexachlorobutadiene	O_2SI	100	31 µL:25 mL	0.08 mg/L
Hexachlorocyclopentadiene	O_2SI	100	32 µL:25 mL	0.08 mg/L
Hexachloroethane	O_2SI	100	33 µL:25 mL	0.08 mg/L
Pentachlorobenzene	O_2SI	100	34 µL:25 mL	0.08 mg/L
1,3-Dichlorobenzene	O_2SI	2500	35 µL:25 mL	2 mg/L

13.0 ANALYTICAL PROCEDURE

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13.1 Assemble the glassware and samples.

13.1.1 Rinse all glassware with methylene chloride prior to use.

13.1.2 Label each separatory funnel, Erlenmeyer flask, K-D concentrator and concentrator tube with the following:

13.1.2.1 The sample ID number

13.1.2.2 The analysis

13.1.2.3 "S" for surrogate addition (Erlenmeyer flasks only)

13.1.2.4 "L" for spike addition, where applicable (Erlenmeyer flasks only)

13.1.2.5 The final extract volume and units

13.2 Determine the sample volume as follows:

13.2.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark (made before the sample was emptied) then measure with a graduated cylinder to attain the sample volume.

13.2.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE. After the sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.

Note: Any sample sediment suspected of containing PCBs and/or high concentrations of pesticides must be disposed of as hazardous waste in accordance with TriMatrix SOP GR-15-102.

13.2.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting since less than 500 mL will result in elevated reporting limits, which must be pre-approved by the client.

Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.

13.3 Extraction must be done at a pH of 5-9. Check the sample pH using wide-range pH paper by transferring a couple drops of sample onto the pH strip using a Pasteur pipette. Adjust the pH as necessary using sodium hydroxide or sulfuric acid solutions.

13.4 Add 1.0 mL of surrogate solution to each separatory funnel with a 1.0 mL microsyringe.

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13.4.1 Surrogate is added to all extractions.

13.4.2 Do not submerge the microsyringe needle into the liquid. Slowly inject approximately one inch above the liquid's surface.

13.4.3 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.

13.4.4 Cross out the "S" on each corresponding Erlenmeyer as surrogate is added.

13.5 Add 1.0 mL PCB spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for PCB analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.6 Add 100 μL pesticide spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for pesticides analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.7 Add 1.0 mL chlorinated hydrocarbons spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for chlorinated hydrocarbons analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.8 Rinse sample jars and graduated cylinders (when cylinders are used for individual samples) with methylene chloride as follows:

13.8.1 Add 100 mL methylene chloride to each sample jar. Replace the lid (very important).

13.8.2 Shake and vent as necessary.

13.8.3 Pour into the corresponding graduated cylinders and swirl to rinse the sides.

13.8.4 Finally, pour from the graduated cylinder into the separatory funnel containing sample.

13.9 Extract the quality control and samples as follows.

13.9.1 Stopper each separatory funnel and invert. Immediately open stopcock to release pressure.

13.9.2 Close the stopcock, shake and vent again.

13.9.3 Repeat until there is no evidence of pressure.

13.9.4 Shake vigorously for an additional two minutes.

13.9.5 Let the separatory funnel sit for at least ten minutes to allow phase separation.

13.9.6 If an emulsion forms during extraction:

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13.9.6.1 If the emulsion is minor drain the emulsion through phase separation paper into a 300 μL Erlenmeyer flask. When the phase separation paper is dripping slowly, rinse it with 20-30 mL of methylene chloride.

13.9.6.2 If separation does not occur and the emulsion looks like pudding, drain the entire contents of the emulsion into PTFE centrifuge tubes. Centrifuge for four minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom layer into a 300 mL Erlenmeyer flask.

13.9.7 If emulsion is minimal, drain the methylene chloride (bottom layer) directly into a 300 mL Erlenmeyer flask.

13.10 Repeat the extraction twice more with 60 mL aliquots of methylene chloride. Rinse the sample jars once more with the first of these solvent volumes.

13.11 Transfer the total extract volume to a K-D concentrator as follows:

13.11.1 Quantitatively transfer each extract from the 300 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into a properly labeled Kuderna-Danish (K-D) concentrators with an attached 10 mL graduated concentrator tube.

13.11.2 Rinse the flasks with several 20-30 mL aliquots of methylene chloride to complete the transfer.

13.12 Concentrate the extracts as follows:

13.12.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.12.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

13.12.3 Place the concentrators in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.12.4 At the proper rate of distillation, balls of each column will actively chatter but not flood. Until chattering begins, rattle Snyder columns periodically.

13.13 Solvent exchange to hexane:

13.13.1 When the apparent volume of liquid reaches 3-5 mL (in ten to fifteen minutes), remove the Snyder column and quickly add 40-60 mL of hexane and two new boiling chips to each concentrator and replace the Snyder column. Add about 10 mL more hexane to the top of the Snyder column. Increase the temperature of the bath to 90-95° C. Concentrate again to an apparent volume of 5 mL. Remove and cool for at least 10 minutes. The volume in the

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concentrator tube after cooling should be approximately 8-10 mL and appropriate for micro-concentration.

13.14 Micro-Snyder column technique is used to adjust the final extract volume to 2.0 mL. However, if using nitrogen blowdown instead of the micro-Snyder column, proceed to Section 13.4.2.

13.14.1 Micro-Snyder Technique:

13.14.1.1 Remove each concentrator tube and rinse the ground-glass joint with hexane.

13.14.1.2 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.14.1.3 Pre-wet the column with 0.5 mL hexane.

13.14.1.4 Place concentrator tubes in a water bath, partially immersed. The water temperature should be 90-95° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.

13.14.1.5 When the apparent volume reaches 1 mL, remove from the bath. Cool for at least ten minutes.

13.14.1.6 Rinse the ground-glass joints with 0.2 mL hexane after removing the Snyder column.

13.14.1.7 Transfer each extract to a 2.0 mL calibrated vial using a clean, disposable, Pasteur pipette.

13.14.1.8 Using approximately 0.8 mL of hexane, rinse each concentrator tube and transfer to the vial.

13.14.1.9 Adjust the final volume to 2.0 mL then cap tightly.

13.14.1.10 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.14.2 If using nitrogen blowdown instead of the micro-Snyder column, proceeed as follows:

13.14.2.1 Position sample bottles on the N-EVAP.

13.14.2.2 With a gentle stream of nitrogen, concentrate extracts to <u>not less than</u> 2.0 mL.

13.14.2.3 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.15 Extract cleanup

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13.15.1 If necessary, pesticide extracts may be cleaned up using Florisil columns (1000 mg or 20 g) in compliance with TriMatrix SOP GR-09-111.

13.15.2 All PCB extracts must be cleaned up as follows:

13.15.2.1 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.2.2 Copper powder to remove elemental sulfur, sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.15.3 If PCB extracts have color and/or do not appear to be free of contaminants after sulfuric acid and copper, then the extracts need cleaned up as follows and in the order listed:

13.15.3.1 Florisil/Silica gel column cleanup in compliance with TriMatrix SOP GR-09-120.

13.15.3.2 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.3.3 Sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.16 Store cleaned up extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

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15.1 An extraction blank (BLK) and blank spike (BS) must be done daily or for each extraction batch of up to 20 samples, whichever comes first, to demonstrate that extraction interferences are under control.

15.2 Matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 20 samples for analysis group or once a week, whichever is more frequent and provided enough sample is received. However, matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 10 samples for method 608 (primarily wastewater effluent samples) or once a week, whichever is more frequent and provided enough sample is received.

15.2.1 Performance records must be maintained to document the data quality generated.

15.2.2 Matrix spikes are extracted by measuring 1.0 mL of PCB spike solution (or 100 μL of pesticides spike solution) in addition to 1.0 mL of surrogate, to 1 L of sample (or 1.0 mL 8121 spiking solution).

15.3 Blank spikes (BS) are prepared by measuring 1.0 mL of PCB spike solution (or 100 μL of pesticides spike solution) in addition to 1.0 mL of surrogate, into 1 L of organic-free laboratory reagent water (or 1.0 mL 8121 spiking solution).

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation in accordance with TriMatrix SOP GR-04-101 may be necessary if surrogate and/or spiked compounds fail laboratory-established control limits.

15.5 Surrogates must be added to all extractions.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by successfully completing an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) is required annually.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare a spiking solution at analyte concentrations listed in Section 12.0. Prepare the spiking solution separately from the instrumental calibration standards. Spike 1.0 mL (100 μL for pesticides) of the solution into four, 1 L aliquots of water and extract following all steps in this procedure. After extracting, have the extracts analyzed then input the results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery as calculated from the spreadsheet must fall within the blank spike acceptability window listed in Element™. Standard deviation of the average recovery must be ≤20%.

16.1.1.2 If the study fails for either recovery or percent difference, locate and correct the source of the problem and repeat the study. If the second study passes, the analyst has demonstrated the procedure successfully. Repeated failure however, will

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indicate a procedure or technique problem. If this occurs, correct the technique or procedure and repeat the study successfully.

16.1.1.3 Samples may not be processed by any analyst or on any instrument until an IDC study has been successfully completed. Copies of successful demonstrations of capability spreadsheets and raw data must be provided to Quality Assurance (QA) for documentation of training.

16.1.2 Continuing Demonstration of Capability (CDC)

16.1.2.1 Annually, a CDC is required by each analyst. The CDC may be accomplished by repeating the IDC study, using the last four results from an exclusively run MDL study or by extracting a successful blind PT study sample. Input results to the same spreadsheet used for IDC studies.

16.2 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

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19.2 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

19.3 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Extraction Standards Log Entry Example

20.3 Organic Preparation Laboratory Waters Logbook Example

20.4 Addendum to the Procedure in Reference to Sample Batching

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Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0703579 Page 1 of 1

Printed: 4/9/2007 4:54:47PM

Semivolatiles GC, Waste Water, 608 Liquid/Liquid Extraction

Surrogate #1 = 7010436 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0704032	608 PCBs (std 7 aroclors)	0704054	608 PCBs (master list)	0704064	608 PCBs ([redacted])
0704064	608 PCBs (std 7 aroclors)	0704064	608 PCBs (master list)	0704071	608 PCBs ([redacted])
0704081	608 PCBs (std 7 aroclors)				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (mL)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0703579-BLK1		Apr-06-07 13:00	BJH	1000	2	1000				BLANK	
0703579-BS1		Apr-06-07 13:00	BJH	1000	2	1000		7010798	1000	LCS	
0703579-MS1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE	
0703579-MSD1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE DUP	
0704032-01	A	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	report MPB at 0.1ug/L; plz add for TOT
0704054-02	B	Apr-06-07 13:00	BJH	1000	2	1000				[redacted]	[redacted]
0704064-01	C	Apr-06-07 13:00	BJH	1010	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-04	E	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	
0704071-01	B	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	1254 only
0704081-01	C	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	
0704081-02	D	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: 8-10-09 **QA Officer** **Approved By:** BJH 8/10/09 **Area Supervisor**

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Attachment 20.2
Extraction Standards Log Entry Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7040565

Description:	1016/1260 Soil Spike	Expires:	Oct-16-07
Standard Type:	Analyte Spike	Prepared:	Apr-16-07
Solvent:	Hexane Lot #E08E21	Prepared By:	Brian J. Hall
Final Volume (mls)	100	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Apr-16-07 15:21 by BJH

Analyte	CAS Number	Concentration	Units
PCB-1016	12674-11-2	10	ug/mL
PCB-1016 [2C]	12674-11-2	10	ug/mL
PCB-1260	11096-82-5	10	ug/mL
PCB-1260 [2C]	11096-82-5	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A604465	(AMP) Aroclor 1016	May-12-06	** Vendor **	Sep-30-08	Apr-12-07 13:29 by JLW	1
A604466	(AMP) Aroclor 1260 stock	Oct-31-05	** Vendor **	Oct-31-08	Apr-12-07 13:35 by JLW	1

Reviewed By _____ Date _____

Page 1 of 1

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Approved By: [illegible] 8-10-09 **Approved By:** BJH 8/10/09
QA Officer **Area Supervisor**

Attachment 20.3
Organic Preparation Laboratory Waters Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Water Extraction Logbook

Date: 4/9/07

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
[redacted]	0704064	BLK, BS, MS, MSD 0704064-01-04	0703613	3510/8270	6120791	100 ul	7030610	100 ul
[redacted]	0704095	0704095-05-07, 09	"	"	"	"		
[redacted]	0704100	0704100-01	"	"	"	"		
[redacted]	0703391	BLK, BS, 0703391-06RE1	0703180	8151	A61008	100 ul	7030846	500 ul
ASC	4/9/07							

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
0703613BLK	70	1000	1.0	BJH					
0704064-01	↓	1010	↓	↓					
0704064-02		1050							
0704064-03		1060							
0704064-04		1060							
0703613MS		1050							
0703613MSD		1050					ASC 4/9/07		
0703613BS	↓	1000	↓	↓					
0704095-05	70	1060	1.0	BJH					
0704095-06	↓	1010	↓	↓					
0704095-07		920							
0704095-09	↓	970	↓	↓					
0704100-01	70	1060	1.0	BJH					
0703182BLK2	68	1000	5.0	ASC					
0703182RE1	↓	1030	↓	↓					
0703182BS2		1000							
		ASC 4/9/07							

Reagent Information

CH_2Cl_2 Lot #: C40467
Hexane Lot #: C50E54
Na_2SO_4 Reagent #: PLR2.3-2
1:1 H_2SO_4 Reagent #: PLR261-15
1:1 HCl Reagent #: NA
10N NaOH Reagent #: 7040188
NaCl Reagent #: NA

file: Prep Lab Water Logbook — page: 1 of 30 — revision: 0.0

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Approved By: [illegible] 8-10-09 QA Officer — Approved By: BJH 8/10/09 Area Supervisor

Attachment 20.4
Addendum to the Procedure in Reference to Sample Batching

Some clarification of method QC batching requirements and refinement of TriMatrix batching policies is necessary.

It is not permissible to extract more than 20 samples of the same method and matrix without the extraction of additional matrix QC samples.

To prevent this from happening, only one QC batch of a given method and matrix may be open at any one time. When reaching 20 samples or when a Level 3 project arrives, close the currently active batch and begin a new one. It does not matter if the current batch contains less than 20 samples. This policy will be applicable to all extracted samples with 2 exceptions:

#1) If multiple Level 3 and above clients come in on the same day, all of which have samples of the same matrix/method combination, it will be necessary to have more than one QC batch open. At the end of the day, close all the open batches except the one with the fewest samples. That one will remain open as the active batch.

#2) Certain clients send in samples over an extended time period. They also specify which of their samples are to be spiked. The selected sample does not always come in with the first round of samples received. For these clients and under these circumstances it will also be acceptable for multiple QC batches for the same method and matrix are open concurrently. Treat these batches as if they did not exist with regards to batching other samples.

Additionally, except for #2 above, batches may not be held open for longer than 7 days. After seven days close the active batch and begin a new one.

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Approved By: MB 8-10-09 **Approved By:** BJH 8/10/09
QA Officer **Area Supervisor**

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides and Polychlorinated Biphenyls from Soil, Sludge and Wipe Samples

SW-846 Method 3550C

APPROVALS:

Area Supervisor: Brian Hall — Date: 3/2/09

QA Officer: Tom C. Boocher — Date: 2-27-09

Operations Manager: Jeff P. Glaser — Date: 3/3/09

Procedure Number: GR-09-108
Revision Number: 4.3

Date Initiated: 3/30/94
Effective Date: 3/27/09

Date Revised: 2/26/09
Pages Revised: All

By: Tom C. Boocher
Total Number of Pages: 21

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of chlorinated pesticides and polychlorinated biphenyls (PCB) from soil, sludge, and wipes.

1.2 Sonication is used to ensure sufficient contact with extraction solvent and facilitate acceptable analyte recovery from most sample matrices. Sonication is not appropriate where extraction efficiencies for low analyte concentrations are required.

1.3 If necessary and when appropriate, extracts will be cleaned up after extraction and concentration.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

3.0 SUMMARY OF PROCEDURE

3.1 Extraction for analyte concentration at or below 20 mg/kg.

3.1.1 Anhydrous sodium sulfate is mixed with 30 g of sample to form a free-flowing solid.

3.1.2 Samples are serially extracted three times by sonication with 1:1 methylene chloride/acetone. Extracts are concentrated then solvent exchanged to hexane. Extracts are concentrated again to 10.0 mL for cleanup and/or GC analysis.

3.1.3 This technique is for low to medium analyte concentrations and negligible matrix interference. If necessary, final extract volumes may be greater than 10 mL or the following technique used.

3.2 Extraction procedure for analyte concentration above 20 mg/kg or difficult sample matrices

3.2.1 Samples with a history of high analyte concentration or matrix interference are extracted using 2.0 g of sample. Extraction analyst discretion based on experience is used to determine the need for this technique.

3.2.2 Anhydrous sodium sulfate is added to form a free-flowing solid.

3.2.3 Samples are extracted once by sonication with hexane. Extracts are filtered through glass wool and collected in a 15 mL vial for cleanup and/or gas chromatographic (GC) analysis.

3.3 Extraction procedure for wipes

3.3.1 Wipes are serially extracted three times by sonication with hexane in the sample container.

3.3.2 The hexane extract is concentrated to 10.0 mL for cleanup and/or GC analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 for an organochlorine pesticide list associated with this procedure.

4.2 Refer to TriMatrix SOP GR-03-128 for a polychlorinated biphenyl (PCB) list associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Semi-Volatile Laboratory Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Semi-Volatile Laboratory Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.4 TriMatrix SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-111, *Florisil® Column Cleanup*, latest revision

5.6 TriMatrix SOP GR-09-120, *Florisil®/Silica Gel Column Cleanup of PCBs, Toxaphene and Chlordane*, latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-09-128, *Soil Mixing and Grinding,* latest revision

5.9 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.10 TriMatrix SOP GR-16-100, *Equipment Cleaning and Preparation for the Organic Extraction Laboratory,* latest revision

5.11 TriMatrix SOP GR-04-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interference can be caused by contaminants in solvents, reagents, glassware, or sample processing equipment, leading to discrete artifacts or elevated baselines. All materials used in this procedure must routinely demonstrate to be free from interference by analysis of blank spikes (BS).

6.2 Use only reagent-grade or better reagents, and pesticide-grade or better solvents. Clean all glassware in accordance with TriMatrix SOP GR-16-100.

6.3 Phthalate esters cause interference with pesticide and PCB analysis. Avoid using flexible plastics in contact with solvent to minimize this type of contamination.

6.4 Matrix interferences can affect analyte recovery. Repeating an extraction and/or cleanup may be necessary if surrogates or other spiked compounds fail laboratory established control limits.

7.0 SAFETY PRECAUTIONS

7.1 Extraction personnel must wear laboratory coats and approved safety glasses while in the organic extractions laboratory area. Also, disposable gloves must be worn whenever samples, reagents and/or solvents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For waste disposal refer to TriMatrix SOP GR-15-102.

7.4 The toxicity and/or carcinogenicity of chemicals used in this procedure has not been fully defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce exposure to the lowest possible level by adherence to established safety practices.

7.4.3 A Material Safety Data Sheet (MSDS) is located on the laboratory intranet for all chemicals used in this procedure.

7.5 Bring safety issues to the attention of the Health and Safety Officer and Area Supervisor.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 60 mL, 125 mL or 250 mL wide-mouth glass jars with PTFE-lined screw-cap lids.

8.2 All samples must be extracted within fourteen days of the collection date and analyzed within 40 days of extraction.

8.3 When not in use, samples must be stored in the walk-in cooler at 4 ±2° C.

8.4 Use care when handling sample containers to avoid sample loss due to breakage.

8.5 After extraction and/or cleanup, store extracts in the GC refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Beakers, heavy-duty Pyrex: 400 mL and 600 mL

9.2 Ultrasonic disrupter:

9.2.1 Fisher model 550 with ¾ inch horn

9.2.2 For the high concentration extraction, use the 1/8 inch tapered microtip attached to a ½ inch horn

9.2.3 Sonabox, for hearing protection

9.3 Kuderna-Danish (K-D) concentrator glassware:

9.3.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.3.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.3.3 Concentrator tube clips

9.3.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.3.5 Snyder columns: Two-ball micro

9.4 Water bath: Variable temperature

9.5 Balance, top-loading, capable of accurately weighing to the nearest 0.01 g

9.6 Spatula, wooden tongue depressors

9.7 Pasteur pipets, disposable, 2 mL

9.8 Boiling chips, PTFE, methylene chloride rinsed

9.9 Vials (14 mL), with PTFE-lined screw-cap lids, calibrated to 10 mL

9.10 Filter paper, qualitative: Fisher P8

9.11 N-EVAP concentrator

9.12 Syringes, micro: 100 uL, 500 µL, and 1000 µL

9.13 Aluminum foil

9.14 Filtration apparatus

9.14.1 Filter flask: 500 mL side-arm

9.14.2 Buchner funnel, 90 mm

9.14.3 Stopper: PTFE, holed

9.14.4 Glass-fiber filter paper for clay samples or samples with fine particulate

9.15 Filter funnel: 100 mm, ribbed

9.16 Sodium sulfate rinsing equipment:

9.16.1 Buchner funnel: 20 cm

9.16.2 Flask: 4 L vacuum

9.16.3 Drying pan: 13 x 9 inch, metal

9.16.4 Vacuum pump

9.16.5 Drying oven

9.16.6 Stopper, PTFE with hole for the Buchner funnel

9.16.7 Clamps, supporting

9.16.8 Filter paper, qualitative fast, 20 cm

9.16.9 Squirt bottle, PTFE, labeled with contents

9.17 Hot Plate: capable of variable temperature control to within ±5° C

9.18 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.19 Amber vials, with PTFE-lined screw-cap lids, 40 mL

9.20 Analytical balances, capable of accurate measurement to the nearest 0.0001g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Disrupters must be tuned at the beginning of each shift.

10.2 Ultrasonic probes must be cleaned before extracting each new sample and thoroughly cleaned at the end of each shift.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene chloride/acetone, 1:1 (v/v): pesticides grade or better

11.2 Methylene chloride: pesticides grade or better

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.3.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.7 After cooling, return to the original container using a glass funnel.

11.3.8 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.

11.4 Hexane: Pesticides grade or better

11.5 Acetone: Pesticides grade or better

11.6 Each lot of methylene chloride, hexane and acetone must be tested before use to show freedom from interference. Submit test results to the quality assurance department for scanning into the certification of analysis file.

12.0 STANDARDS PREPARATION

12.1 Surrogates and spike solutions prepared from neat materials are made by the following rules:

12.1.1 Obtain an analytical balance that weighs to 0.0001 g. Be sure to record mass to the nearest 0.0001 g.

12.1.2 Place the balance in a fume hood and re-calibrate it.

12.1.3 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.4 Use the required solvent for dilutions.

12.1.5 Minimize headspace in storage vials.

12.2 Store surrogate and spiking solutions in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 If solid surrogate or spike compounds are weighed into a volumetric flask and the mass recorded, add enough solvent to dissolve then dilute to volume and invert several times to mix thoroughly.

Note: If solids do not dissolve, place in a small ultrasonic bath to aid in mixing. Do not leave for an extended time because the bath generates heat. After sonication, dilute to volume then transfer to a tightly capped pre-labeled vial and store at 4 ±2° C.

12.4 Weigh liquid surrogate or spike compounds into volumetric flasks containing approximately half-volume of solvent. Use a Pasteur pipet to add compound drop-wise, directly into the solvent. Be careful not to touch the inside of the flask or the solvent surface with the pipet tip. Record the mass and dilute to volume. Invert enough times to mix thoroughly then transfer to pre-labeled, tightly capped vials and store at 4 ±2° C.

12.5 Record surrogate/spike data in an extraction standards log (Attachment 20.1). Store surrogate/spiking solutions in the refrigerator at 4 ±2° C. A dilution of spiking solution must be analyzed by GC/MS and/or GC to check concentration prior to use by the extractions laboratory. Concentration must be within 80 – 120% and hardcopy submitted to the quality assurance department.

12.6 Neat compounds must be ACS grade or better. If the purity of a neat standard is below 95%, the concentration must be accounted for in all subsequent analytical calculations. Do not use any neat compound past its expiration date. If using the last of a compound, promptly re-order.

12.7 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, remove from the laboratory for disposal. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.8 When making surrogate and spiking solutions, serial dilutions are often required to achieve the proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

12.8.1 Using a micro-syringe, inject 1.0 mL of 10,000 mg/L solution into a 100 mL volumetric flask approximately half full of the appropriate solvent. Make sure the syringe needle tip is below the solvent surface. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in the flask is 100 mg/L.

12.8.2 Inject 1.0 mL of the 100 mg/L solution into another volumetric flask half filled with solvent after rinsing the syringe thoroughly with clean solvent. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in this second flask is 1.0 mg/L.

12.8.3 Concentration in the second flask is determined by the following calculation:

$$10000\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 100\ \text{mg/L}$$

$$100\ \text{mg/L} \times \frac{1\ \text{mL stock standard}}{100\ \text{mL final volume}} = 1.0\ \text{mg/L}$$

12.9 When making a dilution, all data must be entered into the laboratory information management system (Element™). Enter each subsequent dilution in a dilution series separately. Use only pesticide grade (or better) solvents to dilute with.

12.10 Before using a laboratory-prepared solution, verify the calculations used by a second analyst. Once calculations have been verified, verify the solution concentration by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a prepared solution cannot be used. Acceptance limits are 80 – 120% of expected value.

12.11 Dispose of vials containing 5 mL or less of a prepared solution at the end of the shift to minimize contamination and analyte concentration. When not in use, keep spike solution vials in the organic extractions refrigerator.

12.12 Surrogates are normally made from purchased solutions and diluted in acetone to make one 200 mL volume. Check by GC before using. If all surrogates in a spiking solution are within 80-120% of the expected value, the solution is approved for use. Remaining individual surrogate stock solutions are used to calibrate the GC.

Surrogate	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Working Concentration (mg/L)
2,4,5,6-Tetrachloro-m-xylene	200	1:200	1
Decachlorobiphenyl	200	1:200	1

12.13 Pesticide analysis spike solution concentration:

12.13.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L in hexane. Samples are spiked with 500 µL of the dilution to give extracts the same concentration as surrogates since final extract volume is 10.0 mL.

Pesticide Stock	Purchased Concentration	Dilution	Final Concentration
Purchased Mixture	1000 mg/L	80 µL/100 mL	0.8 mg/L

12.14 PCB spiking solutions are made from individually purchased Aroclors and diluted in hexane to make 100 mL working solutions, which are checked by GC before using. If the Aroclor in a working solution is within 80 - 120% of expected value when tested, the solution is approved for use. Stock solutions are also used to calibrate the GC. PCB spiking solutions are prepared by the GC analyst.

Aroclor	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Concentration (mg/L)
1221	1000	1:100	10
1242	1000	1:100	10
1248	1000	1:100	10
1254	1000	1:100	10
1260	1000	1:100	10

13.0 ANALYTICAL PROCEDURE

13.1 The Fisher Model 550 ultrasonic disrupter must be tuned at the beginning of each shift, before samples are processed:

13.1.1 Turn the **OUTPUT CONTROL** knob counterclockwise to zero.

13.1.2 Press the **POWER SWITCH** to **ON** (up position). The switch will light up.

13.1.3 When the prompt appears, press **TUNE**. The display will read: [**TUNING - - - PROBE ACTIVE**].

13.1.4 Turn the **OUTPUT CONTROL** knob towards setting 10 (5 if using a microtip).

13.1.4.1 Note the position of the Bar Graph on the display screen. Do NOT exceed 70%.

13.1.4.2 Rotate the **OUTPUT CONTROL** knob clockwise or counterclockwise until a minimum (not maximum) reading is obtained. If a reading of less than 20% cannot be obtained, there is a problem with the disrupter and it must be repaired before using.

13.1.5 Press the **TUNE** key to display prompts for pulse or continuous operation and set the disrupter for pulse operation.

13.1.6 The disrupter is now tuned. Record all tuning information in the disrupter tuning logbook (Attachment 20.2).

13.2 Prior to sonication, homogenize all samples in accordance with the instructions outlined in TriMatrix SOP GR-09-128.

13.3 Label all glassware with sample number and other necessary information including MS, MSD, BS or BLK designations.

13.4 The low concentration extraction is as follows:

13.4.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.4.2 For sandy to dry samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 20 g to 25 g of anhydrous sodium sulfate. Mix well.

13.4.3 For wet to gummy samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 55 g to 60 g of anhydrous sodium sulfate. Mix well.

13.4.4 Samples should have a sandy texture after adding sodium sulfate. Add more sodium sulfate as necessary to achieve this sandy texture.

13.4.5 Measure 1.0 mL of surrogate into all samples and quality control extractions.

13.4.6 For matrix and blank spikes, add 0.5 mL of pesticides <u>or</u> 0.5 mL of PCB spiking solution.

13.4.7 Measure 100 mL of 1:1 methylene chloride/acetone into the beaker.

13.4.8 Sonicate by positioning the 3/4 inch sonication horn about 1/2 inch below the solvent surface without touching any sample solid or the beaker wall.

13.4.9 Sonicate for three minutes at full power (**OUTPUT CONTROL** knob set at 10 and **PERCENT-DUTY CYCLE** set at 50%). Do not use the microtip horn.

13.4.10 Next, assemble a K-D flask for each sample extracted to receive the extraction solvent.

13.4.11 Attach a 10 mL concentrator tube to the flask bottom.

13.4.12 Line 100 mm filter funnels with P8 filter paper then add approximately 1 inch of anhydrous sodium sulfate.

13.4.13 Decant the extraction solvent into the K-D flask through the funnel. Repeat with a different K-D setup for each sample extracted.

13.4.14 If a sample contains very fine particulate that plugs the filter paper, reweigh the sample and filter with a vacuum filter instead.

13.4.14.1 Assemble the vacuum filtration apparatus consisting of a 500 mL side-arm flask, 90 mm Buchner funnel and vacuum pump.

13.4.14.2 Filter using glass fiber filter paper under vacuum and transfer to a K-D flask for concentration.

13.4.15 Repeat the extraction twice more with additional 100 mL aliquots of 1:1 methylene chloride/acetone, decanting off the extraction solvent after each sonication. Repeat for each sample extracted until each sample has been extracted three times and the extract filtered.

13.4.16 If after the third sonication, there is no noticeable reduction in solvent color, up to two additional aliquots of methylene chloride/acetone may be used. Record the total number of aliquots used if more than 3.

13.4.17 After the final sonication, decant the solvent then transfer all solids into the filter funnel and quantitatively rinse with methylene chloride.

13.5 The high concentration extraction is as follows:

13.5.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.5.2 Weigh 2 ±0.1 g of sample into a clean, unused 20 mL vial.

13.5.3 Add 2 g anhydrous sodium sulfate to sample and mix well until free-flowing.

13.5.4 Add 1.0 mL surrogate to samples, blanks and spikes.

13.5.5 For pesticides analysis, add 0.5 mL of pesticide spike solution and 8.5 mL hexane to matrix spikes and laboratory fortified blanks. For PCBs analysis, add 0.5 mL of PCB spike solution and 8.5 mL hexane. The total volume added must be 10.0 mL.

13.5.6 Sonicate using the 1/8 inch tapered microtip horn for two minutes at **OUTPUT CONTROL** setting 5 in **PULSE MODE** and **PERCENT-DUTY CYCLE** set at 50%.

13.5.7 Filter the extract through glass wool and collect in a pre-calibrated 15 mL vial. Assume a final volume of 10 mL even though not all of the extract will be recovered. Do NOT bring to volume in the pre-calibrated vial.

13.5.8 Cap the vial tightly and store at 4 ±2° C until cleanup and/or analysis.

13.5.9 If cleanup is required, proceed to Section 13.9.

13.6 Extraction of wipe samples is as follows:

13.6.1 Measure 1.0 mL of surrogate solution into the jar a wipe sample arrives in. Do not remove any sample material from the jar. For extraction blanks and blank spikes, use a 400 mL beaker containing 30.0 g of sodium sulfate.

13.6.2 Add 0.5 mL of pesticide or 0.5 mL of PCB spike solution to matrix and blank spikes, depending upon the analysis required.

13.6.3 Add approximately 50 mL of hexane to each jar. The wipe sample must be totally covered by the solvent.

13.6.4 Position the 3/4 inch horn in the sample jar approximately 1/2 inch below the solvent surface, above the wipe.

13.6.5 Sonicate for three minutes at full power (**OUTPUT CONTROL** at ten and **PERCENT-DUTY** cycle at 50%).

13.6.6 Repeat Sections 13.4.10 – 13.4.19, except use hexane instead of 1:1 methylene chloride/acetone and rinse with hexane instead of methylene chloride.

13.6.7 Repeat for each sample needing extracted.

13.7 Concentrate extracts as follows:

13.7.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.7.2 Pre-wet the Snyder column by adding approximately 1 mL of methylene chloride through the top.

13.7.3 Place the concentrator in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.7.4 At the proper rate of distillation, the glass column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder column periodically.

13.7.5 Concentrate to an apparent volume of 3 mL. The concentration takes ten to fifteen minutes for low concentration extracts and five to ten minutes for high concentration extracts).

Note: When solvent volume is reduced below 1 mL using the K-D flask, semi-volatile analytes can be lost. If extracts are concentrated to dryness or near-dryness, the entire extraction must be repeated.

13.8 Perform the hexane solvent exchange as follows:

13.8.1 Remove and cool. The volume in the concentrator tube after cooling should be approximately 8 mL. Add 50 mL of hexane, wet the Snyder column and evaporate as before.

13.8.2 Transfer the extract to a 14 mL vial calibrated to 10 mL, rinsing the concentrator tube with several aliquots of hexane, totaling approximately 2 mL. Use only enough to bring the final volume to 10.0 mL. Repeat for each sample extracted.

13.9 Extract Cleanup

13.9.1 If necessary, pesticide extracts may be cleaned up using Florisil® columns (1000 mg or 20 g) as described in TriMatrix SOP GR-09-111.

13.9.2 All PCB extracts are cleaned up with sulfuric acid (TriMatrix SOP GR-09-110), and copper (TriMatrix SOP GR-09-109). If necessary, they are further cleaned up with Florisil® (TriMatrix SOP GR-09-111) or Florisil®/silica Gel (TriMatrix SOP GR-09-120).

13.10 Store finished extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for sample documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing all paperwork accurately. This is mandatory for quality control and to provide clients with defensible data.

14.2 Analysts extracting a batch of up to 20 samples must input all extraction data to Element™.

14.3 Benchsheets and logbooks must be filled in completely to ensure that results are reported correctly and data is associated with the right quality control batch (Attachment 20.3).

14.4 If an internal chain-of-custody report is required, it is important the form be filled in accurately and completely.

14.5 All extraction laboratory hardcopy must be archived appropriately.

14.6 All extraction logbooks must be filled in completely and correctly (Attachment 20.4). Corrections must be made with one line through the error, dated and initials then the correction to the side. Do not perform a write-over or obliterate the error. "Z" out blank areas in logbooks.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK) and blank spike (BS) daily or once per shift to demonstrate that interferences and contamination are under control. Extract a replicate blank spike when insufficient sample volume is received to perform a matrix spike duplicate.

15.2 Blank spikes are prepared by adding 0.5 mL of spike solution and 1.0 mL surrogate spike solution to 30 g of sodium sulfate. Always add 1.0 mL of surrogate solution to blanks.

15.3 An extraction batch is limited to no more than 20 samples.

15.4 When sufficient sample volume is received, extract a matrix spike (MS) and matrix spike duplicate (MSD) with every batch of up to 20 samples.

15.4.1 Prepare a matrix spike by measuring 0.5 mL of PCB spike solution or 0.5 mL of pesticide spike solution to 30 g of sample. Extract a matrix spike in accordance with every stop in the extraction procedure.

15.4.2 Always add 1.0 mL of surrogate to matrix spikes before extraction.

15.5 Sample matrix interference or laboratory-induced contamination can affect analyte recovery. Investigation and re-extraction may be necessary if surrogate and/or spiked compounds fail to pass laboratory established acceptance limits.

15.6 Perform corrective action for out-of-control quality control samples in accordance with TriMatrix SOP GR-04-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting an initial demonstration of capability (IDC) study.

16.2 Prepare a spiking solution to give a final extract concentration mid-range in the GC calibration. Prepare independently from the calibration. Prepare four 30 g aliquots of sodium sulfate then measure spiking solution to each. Extract as normal samples in accordance with all extraction steps in the procedure and all subsequent cleanup steps.

16.3 After extraction and cleanup, have the four extracts analyzed by TriMatrix SOP GR-03-128 or GR-03-120. Whichever is appropriate to the spiked analyte.

16.4 Input results to the IDC spreadsheet (located on the laboratory intranet library) to calculate average recovery and relative standard deviation.

16.4.1 Recovery must be within laboratory established control limits and relative standard deviation must be less than or equal to 20%. If all analytes and criteria are acceptable, the IDC study is complete. The analyst is authorized to extract samples by this procedure.

16.4.2 If one or more analytes fail, the analyst must proceed according through the following steps.

16.5 If any analyte fails, locate and correct the source of error then repeat the study successfully for the failed analyte.

16.6 Repeated failure will indicate a problem with the procedure and/or analyst technique. Locate the problem and correct the procedure and/or techniques used then repeat the IDC study successfully.

16.7 Samples may not be prepared by the analyst until an IDC study has been successfully completed.

16.8 Repeat the demonstration of capability study annually as a continuing demonstration of capability (CDC) by one of the follow approaches:

16.8.1 Repeat the initial demonstration of capability study.

16.8.2 Use the last four results from an MDL study run exclusively by the analyst. Input to the IDC spreadsheet and submit as the CDC.

Note: Results might be abnormal or unacceptable because of the low concentration. If so, repeat the study with a higher concentration of spikes.

16.8.3 Successfully complete a blind performance testing sample extracted during the course of routine sample extraction. Extraction of the sample must have been done by the analyst.

16.8.4 Use four consecutively extracted blank spikes extracted during the course of routine sample preparation. Extraction must have been done by the analyst.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.

18.3 Follow all instructions in SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

20.0 ATTACHMENTS

20.1 Standards Log Example

20.2 Disrupter Tuning Logbook Example

20.3 Preparation Batch Report Example

20.4 Extraction Logbook Example

Attachment 20.1
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

Description	Chlorinated Pesticide Spike	Expires	Sep-12-07
Standard Type	Analyte Spike	Prepared	Mar-12-07
Solvent	MeOH Lot#044685	Prepared By	Andrea S. Colborn
Final Volume (mls)	100	Department	Semivolatiles GC
Vials	1	Last Edit	Apr-20-07 09:54 by JLW

Analyte	CAS Number	Concentration	Units
4,4'-DDD	72-54-8	0.8	ug/mL
4,4'-DDD [2C]	72-54-8	0.8	ug/mL
4,4'-DDE	72-55-9	0.8	ug/mL
4,4'-DDE [2C]	72-55-9	0.8	ug/mL
4,4'-DDT	50-29-3	0.8	ug/mL
4,4'-DDT [2C]	50-29-3	0.8	ug/mL
Aldrin	309-00-2	0.8	ug/mL
Aldrin [2C]	309-00-2	0.8	ug/mL
alpha-BHC	319-84-6	0.8	ug/mL
alpha-BHC [2C]	319-84-6	0.8	ug/mL
alpha-Chlordane	5103-71-9	0.8	ug/mL
alpha-Chlordane [2C]	5103-71-9	0.8	ug/mL
beta-BHC	319-85-7	0.8	ug/mL
beta-BHC [2C]	319-85-7	0.8	ug/mL
delta-BHC	319-86-8	0.8	ug/mL
delta-BHC [2C]	319-86-8	0.8	ug/mL
Dieldrin	60-57-1	0.8	ug/mL
Dieldrin [2C]	60-57-1	0.8	ug/mL
Endosulfan I	959-98-8	0.8	ug/mL
Endosulfan I [2C]	959-98-8	0.8	ug/mL
Endosulfan II	33213-65-9	0.8	ug/mL
Endosulfan II [2C]	33213-65-9	0.8	ug/mL
Endosulfan Sulfate	1031-07-8	0.8	ug/mL
Endosulfan Sulfate [2C]	1031-07-8	0.8	ug/mL
Endrin	72-20-8	0.8	ug/mL
Endrin [2C]	72-20-8	0.8	ug/mL
Endrin Aldehyde	7421-93-4	0.8	ug/mL
Endrin Aldehyde [2C]	7421-93-4	0.8	ug/mL
Endrin Ketone	53494-70-5	0.8	ug/mL
Endrin Ketone [2C]	53494-70-5	0.8	ug/mL

Reviewed By _____ Date _____

Page 1 of 2

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

gamma-BHC (Lindane)	58-89-9	0.8	ug/mL
gamma-BHC (Lindane) [2C]	58-89-9	0.8	ug/mL
gamma-Chlordane	5103-74-2	0.8	ug/mL
gamma-Chlordane [2C]	5103-74-2	0.8	ug/mL
Heptachlor	76-44-8	0.8	ug/mL
Heptachlor [2C]	76-44-8	0.8	ug/mL
Heptachlor Epoxide	1024-57-3	0.8	ug/mL
Heptachlor Epoxide [2C]	1024-57-3	0.8	ug/mL
Methoxychlor	72-43-5	0.8	ug/mL
Methoxychlor [2C]	72-43-5	0.8	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A603424	(AMP) Organochlorine Pesticides	Feb-28-06	** Vendor **	Feb-29-08	Apr-20-07 09:54 by JLW	0.08

Reviewed By _____ Date _____

Page 2 of 2

Approved By: MB 3-2-09
QA Officer

Approved By: BJH 3/2/09
Area Supervisor

Attachment 20.2
Disrupter Tuning Logbook Example

Date	Analyst	Fisher Scientific 550 Inst. #1 Final Tuner Reading	Pass / Fail (<20%)	Fisher Scientific 550 Inst. #2 Final Tuner Reading	Pass / Fail (<20%)
7/16/02	[illegible]	4%	Pass / Fail	8%	Pass / Fail
7/17/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/18/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/19/02	JEO	4%	Pass / Fail	8%	Pass / Fail
7/22/02	[illegible]	[illegible]	Pass / Fail	[illegible]	Pass / Fail
7/23/02	DGM	5%	Pass / Fail	9%	Pass / Fail
7/24/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/25/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/26/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/27/02	DGM	4%	Pass / Fail	8%	Pass / Fail
7/29/02	DGM	5%	Pass / Fail	8%	Pass / Fail
7/30/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/1/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/5/02	[illegible]	[illegible]	Pass / Fail	5%	Pass / Fail
8/8/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/9/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/12/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/13/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/14/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/15/02	DGM	5%	Pass / Fail	8%	Pass / Fail
8/16/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/20/02	DGM	[illegible]	Pass / Fail	[illegible]	Pass / Fail
8/21/02	DGM	[illegible]	Pass / Fail	[illegible]	Pass / Fail
8/22/02	DGM	5%	Pass / Fail	8%	Pass / Fail
8/23/02	DGM	[illegible]	Pass / Fail	[illegible]	Pass / Fail
8/24/02	DGM	4%	Pass / Fail	8%	Pass / Fail
8/25/02	DGM	5%	Pass / Fail	7%	Pass / Fail
8/26/02	DGM	[illegible]	Pass / Fail	[illegible]	Pass / Fail
8/27/02	DGM	4%	Pass / Fail	8%	Pass / Fail
8/28/02	DGM	4%	Pass / Fail	8%	Pass / Fail

file: soniclog page: 16 of 50 revision: 1.0

Approved By: [illegible] 3-2-09
QA Officer

Approved By: BSH 3/2/09
Area Supervisor

Attachment 20.3
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0709022 Page 1 of 1

Printed: 9/21/2007 5:34:48PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Surrogate #1 = 7070018 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0708116	8081A PESTs (master list)	0708257	8081A PESTs (master list)	0708330	8081A PESTs (master list)
0708338	8081A PESTs (master list)				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0709022-BLK1		Aug-08-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0709022-BSD1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS DUP	
0708116-01	C	Aug-08-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK2		Aug-15-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS2		Aug-15-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708257-01	C	Aug-15-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK3		Aug-21-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS3		Aug-21-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708330-04	A	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-01	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-02	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]

Comments:

Analyst Initials:

bch_TriMatrix.rpt

Approved By: M 3-2-09
QA Officer

Approved By: BJH 3/2/09
Area Supervisor

Attachment 20.4
Extractions Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Soil Extraction Logbook

Date: 3/11/04

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
EQ	36357-12	355751-756	212470	8082 PCB 3550	PL4.1-10	1.0mL	PL4.8-2	500mL
SAIC	36278-43	356038-046	213415	8330 EXP	PL3.12-2	500 mL	PL4.2-13	1.0 mL
MDL				Full List Plus	PL4.2-7	100 mL	PL3.12-12 PL3.11-12	5.0mL 1mL

PAK 3/11/04

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials
MPB111	90	30.0	10.0	MBS	MDL 1	80	30.0	1.0	MBS
355751		30.0			MDL 2		30.0		
355752		30.0			MDL 3		30.0		
355753		30.0			MDL 4		30.0		
355754		30.0			MDL 5		30.0		
355755		30.0			MDL 6		30.0		
355756		30.0			MDL 7		30.0		
MPB111	NA	2.0	10.0	DJM					
356038		2.0							
356039		2.0							
356040		2.0							
356041		2.0							
356042		2.0							
356043		2.0							
356044		2.0							
356045		2.0							
356046		2.0							
356042MS		2.0							
356042MSD		2.0							
LFB111		2.0							

PAK 3/11/04

PAK 3/11/04

Reagent Information

CH_2Cl_2 Lot #: 030986
Hexane Lot #: 030702
Acetonitrile Lot #: Y30819
1:1 CH_2Cl_2/Acetone Reagent #: PLR1.48-7
Na_2SO_4 Reagent #: PLR1.51-2
Other #: NA

file: Prep Lab Soil Logbook page: 4 of 20 revision: 0.0

Approved By: 3-2-09 QA Officer

Approved By: BJH 3/2/09 Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Chain-of-Custody (COC)

APPROVALS:

Laboratory Manager: _____ Date: 12/21/06
Douglas E. Kriscunas

QA Manager: _____ Date: 12/21/06
Rick D. Wilburn

Operations Manager: _____ Date: 12/21/06
Jeff P. Glaser

Procedure Number: GR-10-104
Revision Number: 2.2

Date Initiated: 8/30/96
Effective Date: 12/15/06

Date Revised: 11/30/06
Pages Revised: All

By: Rick D. Wilburn

Total Number of Pages: 9

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4/6/09		4/6/10

1.0 SUMMARY OF PROCEDURE

1.1 This procedure establishes uniform policies for a legally defensible internal chain-of-custody (COC) system for samples requiring defined levels of security.

1.2 Following these guidelines will provide unbroken sample tracking from the client source, through the analytical process and to final sample disposition.

1.3 There are two approaches to maintaining sample custody following receipt by the laboratory and the initial transfer of internal custody as follows:

1.3.1 The first approach is used for samples not associated with a project where litigation is expected or likely to be needed, or is requested by the client. In such a case, the project chemist designates in the laboratory information management system (Element™) that project samples are handled according to an "external" or general sample custody protocol.

1.3.2 The second approach is used for samples associated with a project where litigation is expected or likely to be needed, or is requested by the client. In such a case, the project chemist designates in the laboratory information management system (Element™) that project samples are handled according to an "internal" or specific sample custody protocol.

1.4 This procedure also includes general security measures taken to ensure laboratory integrity is maintained in each analytical area:

2.0 DETAILED PROCEDURE

2.1 External COC is defined and adhered to as follows:

2.1.1 General building security measures and sample or information access by authorized personnel only must remain in effect at all times.

2.1.2 All laboratory records (paper and electronic), must be maintained in such a way as to minimize physical deterioration, be available and identifiable.

2.1.3 All laboratory records (paper and electronic) must identify the person or persons responsible for information entered which permits the event and chronological reconstruction of TriMatrix Laboratory's involvement with any sample in direct connection with the client report.

2.1.4 Records (both on-site and off-site) include but are not necessarily limited to the following:

2.1.4.1 Sample receipt forms

2.1.4.2 Sample storage information

2.1.4.3 Sample preparation and/or analysis data printouts

2.1.4.4 Data reduction

2.1.4.5 Client correspondence

2.1.4.6 Preliminary and final reports

2.2 Internal COC is defined and adhered to as follows:

2.2.1 In addition to external COC, internal chain-of-custody establishes an intact, continuous record of all physical possession, storage and disposal of sample containers, collected samples, sample aliquots and sample extracts/digestates. Any form of which can be referred to as the "sample".

2.2.2 Internal chain-of-custody must be maintained by one of the following criteria at all times:

2.2.2.1 The sample is in actual physical possession by a laboratory employee.

2.2.2.2 The sample is in constant view of the laboratory employee responsible for physical possession of the sample.

2.2.2.3 The sample is stored in a secure area of the laboratory restricted to authorized personnel only.

2.2.3 An internal COC record must identify all individuals who physically handle a sample. Refer to Attachment 6.12 for an example internal COC form that is automatically generated by the Laboratory Information Management System (Element™) when the internal COC is required.

2.2.4 The internal COC record must account for all times and events associated with sample handling and/or movement and must begin at the point established by the applicable federal or state oversight program. Internal COC typically begins at sample collection. However, it may begin at the time clean sample containers are provided by the laboratory.

2.2.5 For projects in which sample containers originate from TriMatrix Laboratories, the internal COC form must accompany containers during transport to the collection site and be returned when collected samples are returned to the laboratory.

2.2.6 Shipping cooler custody seals and/or individual sample containers (if affixed by the client) are to be submitted. Receipt of a cooler without an intact seal must be documented on the sample receipt checklist.

2.2.7 Sample login staff are responsible for maintaining sample storage areas and related receipt documentation which are part of an internal COC project. In the event a sample login technician is not available when samples arrive (such as, after normal business hours), the following persons must assume responsibility for initiating internal COC:

2.2.7.1 Any Laboratory analyst or technician
2.2.7.2 Any Project chemist

2.3 A COC sequence of events is as follows:

2.3.1 Upon receipt and proper acknowledgment of coolers containing samples designated for internal COC, sample technicians must perform a thorough inspection of any and/or all custody seals. Should any seal arrive damaged, the project chemist and/or client must be immediately notified.

2.3.2 All paperwork accompanying sample coolers must be retrieved and checked for completeness and accuracy. Any discrepancies between received samples and the paperwork or any project information must be noted and recorded. Inform the project chemist of all COC discrepancies. Document the discrepancy and take or suggest all appropriate corrective action to minimize a repeat discrepancy.

2.3.3 Only when samples have been properly accounted for and any discrepancies resolved may they be logged into the laboratory information management system (Element™). During log-in, the option to designate samples as internal COC must be selected. This initiates the creation of the COC form for each analysis/sample (Attachment 6.1) which must be signed with each change in location and/or custody.

2.3.4 After log-in, samples must be moved to a designated storage area (Attachment 6.2). Access to all sample storage areas is limited to employees only for security reasons.

2.3.5 Chain-of-custody forms are placed in folders located in the laboratory by the walk-in cooler. The technician logging in samples must ensure each sample has the appropriate type and number of sign-out sheets.

2.3.6 Samples requiring internal COC status are communicated to laboratory areas through arrival logs, worklists and benchsheets. These forms are generated by Element™. Area supervisors and group leaders must ensure all chemists and technicians are aware of any and all internal COC samples. The signing of COC forms must be strictly and consistently done. Any technician or analyst not completing the COC form will be reprimanded accordingly.

2.3.7 When preparing to analyze internal COC samples, the COC form must be completely filled in with the removal time, analyst's name and the return time.

2.3.7.1 Internal COC forms must be filled in each time samples are removed from a designated storage area.

2.3.7.2 Interim sample storage areas are located in each laboratory area for use on in-process samples which also limit access to employees only. After signing for an internal COC sample to remove from a designated storage area, samples may be stored in these areas until analysis is complete.

2.3.7.3 Once analysis is complete, samples must be returned to the original designated storage area. The internal COC form must be completed before the analyst may proceed to any other task.

2.3.8 Once a project submittal has been completed by the laboratory, the project chemist must review the COC folder contents by checking for COC form completeness. After reviewing, include the completed COC paperwork in the project folder.

2.4 The transfer of samples to an approved subcontract laboratory for analysis must adhere to the same COC requirements specified for the project.

3.0 REPORTING AND DELIVERABLES

3.1 The completed internal chain-of-custody form must accompany the client report unless otherwise specified.

4.0 QUALITY ASSURANCE

4.1 All laboratory quality assurance activities as outlined in the TriMatrix Quality Assurance Manual and as specified in any related Standard Operating Procedure, must be adhered to.

4.2 The laboratory is a restricted access area. Only authorized personnel are allowed into the laboratory without an escort. Visitors must sign the Visitor Logbook when entering and exiting the laboratory.

4.3 Project chemists are responsible for the following with respect to COC communications:

4.3.1 Communicating COC sample information to sample technicians and/or analysts as soon as this knowledge is available.

4.3.2 Monitoring sample submittal progress.

4.3.3 Collecting the COC sign-out forms when analysis is completed

4.3.4 Checking each COC form for completeness.

4.4 Sample technicians are responsible for receiving, inspecting and logging in the samples correctly for COC status, and for printing and placing COC sign-out forms into folders.

4.5 Chemists and analysts must follow a strict protocol when performing analyses on COC samples. Each time a sample is removed from a designated storage area, the COC form must be signed with the following information:

4.5.1 Removal date and time

4.5.2 Return date and time

4.5.3 Name and signature

4.6 Area supervisors and group leaders are responsible for mandating area chemists follow all internal COC protocol.

5.0 REFERENCES

5.1 TriMatrix Laboratories Quality Assurance Manual, latest revision.

Approved By: 12/2/06 **Approved By:** 12/2/06
QA Manager **Operations Manager**

6.0 ATTACHMENTS

6.1 Internal Chain-of-Custody Form Example

6.2 Facility Security Layout

6.3 Internal Chain-of-Custody Flowchart

Approved By: RL 12/21/06 **Approved By:** JB 12/21/06
QA Manager **Operations Manager**

Attachment 6.1
Internal Chain-of-Custody Example

TriMatrix Laboratories, Inc.

Internal Chain of Custody --- Work Order # **0610028**

Page 1 of 4

Client: [redacted]
Project: [redacted]

Project Manager: **Gary L. Wood**
Date Received: **Sep-30-06 09:00**

Department: **Semivolatiles MS**

Analysis: 8270

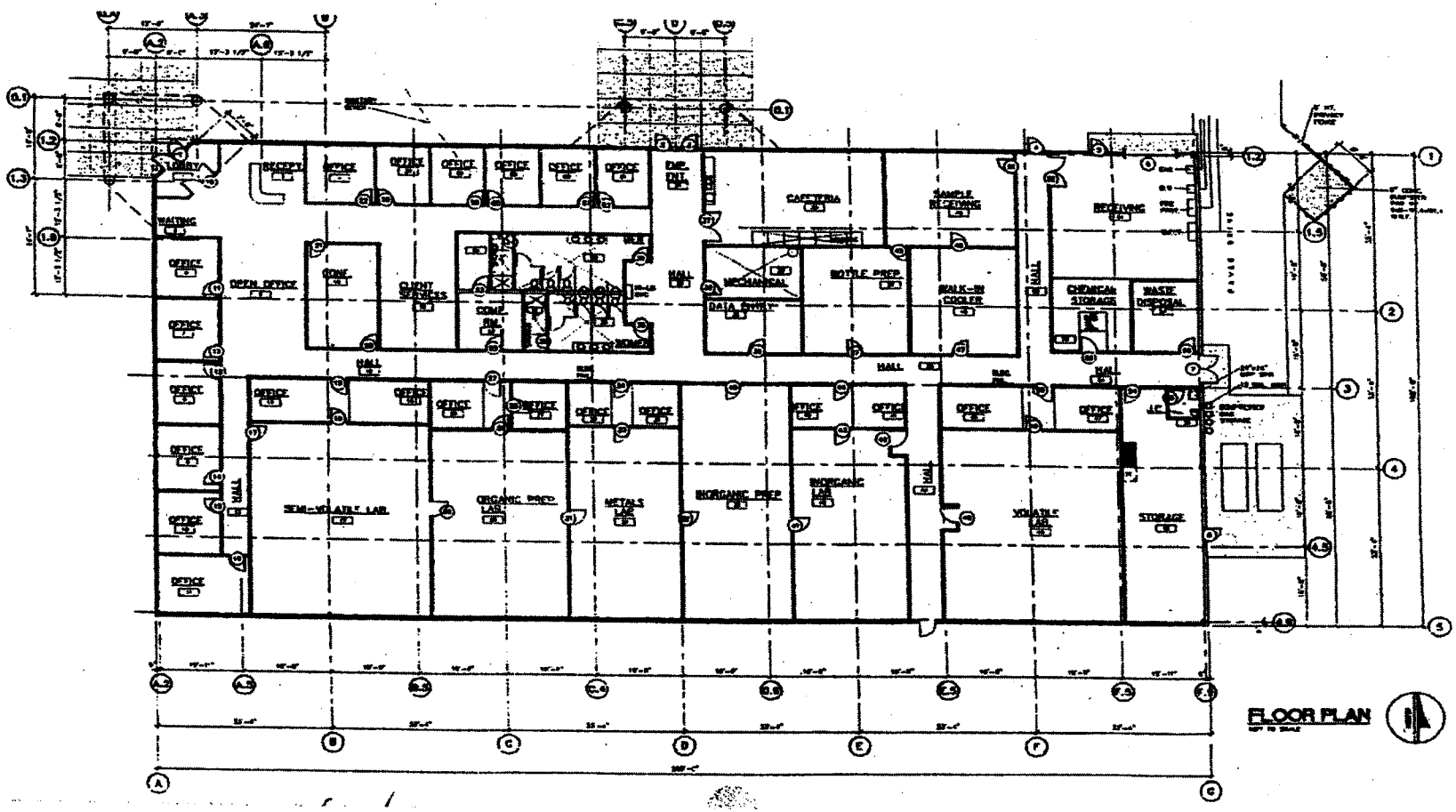
Lab Number / Sample Name	*Container*	*Removed by (Signature)*	*Date & Time Removed*	*Date & Time Returned*	*Consumed?*	*Extract Container*
0610028-01 MW-27-18	D	Andrea Calhoun	10/4/06 12:40PM		yes	D01
0610028-02 MW-27-19	C					C01
0610028-03 MW-27-6	C					C01
0610028-04 MW-27-3	D					D01
0610028-05 MW-27-17	D					D01
0610028-06 MW-27-9	D					D01
0610028-07 EW-27-2	G					G01
0610028-08 EW-27-1	C					C01
0610028-09 EW-27-1 Duplicate	C					C01
0610028-10 EW-27-4	C					C01
0610028-11 MW-5	C					C01
0610028-12 MW-27-1	C					C01
0610028-13 MW-27-16	C					C01
0610028-14 EW-27-3	C					C01

wko_TriMatrix_custody.rpt

Approved By: [signature] 12/21/06
QA Manager

Approved By: [signature] 12/21/06
Operations Manager

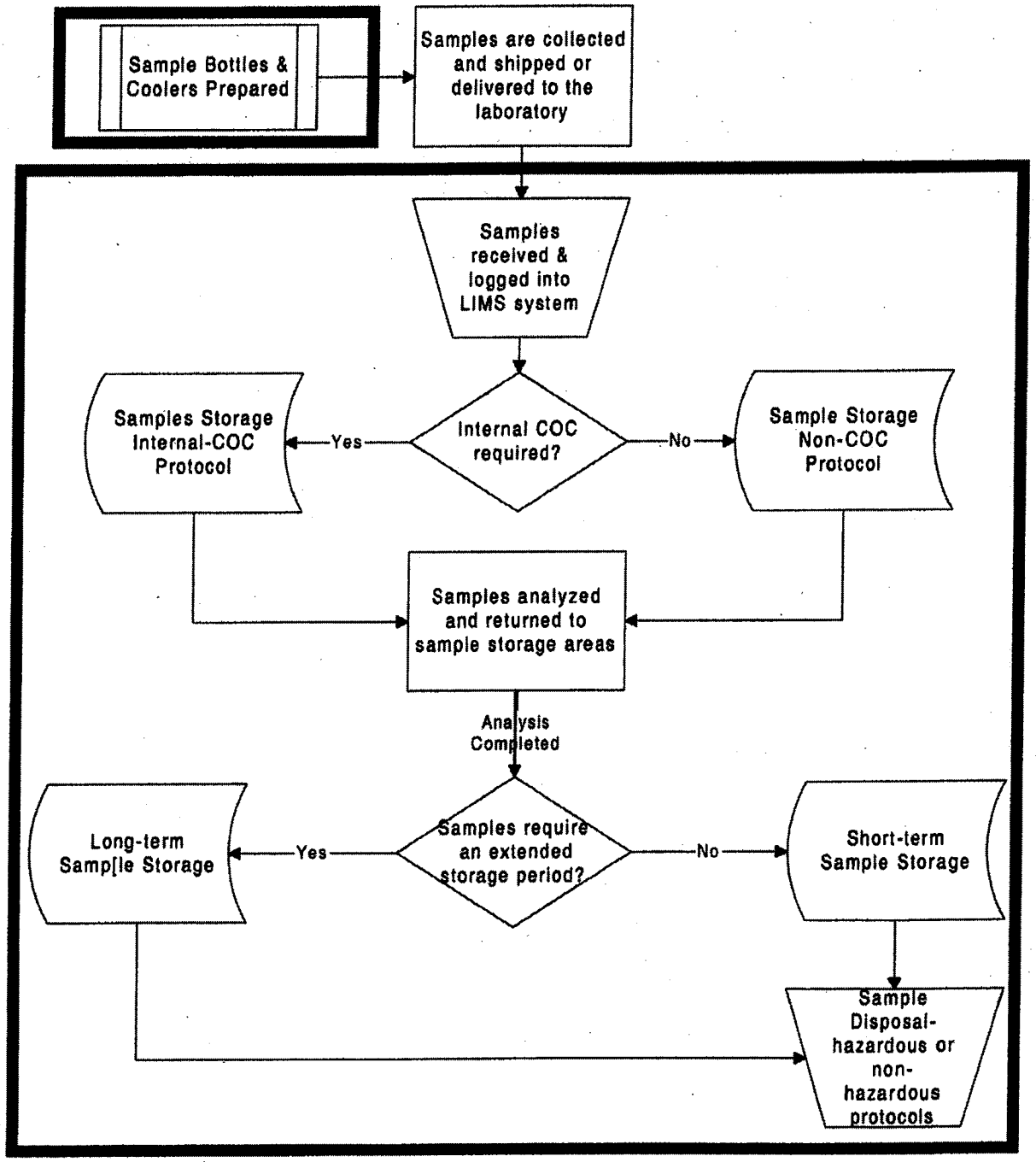
Attachment 6.2
Facility Security Layout



Approved By: Rdm 12/21/06
QA Manager

Approved By: JS 12/21/06
Operations Manager

Attachment 6.3
Internal Chain-of-Custody Flowchart



Approved By: RB 12/21/06 QA Manager

Approved By: JB 12/21/06 Operations Manager

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Sample Receipt and Log-in

APPROVALS:

Area Supervisor: _____ Date: 12/15/08
Gary L. Wood

QA Officer: _____ Date: 12-15-08
Tom C. Boocher

Operations Manager: _____ Date: 12/15/08
Jeff P. Glaser

Procedure Number: GR-15-100
Revision Number: 3.2

Date Initiated: 2/27/96
Effective Date: 12/31/08

Date Revised: 12/15/08
Pages Revised: All

By: Gary L. Wood

Total Number of Pages: 32

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SUMMARY OF PROCEDURE

1.1 This document describes in detail the procedures followed during the sample receipt and log-in process at TriMatrix Laboratories. This procedure begins with the arrival of samples at the laboratory and does not end until the samples have been properly stored.

1.2 Samples are received by the laboratory from 8:00 am to 5:00 pm, Monday through Friday. With advance notice, samples can be received on Saturday from 8:00 am to 12 noon. Integrity checks, custody transfer and placement in cold storage will be performed on Saturday. However, a complete sample log-in will not be done until the following business day.

1.3 Sample receipt at other times must be arranged through a project chemist or the log-in staff.

1.4 Technicians receiving samples that require internal chain-of-custody must also review and adhere to TriMatrix SOP GR-10-104.

2.0 DETAILED PROCEDURE

2.1 There are four distinct steps to the sample receipt/log-in process.

Step 1 The initial step acknowledges receipt of samples by the laboratory. The Chain-of-Custody (COC) is signed and required entries are made in the Sample Receipt Records Logbook. Step 1 must be completed immediately, at the time of sample receipt. However, the cooler is not opened during Step 1.

Step 2 A Sample Receiving/Log-In Checklist is initiated. The cooler is inspected for intact custody seals then opened. The sample type, sample quantity and coolant location are documented, and the sample temperature is taken and recorded. When necessary, a Sample Receiving Non-Conformance Report is initiated.

IMPORTANT: Never open a cooler unless planning on immediately completing Step 2 of the receipt/log-in process.

Step 3 Samples are removed from the cooler during the third step of the receiving process. Upon removal, samples are physically inspected and compared to the COC. The preservation of chemically preserved samples is verified and the laboratory is informed of any short hold time analysis requirements. All initial paperwork is collected and delivered to the project chemist.

Step 4 The final step of the sample receipt/log-in process involves performing any authorized pH adjustments, logging samples into the LIMS system and storing samples in assigned locations.

2.1.1 To safely and correctly complete the sample receipt/log-in process, the following equipment is required:

- Laboratory coat
- Approved safety glasses
- Disposable gloves
- Infrared thermometer, Raytek Raynger, model ST60XX95, verified as NIST-traceable

Alternative: stem-type digital thermometer, verified as NIST-traceable
Alternative: liquid-in-glass thermometer, verified as NIST-traceable

- ColorpHast strips, pH 0-14 universal, EMD catalog 9590 or equivalent
- Preservation reagents, contaminant-free
- Fume Hood

2.1.2 Personal protective equipment must be worn during sample receipt/log-in. A laboratory coat and approved safety glasses must be worn in the sample receipt and bottle preparation area except when entering data at a computer terminal.

2.1.3 Many received coolers are quite heavy. A back brace is available and highly recommended when lifting and moving any heavy object. A wheeled table must be used when transporting coolers from the garage into the sample receipt/log-in area.

2.1.4 For samples known to be extremely hazardous or toxic, additional safety precautions are required and noted throughout the procedure. Notify the Health and Safety Officer and Project Chemist before sample receipt/log-in of non-routine hazardous or toxic samples unless otherwise specified.

2.1.5 No food or drink is permitted in the sample receipt/log-in area at any time, including chewing gum.

2.2 Step 1 is outlined as follows:

2.2.1 Immediately upon receipt of a sample or sample cooler, the receipt must be recorded in the Sample Receipt Record logbook (Attachment 6.1). The logbook must contain the following entries:

- Delivery method
- Date received
- Time received
- Number of coolers received
- Client
- Name of the technician receiving coolers and recording the information

2.2.2 The "received by", date and time must also be used to complete the "received for lab by" section of the field Chain-Of-Custody (COC) form (Attachment 6.2).

2.2.3 Samples arriving via walk-in clients or TriMatrix field services personnel must be accompanied by a COC form. When possible, have the delivery person sign in the first available "relinquished by" section on the COC. The TriMatrix technician receiving the samples must sign the "received for lab by" section. This information must match that entered in the Sample Receipt Logbook. Return the pink COC copy to the delivery person.

NOTE: It is possible that samples delivered by a TriMatrix employee will be "relinquished by" and "received for lab by" the same person. This can occur after normal receiving hours or on weekends when log-in staff are not available.

NOTE: The number of coolers delivered by different delivery methods but from the same project-submittal must be additively entered into a column of the Sample Receipt Record logbook as the total number of coolers received for the project. This number must be verified when the logbook is checked, by counting to confirm the number of coolers received from all delivery methods is the same as the number recorded.

2.2.4 If Step 2 will not immediately begin, the sample cooler must be placed in the walk-in cooler.

NOTE: When samples requiring temperature preservation are received at ambient temperature, Step 2 must be performed immediately. Refrigerating non-chilled samples prior to recording their temperature results is a misrepresentation of the preservation status of the sample.

2.3 Step 2 is outlined as follows:

2.3.1 Observations associated with any step of the process must be recorded on the Sample Receiving/Log-In Checklist (Attachment 6.3). The Checklist must be completed for all samples in a given project received during the day. A Sample Receiving/Log-In Checklist Additional Cooler Information is available when receiving samples in a given project consisting of more than four coolers (Attachment 6.4).

NOTE: Shaded boxes on the Checklist indicate an out-of-control situation. The selection of ANY shaded box during Checklist completion requires initiation of a Sample Receiving Non-Conformance Report (Attachment 6.5).

2.3.2 When sample coolers are received, segregate by project. Complete the upper two Checklist sections to the extent possible. To maintain cooler temperature integrity, open and determine sample temperature on one cooler at a time.

NOTE: It is important when initiating a Checklist to be sure the page/line number from the Sample Receipt Record logbook is recorded at the top of the Checklist. The number associates a sample cooler with the samples received in the cooler.

2.3.3 Document whether custody seals are present and their condition. Remove and retain all custody seals, then open the cooler.

NOTE: Opening a cooler known or suspected to contain highly hazardous or toxic samples MUST be performed in the fume hood.

2.3.4 Before discarding any packing material collect all paperwork from inside the cooler. Paperwork sent with samples can contain significant project information, including but not limited to field screening readings, general field information, and hazard warnings. These must be retained.

2.3.5 Record the cooler number and the time the cooler was opened.

2.3.6 Observe and record the type and location of coolant used.

2.3.7 Temperature Measurement

2.3.7.1 When possible, measure and record the temperature of three representative samples from random locations within the cooler. Also, measure and record the temperature blank, if one is included.

2.3.7.2 Sample temperatures are measured using a calibrated infrared (IR) thermometer (Section 4.6). The IR thermometer measures sample temperature indirectly by taking a surface reading. Consequently, IR readings MUST be taken just as a sample is lifted from the cooler. Sample container surfaces warm quickly and any other technique will result in an incorrect measurement. Do not dry the container before taking a reading. Containers wet from melt-water are more representative when wet than those not having been in direct contact with coolant. However, do not replace a sample chosen at random that does not have melt-water. It must be used.

2.3.7.3 Aim the IR thermometer approximately six inches from the surface to be measured and pull the trigger. On clear glass containers aim the laser through the container onto the back of the label. This is not required with opaque containers (measurements can be taken without a label shot).

2.3.7.4 Laser aiming from the thermometer will illuminate the surface being measured. Hold the trigger in for 3 to 5 seconds then release and observe the result shown on the screen. Record the result obtained in degrees centigrade.

2.3.7.5 If the infrared thermometer is not available, a digital or liquid-in-glass thermometer may be used, if verified as NIST-traceable (a tag will be attached indicating when verification was last performed). These thermometer types have a potential for contamination if used directly on samples. Consequently, temperature readings must be taken only on a temperature blank or the coolant melt-water. To take a digital or liquid-in-glass thermometer reading, immerse the stem in an opened temperature blank or directly in coolant melt-water. Observe and record the temperature after the reading stabilizes. Do NOT open samples to take a temperature reading. See the quality assurance manager for assigning an alternative thermometer to the department when needed.

2.3.7.6 Record individual temperature readings and the average from each cooler on the Sample Receiving/Log-In Checklist. Report all values to the nearest 0.1° C. If a correction factor is necessary (based on the daily NIST traceability verification), record the correction factor and the corrected temperature on the Sample Receiving/Log-In Checklist.

2.3.7.7 If a temperature average or temperature blank exceeds 6° C, note on the Sample Receiving/Log-In Checklist. A Sample Receiving Non-Conformance Report will be required.

2.3.7.8 The entire procedure must be followed for each sample cooler received.

NOTE: If a trip blank is received, record the cooler number on the trip blank tag.

2.3.8 If sample receipt and temperature determination are performed outside of normal business hours, replace the temperature blank or sample removed for the temperature determination in the original cooler and transfer to the laboratory walk-in cooler. Collate all paperwork and place in the after-hours basket. The receiving process will continue on the next business day. Otherwise, proceed to Step 3.

NOTE: Accompanying paperwork including bills of lading, packing slips, air bills, manifests, field sampling forms, and correspondence documents must be retained and saved with the COC.

NOTE: If placing the sample cooler in the walk-in, first remove any large quantities of ice present on top of samples before placing in the walk-in. This will prevent damage to sample identification tags from residual melt-water. The walk-in will keep samples at an acceptable temperature.

2.4 Step 3 is outlined as follows:

2.4.1 Verify that the COC "received for lab by" is filled in. If not, it must be done immediately. To maintain the chain-of-custody timeline, enter the initials, date and time recorded in the Sample Receipt Record logbook. If verification is done by the person who received samples, record a signature (not initials) on the COC. Complete the "paperwork received" and "COC ID Nos." section of the Sample Receiving/Log-In Checklist.

2.4.2 The COC must be reviewed for accuracy. Complete the "check COC for accuracy" section of the Sample Receiving/Log-In Checklist. Additional information that must be present and correct on the COC include:

- Client name, address and contact phone number, unless on file
- Number of containers per sample
- Requested analyses (if not initially present, will be filled in by project chemist via the client)
- Page/Line number from the Sample Receipt Record logbook

2.4.3 Sample collection personnel should have filled in sample label information. If the information is missing or incomplete, notify the project chemist. In many cases, missing sample label information can be determined after checking label information on remaining samples against the COC. However, assignment of client-based sample information by TriMatrix receiving technicians must first be brought to the project chemist's attention and documented on a Sample Receiving Non-Conformance Report.

NOTE: Additional information may also be supplied by the COC, depending upon project specifications and COC type (TriMatrix or client). The project chemist must evaluate what additional information is pertinent to the log-in process and communicate this to applicable log-in staff.

2.4.4 Affix appropriate labels onto sample containers. These include MS/MSD, USDA-regulated, Composite Before Analysis, PT Sample, Caution!!!, and Return Sample to Waste Cabinet labels (Attachment 6.6).

2.4.5 Complete the "sample condition summary" section. If sample containers were broken during shipment and/or uncontained sample is observed in a cooler, unless it is known that the samples contained in the cooler are not hazardous, implementation of the following steps is required.

2.4.5.1 Immediately reseal the shipping cooler and place in the sample receipt room hood. If a cooler is too large for the hood, place in a well-ventilated area in the garage. Place a note on the cooler that it may contain contamination and is not to be opened.

2.4.5.2 Immediately notify the project chemist and when appropriate the health and safety officer. Do not continue with sample log-in for the affected samples until sufficient safety information is obtained.

2.4.5.3 The project chemist must consult with the client on how to proceed and when appropriate, request safety information. If instructed to proceed with the log-in process, review the safety information obtained and observe appropriate safety precautions. Remove the cooler contents, properly dispose of any adsorbent and decontaminate the inside and outside. Record all resolutions on the Sample Receiving Non-Conformance Report.

2.4.6 In addition to COC and cooler/sample discrepancies, project-specific issues may also be observed. Any aberration from acceptable sample receipt protocol must be documented on a Sample Receiving Non-Conformance Report when found. Such issues include the following:

- Project name correctness
- Hold time violations due to late receipt
- Illegible paperwork
- Incorrect project or purchase order numbers
- Missing project chemist name
- Questionable parameter list
- General information missing

2.4.7 Many aqueous samples received are subjected to some form of chemical preservation. Verification of the preservation is required. Depending on the analysis, preservative verification may not occur during log-in. The Sample Preservation Verification Form (Attachment 6.7) specifies which sample types will have preservation verified during log-in. The form also specifies which preservation may be adjusted by log-in, if samples are received incorrectly. No preservation adjustment may be made without project chemist approval.

2.4.7.1 Acid and base preservation is checked using 0-14 pH strips. Determine sample pH by briefly dipping (1-3 seconds) a pH strip in a sample and comparing the pH strip color against the chart on the pH strip container. The color that matches is the pH reading. Record preservation verification results on the Sample Preservation Verification Form. If preservation is acceptable, place a check mark in the box corresponding to the checked sample. If preservation is not acceptable, record the actual pH obtained in the appropriate box. Use only pH strips located in the log-in room. Each lot is verified for accuracy and tested as contaminant-free (section 4.3).

IMPORTANT: It has been previously documented that over time barium will leach off a pH strip. Because of the potential for barium contamination, when testing the pH of a number 6 container, never allow the pH strip to remain in the water longer than 3 seconds.

NOTE: If there is insufficient evidence that other preservation (for example, zinc acetate for sulfides) is present, a Sample Receiving Non-Conformance Report must be initiated and the project chemist contacted immediately.

2.4.7.2 If a preservation check indicates unacceptable pH at sample-receipt, an orange Out-of-Control Sample pH tag (Attachment 6.8) must be placed on the container and "initial pH" filled in. In some cases, the sample hold time will be shortened. Except for type 6 bottles, the pH adjustment will be made in Step 5.

2.4.7.2.1 Acid preservation on type 6 bottles at the time of (or following) sample receipt requires sample pre-treatment or analysis be delayed for a minimum of 16 hours. The delay allows the added acid to re-solubilize precipitated or wall-adsorbed analyte (Refer to TriMatrix SOP GR-01-145, Section 8.1). To avoid additional delay it is important that the metals preparation staff be informed immediately of the preservation adjustment. Provide a copy of the COC and the Sample Preservation Verification Form. Upon receipt of the COC/SPV forms, the metals preparation staff must obtain project chemist approval to perform the pH adjustment.

2.4.7.2.2 The metals preparation analyst must record all adjustment information on the out-of-control pH tag when making an adjustment.

2.4.8 Sample hold time is defined as the maximum storage time allowed between sample collection and analysis when designated preservation and storage techniques are employed. Hold times are typically specified to include any sample preparation as part of the analysis. Sample preparation and analysis must occur within the EPA-prescribed hold time or sample data must be qualified. Consequently, it is important that appropriate laboratory areas be informed immediately of any received samples with a significant time delay between collection and receipt by the laboratory. Also, it is important that appropriate laboratory areas be informed immediately of any received samples with very short hold times.

NOTE: It is the log-in technician's responsibility to be familiar with hold times and to identify any received sample parameter with a short hold time. A Short Hold Time Analysis List is provided in Attachment 6.9.

2.4.9 If samples with short hold times are received, check the appropriate box or boxes on the Sample Receiving Log-In Checklist and notify the project chemist and all applicable laboratory chemists. Document the completion of all notifications on the Checklist. Provide each affected laboratory area with a copy of the COC.

2.4.10 If a sample is received with an expired hold time or with a hold time due to expire during sample log-in, contact the project chemist immediately.

2.4.11 Samples must be placed in temporary storage until the Laboratory Information Management System (LIMS) data entry is complete.

2.4.11.1 Water and wastewater VOC vials, charcoal tubes and airbags must be stored in the water-volatiles refrigerator located in the log-in area. Samples requiring refrigeration must not be left un-refrigerated longer than 15 minutes after unpacking. Bulk soil VOC containers must be stored in the soil-volatiles refrigerator, located in the log-in area. EnCore®, methanol-preserved and bisulfate-preserved volatiles soil samples must also be stored in the soils refrigerator. However, due to short hold times these are typically delivered and stored in the appropriate refrigerator located in the volatiles laboratory. Received microbiological samples must be stored on the designated shelf in the walk-in cooler.

2.4.11.2 All waste samples must be placed in the log-in hood when received. Waste-volatile samples must be stored inside the waste-volatiles refrigerator located in the log-in hood.

NOTE: If a received waste sample is to be analyzed for volatiles but no dedicated sample container is received, inform the laboratory. Have an analyst remove an aliquot for the volatiles analysis before the sample is opened for other analyses.

2.4.11.3 All other water, wastewater and soil containers must be placed on a numbered cart and stored in the walk-in refrigerator. Record the cart number on the Chain-of-Custody to help laboratory chemists locate short hold time samples that have not been logged in.

2.4.12 Additional information or questions for the project chemist can be recorded in the "notes" section of the Checklist. Record whether a trip blank was received/not received. If received, mark that the trip blank was recorded on the COC. Record the date and time the project was received at the laboratory and the time paperwork was brought to the project chemist.

2.4.13 All paperwork received with samples such as bills of lading, packing slips, air bills, manifests, field sampling forms, correspondence documents, the COC and all other paperwork generated during the log-in process must be placed in a green folder and delivered to the appropriate project chemist for an initial paperwork review. If the project chemist is not in the office, place the folder in the project chemist's bin.

2.4.14 The project chemist must review all paperwork, create a Sample Login Specification Form and return all paperwork to the log-in area.

2.5 Step 4 is outlined as follows:

2.5.1 After reviewing the paperwork and creating the Sample Login Specification Form, the project chemist must return the green folder to the log-in area. The assigned technician must then

review page two of the Sample Receiving/Log-In Checklist for any additional instructions made by the project chemist. Using the Sample Receiving/Log-In Checklist, the COC and any associated receipt paperwork the assigned technician may proceed to log samples into LIMS (Element™). Each step of this process must be documented on page two of the Checklist.

2.5.2 The information listed below is required to properly log samples into LIMS. After the information has been entered and compiled, LIMS will print labels for each sample container. Printed labels are affixed to each associated sample container tag.

2.5.2.1 Client and project names - All narratives entered into LIMS by the project chemist must be reviewed.

2.5.2.2 Schedules to be used – All relevant analysis, matrix, container type and size, and quantities must be scheduled into the project by the Project Chemist prior to sample login unless otherwise specified. The login technician may then select the schedule referenced on the COC and detailed on the Sample Login Specification Form. After selecting the schedule, modify the field sample name (if different than the schedule name) then add the sampler's name and the collection date and time from the COC. Each number must be unique and designated as the laboratory identifier for the given sample.

2.5.2.3 Bottle description (as received) - The number corresponding to the bottle-type on the Container Packing List (Attachment 6.10). The Container Packing List is generated from the bottle order that supplied sample collection containers.

2.5.2.4 Client (field) sample identification from the COC.

2.5.2.5 Sample collection date and the military time.

2.5.2.6 When supplied, first initial and last name of the sampler.

2.5.2.7 COC number if available (TriMatrix COC forms are uniquely numbered).

2.5.2.8 The number of containers received for each sample.

2.5.2.9 Vial rack or tray number (for a volatiles sample) and storage area identification.

2.5.2.10 When complete, record the initials/date/time on the Receiving/Log-In Checklist.

NOTE: All trip blanks must be assigned a "date sampled" and "time sampled". Assign a "date sampled" equal to the latest sample date of all the samples in the submittal. Assign a time collected of 00:00.

NOTE: It is common for a client to assign a different collection date and/or time for trip blanks such as the date a trip blank is shipped or no collection time. Any correction to a client-specified collection date and/or time must be made or authorized by the project chemist with the change properly noted on the COC. Refer to Section 4.13 regarding COC corrections.

2.5.3 Perform the following after entry of the above information into LIMS.

2.5.3.1 Affix each printed label to the associated sample tag or container.

NOTE: If there are samples unaccounted for at this time (i.e. a sample is encountered that was not found on the COC, initiate a non-conformance and inform the project chemist. Check the Discrepancy box on the non-conformance form.

2.5.3.2 If approval is given, any necessary preservation adjustments may now be made to orange-tagged samples.

2.5.3.2.1 Only for those container types listed on the Sample Preservation Verification Form and only with project chemist/client approval, add acid or base to samples requiring a pH adjustment. All post-receipt preservations must use reagents from the bottle preparation room. To make a pH adjustment, add just enough acid or base to correct the discrepancy but do not exceed twice the original preservative volume used for normal bottle preparation. Normal volumes are listed on the Sample Preservation Verification Form. If pH is not adjusted using twice the normal volume, the sample exhibits buffering and must be noted as such on the Sample Preservation Verification Form.

2.5.3.2.2 After pH adjustment, record the final pH in the box adjacent to the original pH on the Sample Preservation Verification Form. Also, record the final pH and acid/base volume added in the appropriate box on the orange "out-of-control sample pH" tag. Check "ok" or "still out" relative to the final pH attained.

2.5.3.2.3 The technician performing a pH adjustment must complete the "adjusted by" box on the Sample Preservation Verification Form.

2.5.3.3 Initial, date and check the "label sample containers" section of the Sample Receiving/Log-In Checklist after labeling.

2.5.3.4 All tagged and labeled sample information must be verified by initialing and dating the "verified by" section of the Checklist.

2.5.4 Sample Storage

2.5.4.1 All samples must be stored in a secure restricted-access area and/or unit.

2.5.4.2 Samples are stored in the walk-in cooler shelf areas according to sample number, in a designated refrigerator or in numbered container racks.

2.5.4.3 Sample storage refrigerator information is recorded in the Controlled Temperature Unit (CTU) Daily Monitoring Logbook.

2.5.4.4 No sample may be stored in the same refrigerator as analytical standards, chemicals or food/drink. These items are prohibited from storage in any refrigerator designated for sample storage.

2.5.4.5 Volatiles samples are stored in a refrigerator in the volatiles laboratory. Soil and water samples are stored separately. Samples are further organized by shelf and rack number.

2.5.4.6 Non-volatile water and soil samples are stored in the walk-in cooler. Waste samples for volatiles analysis are stored in a designated refrigerator in the sample receipt/log-in fume hood.

2.5.4.7 Non-volatile waste samples are stored in a vented storage cabinet in the garage area. Samples are segregated by hazard class. The client name and sample number of each sample stored must be written on the Waste Cabinet Sample Inventory Form (Refer to Attachment 6.11).

2.5.4.8 If a received sample is determined to be extremely hazardous or toxic during log-in or analysis, all aliquots of the sample must be relocated to the waste storage cabinet.

2.5.4.9 Tedlar® bags and charcoal tubes for air analysis are stored in the log-in water-volatiles refrigerator.

2.5.4.10 Microbiological samples are stored on a designated shelf in the walk-in cooler.

2.5.4.11 As sample numbers change in the walk-in refrigerator, storage shelf labels must be clearly marked with the new range of numbers.

2.5.5 Sub-sample compositing is performed by applicable laboratory analysts when needed. A composite sample must be identified as such on the composite container tag. A LIMS narrative must also be entered by the compositing analyst.

2.6 LIMS-assigned Laboratory Sample Identifier (ID)

2.6.1 A unique LIMS-assigned laboratory sample identification number (ID) is generated during the log-in process. The sample ID consists of the workorder number followed by a hyphen and a numeric identifier unique to each sample received (for example: -01, -02, -03).

2.6.2 The workorder number consists of the truncated year and month followed by a three-digit number representing how many workorders were received month-to-date. For example, 0704456 reflects the year 2007, April and the 456th workorder logged in during April, 2007.

2.6.3 The workorder number appears on laboratory benchsheets and is used as the sample ID on all instrument run logs and associated raw data. It is traceable to the client-assigned sample identifier.

2.6.4 A letter suffix follows any sample ID to uniquely identify each sample container received. For example, 0704456-04A identifies the first sample container received; 0704456-01B identifies the second and so on until all sample containers are identified.

2.7 At the completion of the log-in process, collect all chain-of-custody forms and other documents received, the Sample Receiving/Log-In Checklist, all other log-in documents and the LIMS-generated workorder report (once available) in a folder. Deliver the folder to the project chemist for review and approval.

2.8 Sample Disposal is as follows:

2.8.1 The number of days samples must be retained by the laboratory after the completion of all analyses is specified in LIMS by the project chemist. The LIMS default is 14 days after the final data report has been generated.

2.8.2 When the sample retention time has expired, samples may be appropriately disposed of (non-hazardous or hazardous) or returned to the client.

2.8.3 The waste disposal technician or other designated person must determine how samples are disposed of in accordance with the latest revision of TriMatrix SOP GR-15-102.

3.0 REPORTING AND DELIVERABLES

3.1 There is no reporting and deliverables directly associated with this procedure other than those described above.

4.0 QUALITY ASSURANCE

4.1 For each workorder generated, workorder numbers must be entered into the Sample Receipt Record Logbook. Verification that a workorder folder has been compiled must also be documented by recording in the "folder prepared" column of the logbook. This logbook must be reviewed on a daily basis.

4.2 Each lot of pH strips must be checked against certified pH buffers and documented in the pH Strip Calibration Logbook (Attachment 6.12). This test is performed by the wet chemistry laboratory.

4.3 New pH strip lots must also be tested for contaminants and interferences.

4.3.1 When an approximately one-month supply of pH strips remains, place an order for another four to six-month supply. The ordered pH strips must be from one lot number. Upon receipt by the laboratory, provide the Quality Assurance Department with the lot number. The Quality Assurance Department will act as the project chemist and create a LIMS workorder for the test.

4.3.2 To test a new pH strip lot, add the appropriate preservative to container types 3, 4, 5, 6, 13 in accordance with TriMatrix SOP GR-15-101. Fill with laboratory reagent water from the applicable laboratory area and deposit a pH strip in each container. Let the containers stand for 24 hours in the sample receipt/log-in fume hood.

NOTE: It has been previously documented that barium will leach from a pH strip during this test. For that reason, prepare a second type 6 bottle without the addition of a pH strip. Rather than depositing the strip in the container the metals analyst must quickly dip the strip in the container to mimic a typical pH sample check. Analyze the water in the dipped container for barium only.

4.3.3 After 24 hours, deliver all test containers to the appropriate laboratory area for analysis. The following analyses must be performed on the test water:

Container Number	Tag Color	Preservative	Test
5	Light Blue	NaOH	Cyanide
3	Green	none	Nitrate, Nitrite, Sulfate, Chloride, Fluoride, Bromide, Cr^{6+}, Alkalinity, Acidity, o-Phosphate
4	Dark Blue	H_2SO_4	TKN, Nitrate-Nitrite, Phosphorus, COD, Ammonia
13	Brown	H_2SO_4	Phenolics
6	Red	HNO_3	Ca, Fe, Li, Mg, Hg, K, Na, Sr, Sn, Ti, Al, Sb, As, B, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, V, Zn
6	Red	HNO_3	Ba

4.3.4 The Quality Assurance Officer must review the test results and approve/reject the new lot of pH strips. Do not use the new lot until approval has been received.

4.4 Liquid-in-glass thermometers must be verified against a NIST-traceable thermometer, at least annually.

4.5 Digital stem-type thermometers must be verified against a NIST-certified thermometer, at least quarterly.

4.6 The Infrared thermometer must be verified daily against the NIST-traceable thermometer located in the walk-in cooler. This verification is performed by the Quality Assurance Department. The IR reading must agree within ±0.5° C of the cooler thermometer. Document the test results in the Infra-Red Thermometer Calibration Record logbook (Attachment 6.13). A difference of greater than ±0.5° C will require the use of a correction factor on all temperature measurements made that day.

4.7 Refrigerator temperatures must be verified daily. Results of the verification are recorded in the Controlled Temperature Unit Logbook (Attachment 6.14). This verification is performed by the Quality Assurance Department.

4.8 Storage blanks are in all refrigerators used for volatile organic samples storage. These blanks are analyzed weekly by the volatiles laboratory.

4.9 All tagged and labeled sample information must be verified by initialing and dating the "verified by" section of the Checklist

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QA Officer **Area Supervisor**

4.10 Paperwork contained in the project submittal receipt document folder must be reviewed by the project chemist.

4.11 Samples whose pH preservation is incorrect must be tagged with an out-of-control sample pH tag. Improper preservation is corrected only with approval from the project chemist. Record the discrepancy on the tag and all appropriate paperwork (Section 2.4.7).

4.12 A Sample Receiving Non-Conformance Report must be generated for the following problems:

- A sample ID discrepancy between a COC and container
- Missing, broken, inappropriate or extra container
- Illegible, missing or incomplete label
- Headspace in a container
- Low sample volume
- Incorrect preservation (chemical and/or temperature)

4.13 Irregularities and non-conformances in sample receipt/log-in documentation must be appropriately corrected and fully documented. The chain-of-custody and associated documents comprise an agreement between TriMatrix Laboratories and clients for analytical services requested and performed.

4.13.1 If a COC item requires correction, draw one line through the item then date and initial the line. This must be done only by client request or authorization. Consequently, always include "per <Client representative's name>".

4.13.2 If a client provides pre-digested, pre-leached or pre-extracted samples and is requesting only an analysis, notify the project chemist before proceeding with sample log-in. The exception is when prior arrangements were made and previously communicated to log-in by the project chemist.

4.13.3 Do NOT obliterate an original communication by scribbling or any other type of write-over. The original must remain legible. Strike out the original communication with a single line then date and initialize the correction.

4.13.4 Use ONLY indelible blue or black ink.

5.0 REFERENCES

5.1 TriMatrix SOP GR-15-101, *Bottle Preparation*, latest revision

5.2 TriMatrix SOP GR-10-104, *Internal Chain-of-Custody*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 3, December, 1996, Chapter 2 Table 2-36, Chapter 4 Table 4-1, Chapter 3 Table 3-1

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5.5 *Code of Federal Regulations*, Title 40, Protection of the Environment, Volume 19, latest edition, Chapter I, Part 136, Subpart D, Guidelines Establishing Test Procedures for the Analysis of Pollutants, Environmental Protection Agency

5.6 *Code of Federal Regulations*, Title 40, Protection of the Environment, Volume 19, latest edition, Chapter I, Part 141, Subpart D, National Primary Drinking Water Regulations

5.7 Competency Requirements for Calibration and Testing Laboratories: International Standards Organization, ISO/IEC Guide 17025, latest revision

5.8 National Environmental Laboratory Accreditation Conference (NELAC), *Quality Systems*, Chapter 5, latest revision

5.9 TriMatrix SOP GR-01-145, *Inductively Coupled Plasma Atomic Emission Spectroscopy*, latest revision

6.0 ATTACHMENTS

6.1 Sample Receipt Record logbook

6.2 Field Chain-of-Custody form

6.3 Sample Receiving/Log-In Checklist

6.4 Sample Receiving/Log-In Checklist-Additional Cooler Information

6.5 Sample Receiving Non-Conformance Report

6.6 TriMatrix Sample Container Tag and Log-In Labels

6.7 Sample Preservation Verification Form

6.8 Out-of-Control Sample pH Tag

6.9 Short Hold Time Analysis List

6.10 Container Packing List

6.11 Waste Cabinet Sample Inventory form

6.12 pH Strip Calibration Logbook

6.13 Infra-Red Thermometer Calibration Record

6.14 Controlled-Temperature Unit Daily Log Sheet

Attachment 6.1
Sample Receipt Record

Sample Receipt Record

TriMatrix Laboratories, Inc.

Date: _____

Delivery Method A: _____ No. of Sample Boxes: _____ Number of Coolers: _____ Signed for By: _____ Time: _____
Delivery Method B: _____ No. of Sample Boxes: _____ Number of Coolers: _____ Signed for By: _____ Time: _____
Delivery Method C: _____ No. of Sample Boxes: _____ Number of Coolers: _____ Signed for By: _____ Time: _____
Delivery Method D: _____ No. of Sample Boxes: _____ Number of Coolers: _____ Signed for By: _____ Time: _____
TriMatrix Courier (TC): _____ No. of Sample Boxes: _____ Number of Coolers: _____ Signed for By: _____ Time: _____

Page/ Line Number	Client	Quantity of Coolers OR TriMatrix Cooler Number	Arrived in Laboratory					Submittal Number (Project Chemist)	Folder Prepared (Log-In ✓)
			Time	AM	PM	Received By	Delivery Method Letter		
1-1									
1-2									
1-3									
1-4									
1-5									
1-6									
1-7									
1-8									
1-9									
1-10									
1-11									
1-12									
1-13									
1-14									
1-15									
1-16									
1-17									
1-18									
1-19									
1-20									
1-21									

file: samplereceipt 2 page: 1 of 10 revision: 1.8

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.2
Field Chain-of-Custody Record

TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record COC No. **105754**

Page ____ of ____

For Lab Use Only
Cart
VOA Rack/Tray
Receipt Log No.
Project Checklist
Laboratory Project No.

Client Name
Address
Phone
Fax
Project Name
Client Project No. / P.O. No.
Invoice To ☐ Client ☐ Other (comments)
Contact/Report To

Analyses Requested

⇦ PRESERVATIVES
A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Test Group	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	COMP	GRAB	Matrix	Container Type (corresponds to Container Packing List) / Number of Containers Submitted	Total	Sample Comments
			1										
			2										
			3										
			4										
			5										
			6										
			7										
			8										
			9										
			10										

Sampled By (print)
Sampler's Signature
How Shipped? Hand Carrier_____
Tracking No.
Comments

Company
1. Relinquished By Date Time
2. Relinquished By Date Time
3. Relinquished By Date Time
1. Received By Date Time
2. Received By Date Time
3. Received For Lab By Date Time

TriMatrixCOC -- COC WHITE COPY - REPORT YELLOW COPY - LABORATORY PINK COPY - FIELD

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.3
Sample Receiving Log-In Checklist (page 1)

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST

Client	Project-Submittal No. new / add to
Receipt Record Page/Line No.	Project Chemist / Sample Nos.

Coolers Received

Recorded by (initials/date)	☐ Cooler ☐ Box ☐ Other_____	Qty Received	Thermometer Used: ☐ IR Gun (#94) ☐ Digital Thermometer (#54) ☐ Other (#_____)	☐ See Additional Cooler Information Form

Cooler No. / Time	Cooler No. / Time	Cooler No. / Time	Cooler No. / Time
Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact	Custody Seals: ☐ none ☐ present / intact ☐ present / not intact
Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom	Coolant Location: Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers	Coolant / Temperature Taken Via: ☐ loose ice / avg 2-3 containers ☐ bagged ice / avg 2-3 containers ☐ blue ice / avg 2-3 containers ■ none / avg 2-3 containers
Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container	Alternate Temperature Taken Via: ☐ temperature blank (tb) ☐ 1 container
Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C	Recorded °C / Correction Factor °C / Actual °C
tb	tb	tb	tb
tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice	tb location: representative / in ice
1	1	1	1
2	2	2	2
3	3	3	3
Average °C	Average °C	Average °C	Average °C
☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?	☐ Cooler ID on COC?
☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?	☐ VOC trip blank received?

If any shaded areas checked, complete Sample Receiving Non-Conformance Form

Paperwork Received ☐ No COC received

N/A	Yes	No	
	☐	☐	Chain of Custody Record(s)?
	☐		If No, COC initiated by _____
	☐		Rec'd for Lab signed/date/time?
☐	☐		Shipping Document?
☐	☐		Other _____

COC ID Nos.

☐ TriMatrix

☐ Other (name or ID#)_____

Check COC for Accuracy ☐ No analysis requested

Yes	No	
☐	■	Sample ID matches COC?
☐	■	Sample date and time matches COC?
☐		Container type completed on COC?
☐	■	All container types indicated are received?

Sample Condition Summary ☐ Non-TriMatrix containers, see Notes

N/A	Yes	No	
	■	☐	Broken containers/lids?
	■	☐	Missing or incomplete labels?
	■	☐	Illegible information on labels?
	■	☐	Low volume received?
	■	☐	Inappropriate containers received?
☐	■	☐	VOC vials have headspace?
☐	■	☐	Extra sample locations / containers not listed on COC?

Check Sample Preservation

N/A	Yes	No	
	☐	■	Average sample temperature ≤6° C?
☐	☐		Completed Sample Preservation Verification Form?
☐	☐	■	Samples preserved correctly?
☐	☐		If "No", added orange tag?
☐	☐		Received pre-preserved VOC soils? ☐ MeOH ☐ Na_2SO_4

Check for Short Hold-Time Prep/Analyses

- ☐ Bacteriological
- ☐ Air Bags
- ☐ EnCores / Methanol Pre-Preserved
- ☐ Formaldehyde/Aldehyde
- ☐ Green-tagged Containers
- ☐ Yellow/White-tagged 1L Ambers (SV Prep-Lab)

AFTER HOURS ONLY:
COPIES OF COC TO LAB AREA(S)
☐ NONE RECEIVED
☐ RECEIVED, COCs TO LAB(S)

Notes

☐ Trip blank received ☐ Trip blank not listed on COC
☐ No COC received, Proj. Chemist reviewed (init./date) _____
☐ No analysis requested, Proj. Chemist completed (init./date) _____

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤1 Hour Goal Met?
		Yes / No

revision: 2.5 Log In Forms - Receiving.Log-In_Checklist

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.3
Sample Receiving Log-In Checklist (page 2)

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST - page 2

Project Chemist Use

Notify Laboratory Personnel of Short Hold-Times and/or Rush Work ☐ NONE

(Lab personnel notified/date)

- ☐ Inorganics _____
- ☐ Microbiology (bacteria) _____
- ☐ Metals Prep _____
- ☐ Metals _____
- ☐ GC-Volatiles _____
- ☐ MS-Volatiles _____
- ☐ Semi-Vol. Prep _____
- ☐ GC-Semi-Volatiles _____
- ☐ MS-Semi-Volatiles _____

Log-In Priority ☐ RUSH ☐ Standard

Project Chemist Notes to Log-In Personnel

Trip Blank: ☐ Log-in ☐ Do not log-in

☐ Prep Storage Blank for Client (VOCs)

☐ Sub-Contracting required ☐ Coolant required

☐ Non-TriMatrix or non-standard container type(s) received
Check pH of container type _____
Expected pH:_____ ☐ Adjust if needed

☐ Adjust pH of orange-tagged containers

☐ Lab-filter samples and document on Preservation Form

Sample Narratives to be added at Log-in

_____
_____
_____
_____
_____

Log-In Use

Log Samples into LIMS Sample Nos.

N/A	Yes	
	☐	Receive samples in LIMS
	☐	Date/Time received entered in LIMS match COC
	☐	Read project and submittal narratives
☐	☐	Enter VOC rack/tray number into submittal narrative
	☐	Enter sample information into LIMS
☐	☐	Add any sample narratives
☐	☐	If non-conformance issues, add sample qualifiers
	☐	Print sample number labels

Log-in Analyst (initials/date/time)

Label Sample containers

N/A	Yes	No	
	☐	☐	LIMS label matches tag?
☐	☐		DISCREPANCIES CORRECTED IN LIMS Initials/Date: _____
☐	☐		Applicable stickers applied to labels?
			☐ MS/MSD sample
			☐ Composite before analysis
☐	☐		Applicable stickers applied to containers?
			☐ Waste sample
			☐ PT sample
			☐ USDA regulated
	☐	☐	Orange-tagged containers present?
☐	☐	☐	Adjust pH per Project Chemist
☐	☐		Initials and Date/Time Adjusted on orange tag?
☐	☐		Initials and Date/Time Adjusted on Preservation Form?

Verify Label Accuracy

	☐	Second analyst checked labels for accuracy?
☐	☐	Verify that Orange-tagged containers adjusted/initialed?

Labeled by (initials/date)	Verified by (initials/date)

Sample Storage Check all that apply

bacteria.....	☐ bacteria refrigerator
non-volatiles.....	☐ walk-in cooler
volatiles.....	☐ volatile lab refrigerator
waste.....	☐ waste cabinet
waste VOCs.....	☐ log-in hood refrigerator
low-level Hg	☐ metals lab - DO NOT STORE IN WALK-IN

Paperwork

N/A	Yes	
	☐	original COC (white)
	☐	copy of COC (yellow)
	☐	receiving/log-in checklist
☐	☐	additional cooler information form
☐	☐	sample preservation verification
☐	☐	sample receiving non-conformance form
☐	☐	shipping documents
☐	☐	custody seals
	☐	arrival log
☐	☐	other (note)

For Internal Use Only - DO NOT COPY

revision: 2.5 Log In Forms - Receiving.Log-In_Checklist

Approved By: DM 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.4
Sample Receiving/Log-In Checklist
Additional Cooler Information

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING / LOG-IN CHECKLIST
ADDITIONAL COOLER INFORMATION

Recorded by (initials/date)	Client	Project-Submittal No.
	Receipt Log No. / Sample Nos.	Project Chemist

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant/Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Cooler No.	Time

Custody Seals
- ☐ none
- ☐ present / intact
- ☐ present / not intact

Coolant Location:
Dispersed / Top / Middle / Bottom

Coolant / Temperature Taken Via:
- ☐ loose ice / avg 2-3 containers
- ☐ bagged ice / avg 2-3 containers
- ☐ blue ice / avg 2-3 containers
- ☐ none / avg 2-3 containers

Alternate Temperature Taken Via:
- ☐ temperature blank (tb)
- ☐ 1 container

Recorded °C	Correction Factor °C	Actual °C
tb		
tb location: representative / in ice		
1		
2		
3		
	Average °C	

- ☐ Cooler ID on COC?
- ☐ VOC trip blank received?

Comments

revision: 2.0 | Log In Forms - Additional_Cooler_Info

Approved By: 12-15-08 **QA Officer** | **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.5
Sample Receiving Non-Conformance Report

TriMatrix Laboratories, Inc.

SAMPLE RECEIVING NON-CONFORMANCE REPORT

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

List non-conformance issues associated with this submittal in the chart below/left. Identify discrepancies between the COC and sample tags in the chart below/right. Add comments as needed. Give to Project Chemist for immediate action.

COC ID No.	Line No.	Type of Problem: Discrepancy	Missing Container	Broken Container	Label Missing/Incomplete	Label Illegible	Low Volume	Inappropriate Container	Headspace	Not Listed on COC	Preservation	COC: Sample Field ID	COC: Date Sampled	COC: Time Sampled	COC: Container Type	COC: Qty	Sample Tag: Sample Field ID	Sample Tag: Date Sampled	Sample Tag: Time Sampled	Sample Tag: Container Type	Sample Tag: Qty	LIMS Qualifiers entered for this sample

Comments:

Project Chemist (initials/date)

revision: 1.2 Log In Forms -- Sample_Receipt_Non-Conformance page ___ of ___

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.6
TriMatrix Sample Container Tag and Log-In Labels

381536
WG-011305- ML-011
[redacted] Office
PROJ: [redacted]
SUBM: January 13, 2005 Samples
F=0 R=0 C=0 H=0

Project Specific Sample
MS/MSD

TriMatrix Laboratories, Inc.

USDA REGULATED SOIL SAMPLE

Client _____
Project Number: _____
Date: _____ Time: _____
Preservative: H_2SO_4
Sampled By: _____
Sample Location: _____
_____

PINK

Field pH _____
Please verify pH in laboratory.
_____ Initials _____ Date

PE SAMPLE
do not dispose of until notified

Store at Room Temperature

Composite Before Analysis

Composite Before Analysis

Waste Sample
Return to appropriate shelf in waste cabinet

Approved By: [signature] 12-15-08
QA Officer

Approved By: [signature] 12/15/08
Area Supervisor

Attachment 6.7
Sample Preservation Verification Form

TriMatrix Laboratories, Inc.

SAMPLE PRESERVATION VERIFICATION FORM

page ___ of ___

Client		Project-Submittal No.
Receipt Log No.	Completed By (initials/date)	Project Chemist

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	>12	<2	<2		~7	<2	<2	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								
Comments								

COC ID No.			Adjusted by:_____ Date:_____		DO NOT ADJUST pH FOR THESE CONTAINER TYPES			
Container Type	5	4	13		3	6	15	
Tag Color	Lt. Blue	Blue	Brown		Green	Red	Red Stripe	
Preservative	NaOH	H_2SO_4	H_2SO_4		None	HNO_3	HNO_3	
Expected pH	>12	<2	<2		~7	<2	<2	
COC Line No. 1								
COC Line No. 2								
COC Line No. 3								
COC Line No. 4								
COC Line No. 5								
COC Line No. 6								
COC Line No. 7								
COC Line No. 8								
COC Line No. 9								
COC Line No. 10								
Comments								

pH strip lot No.
- ☐ OC481846
- ☐ _____

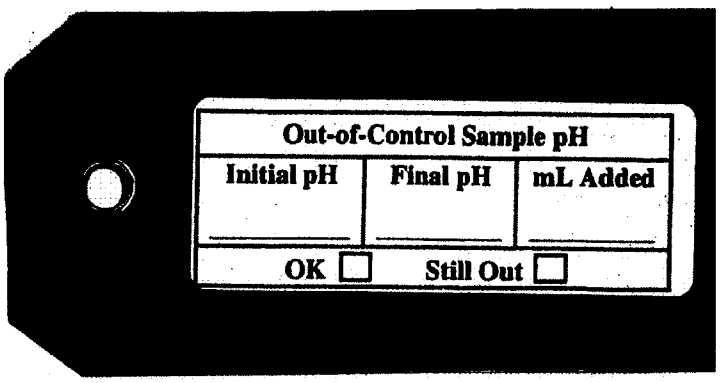
Aqueous Samples: For each sample and container type, check the box if pH is acceptable. **If pH is not acceptable for any sample container, record pH in box, and note on Sample Receiving Checklist and on Sample Receiving Non-Conformance Form.** If approved by Project Chemist, add acid or base to the sample to achieve the correct pH. Add up to, but do not exceed 2x the volume initially added at container prep (see table below for initial volumes used). **Add orange pH tag to sample container and record information requested. Record adjusted pH on this form. Do not adjust pH for container types 3, 6, and 15.**

Container Size (mL)	Original Vol. of Preservative (mL)
Container Type 5:	NaOH
500	2.5
1000	5.0
Container Type 4:	H_2SO_4
125	0.5
250	1.0
500	2.0
1000	4.0
Container Type 13	H_2SO_4
500	2.5

version: 1.5 Log In Forms -- Sample_Preservation_Checklist

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.8
Out-of-Control Sample pH Tag



Approved By: 12-15-08 **QA Officer**

Approved By: 12/15/08 **Area Supervisor**

Attachment 6.9
Short Hold Time Analysis List

Short Hold Time Analysis List

Parameter	Hold Time (From Date/Time Sampled)
Inorganics	
Dissolved Oxygen	Field or ASAP
Conductivity	Field or ASAP
Ferrous Iron	Field or ASAP
pH	Field or ASAP
Residual Chlorine	Field or ASAP
Sulfite	Field or ASAP
Coliform, Total and Fecal - Potable Water	30 Hours
Coliform, Total and Fecal - Wastewater	6 Hours
Coliform, Total and Fecal - Non-Potable Water for Compliance Purposes	8 Hours
Coliform, Total and Fecal - Non-Potable Water for Non-Compliance Purposes	24 Hours
Heterotrophic Plate Count - Potable Water	8 Hours
Heterotrophic Plate Count - Non-Potable Water for Compliance Purposes	8 Hours
Heterotrophic Plate Count - Non-Potable Water for Non-Compliance Purposes	24 Hours
Chromium, Hexavalent	24 Hours
BOD	48 Hours
Color	48 Hours
MBAs / Foaming Agents	48 Hours
Nitrogen, Nitrate	48 Hours
Nitrogen, Nitrite	48 Hours
Phosphorus, Ortho	48 Hours
Phosphorus, Dissolved	48 Hours
Turbidity	48 Hours
Organics Prep Lab	
Aldehydes/Formaldehyde - Water	3 Days to extract
Aldehydes/Formaldehyde - Soil	14 Days to Leach, 3 Days after leach to Analyze
DRO - Water 8015	7 Days
DRO - Soil 8015	14 Days
Wisconsin DRO - Water	7 Days
Wisconsin DRO - Soil	10 Days
Semi-Volatiles (BNA/PNA/BN or AN) - Water 8270	7 Days
Semi-Volatiles (BNA/PNA/BN or AN) - Soil 8270	14 Days
PCBs - Water 8082	7 Days
PCBs - Soil 8082	14 Days
Pesticides - Water 8081	7 Days
Pesticides - Soil 8081	14 Days
Herbicides - Water 8151	7 Days
Herbicides - Soil 8151	14 Days
HPLC PNAs - Water	7 Days
HPLC PNAs - Soil	14 Days
Organics in Waste-PCB/Pesticides	14 Days
Organics in Waste-Herbicide	14 Days
Organics in Waste-BNA/PNA/BN or AN	14 Days
Volatiles	
EnCore	48 Hours to Pretreat, 14 Days to Analyze
MeOH Preserved Soil	48 Hours to Pretreat, 14 Days to Analyze
Unpreserved Styrene - Water	7 Days
Unpreserved 2-Chloroethyl Vinyl Ether - Water	7 Days

Short Hold List | Revision 2.1

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.10
Container Packing List (page 1)

TriMatrix Laboratories, Inc.

Container Packing List

For any questions regarding these containers, contact a Project Chemist at (616) 975-4500.

Client: Project: Page 1 of 1

#	Sets	Sample Locations	Sample Container Types and Quantities Requested																										
			0	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
1																													
2																													
3																													
4																													
5																													
6																													
7																													
8																													
9																													
10																													
11																													
12																													
13																													
14																													
15																													
16																													
17																													
18																													
19																													
20																													
Total Containers																													

This container type requires field-filtering

MATRIX	#	TEST	SIZE (mL) / TYPE CONTAINER	OPTIONS	PRESERVATIVE	TAG COLOR
WATER	0	Unpreserved Purgeable Organics	40 mL Clear Glass Vial	40	Cool to 4° C	Yellow & Black Stripe
	1	Preserved Purgeable Organics	40 mL Clear Glass Vial (pre-preserved)	40	HCl; Cool to 4° C	Yellow
	2	Non-Purgeable Organics	1000 mL Amber Glass	1000	Cool to 4° C	Salmon
	3	General Short Hold	Plastic	125, 250, 500, 1000	Cool to 4° C	Green
	4	Nutrients	Plastic	125, 250, 500, 1000	pH <2 w/ H_2SO_4	Dark Blue
	5	Cyanides	500 mL Amber Plastic	500	pH >12 w/ NaOH	Light Blue
	6	Total Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red
	7	Oil & Grease/TPH	Clear Glass	1000WM, 1000NM	pH <2 w/ H_2SO_4	Dark Blue
	8	Bacteria	125 mL Plastic (pre-preserved)	125	$Na_2S_2O_3$; Cool to 4° C	Pre-Labeled (White)
	9	Sulfide	500 mL Amber Glass + NaOH ampule	500	Zinc Acetate at Lab; NaOH in Field	Light Green
	10	TOX	250 mL Amber Glass w/ Septa Lid	250	pH <2 w/ H_2SO_4	Lilac
	11	TOC	40 mL Amber Vial	40	pH <2 w/ H_2SO_4	Pink
	12	DRO	1000 mL Amber Glass	1000	pH <2 w/ HCl	Gray
	13	Phenols	500 mL Amber Glass	500	pH <2 w/ H_2SO_4	Brown
	14	Formaldehyde	250 mL Amber Glass	250	Cool to 4° C	Orange
	15	Dissolved Metals	Plastic	125, 250, 500, 1000	pH <2 w/ HNO_3	Red & White Stripe
SOIL	16	Inorganics/Metals	WM Plastic	125, 250, 500, 1000	Cool to 4° C	White
	17	Non Purgeable Organics	WM Clear Glass	125, 250, 500, 1000	Cool to 4° C	Manila
	18	Purgeable Organics - Bulk	60 mL WM Clear Glass	60	Cool to 4° C	Light Yellow
	19	TCLP Volatiles	125 mL Clear Glass Vial	125	Cool to 4° C	Yellow & Black Stripe
	20	% Solids	125 mL WM Plastic	125	Cool to 4° C	Yellow & White Stripe
	21	Purgeable Organics	Encore Sampler	5g, 25g	Cool to 4° C	Label on Bag
	22	Purgeable Organics - PrePres.	40 mL Pre-Tared Clear Glass Vial + 10 mL MeOH ampule	40	MeOH in field; Cool to 4° C	Pre-Labeled (Light Yellow added at Lab)
MISC	23					
	24					
	25	Pesticide WWs by Method 608	1000 mL Amber Glass	1000	pH 5-9; Cool to 4° C	Yellow & White Stripe
	26	Drinking Water Volatiles	40 mL Clear Glass Vial	40	Ascorbic Acid at Lab; HCl in Field	Yellow

Notes:

DI Water for Equipment Blanks	Container Type and Size	Qty.
VOC Free		
Millipore		
ASTM Metals Free		

Container Packing List New revision 4.4

Approved By: 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.10
Container Packing List (page 2)

TriMatrix Laboratories, Inc.

Project Chemist Initials	Added to Calendar & Folders (initials/date)	Revision:	Revised By/Date:

Client: _____ Project Manager: _____

Project: _____ Contact: _____

TriMatrix Project No: _____ Date of Request: _____

Type of Order: ○ One-Time ⟹ Due to Client: _____ ○ AM ◉ PM

or

◉ Calendar ⟹ Frequency: ○ Weekly ○ Semi-Annually ○ Monthly ○ Annually ○ Quarterly ◉ Daily

Prepare Containers For:

Months	☐ Jan	☐ Feb	☐ Mar	☐ Apr	☐ May	☐ Jun
	☐ Jul	☐ Aug	☐ Sep	☐ Oct	☐ Nov	☐ Dec
Weeks	☐ 1	☐ 2	☐ 3	☐ 4	☐ 5	
Days	☐ M	☐ T	☐ W	☐ TH	☐ F	

Containers will be ◉ Picked Up or ○ Shipped via: ○ First Overnight ◉ Standard Overnight

Pick up/Ship Date: _____ ○ Priority Overnight ○ Express Saver

Ship Containers to: ○ 2-Day ○ Ground

_____ ○ Saturday Delivery ○ TriMatrix Courier

_____ ○ Other: _____

_____

☐ Shipment to be billed to FedEx Account No.:

Telephone No: _____

Shipment to include: ☐ COCs (Qty) ____ ☐ Custody Seals ☐ Temperature Blanks

☐ MSDS Sheets for all preservatives used ☐ WB TM#? ○ Y ◉ N

Comments: ☐ Cooler Banding Required

Assembled by/Date:	Checked by/Date:	Shipped by/Date:

Cooler Number(s) Used:	Coolers Sealed With Tape	Coolers Sealed With Banding Strap	Tracking Number Label(s):
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	
	☐	☐	

TriMatrix Laboratories, Inc. 5560 Corporate Exchange Court, Grand Rapids, MI 49512 (616)975-4500

Container Packing List New revision 4.4

Approved By: PM 12-15-08 **QA Officer** **Approved By:** 12/15/08 **Area Supervisor**

Attachment 6.11
Waste Cabinet Sample Inventory

TriMatrix Laboratories, Inc.

Waste Cabinet Sample Inventory **Waste Shelf ____**

Client Name:	Sample #('s)	Disposal Method	Date

Approved By: DM 12-15-08 **QA Officer**

Approved By: 12/15/08 **Area Supervisor**

Attachment 6.12
pH Strip Calibration Logbook

TriMatrix Laboratories, Inc.

pH Strip Calibration Logbook

Date	Lot #	pH 4	pH 7	pH 10	Area

pH STRIP CALIBRATION CRITERIA CORRECTIVE ACTION

1. The acceptance range for the strips is to read the exact pH of the buffer being checked. The wide range strips must pass this criteria at all three levels, 4, 7, and 10. The narrow range pH 5-7 strips are only checked at a pH of 7.

2. If the pH strips do NOT read at their appropriate levels, that lot number must NOT be used. Return them to purchasing.

file: PHSTRIPS | page number 1 of 12 | revision: 1.1

Approved By: DM 12-15-08 **QA Officer**

Approved By: 12/15/08 **Area Supervisor**

Attachment 6.13
Infra-Red Thermometer Calibration Record

TriMatrix Laboratories, Inc.

INFRA-RED THERMOMETER DECEMBER CALIBRATION RECORD

Date	Time	Initials	NIST Thermometer Reading*	IR Gun Reading	Difference	Acceptable? (<0.50°C Difference)	Comments
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	
12/ /05						Yes / No	

*Calibrated vs. Thermometer # (<0.50°C difference at 4° C vs. NIST Thermometer #97 (serial # 1740)

Approved By: PM 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

Attachment 6.14
Controlled-Temperature Unit Daily Log Sheet

TriMatrix Laboratories, Inc.

Controlled Temperature Unit #32
Daily Log Sheet

Description: Precision Economy Incubator
Model Number: 31750
Serial #: 699091394
Location: Inorganic Laboratory
Purpose: Incubation of Bacteriological Sample Plates
Control Windows: Low: 34.5° C High: 35.5° C
Thermometer #'s: 37, 36
Thermometer Serial #'s: 6187, 6171

Date	Initials	Time of First Readings	First Reading (°C): Top Thermometer	First Reading (°C): Bottom Thermometer	Initials	Time of Second Readings	Second Reading (°C): Top Thermometer	Second Reading (°C): Bottom Thermometer	Adjustments/Observations/Comments

file: NEWTEMP10 page: 14 of 27 revision: 2.2

Approved By: 12-15-08
QA Officer

Approved By: 12/15/08
Area Supervisor

TriMatrix Laboratories, Inc.

UNCONTROLLED COPY

STANDARD OPERATING PROCEDURE

Laboratory Waste Disposal

APPROVALS:

Area Supervisor: _____ Date: 5/22/06
Gary L. Wood

QA Officer: _____ Date: 5-22-06
Tom C. Boocher

Operations Manager: _____ Date: 5/22/06
Jeff P. Glaser

Procedure Number: GR-15-102
Revision Number: 2.1

Date Initiated: 9/30/03
Effective Date: 6/10/06

Date Revised: 5/18/06
Pages Revised: All

By: Marge A. Scott

Total Number of Pages: 18

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires
4-30-09		4-30-10

1.0 SCOPE AND APPLICATION

1.1 Correct disposal of laboratory waste is important from environmental and regulatory standpoints.

1.2 This procedure specifies general criteria used in laboratory waste characterization and disposal.

2.0 PRINCIPLE METHOD REFERENCES

2.1 Hazardous Waste Management Program Administrative Rules Promulgated Pursuant To Part 111, Hazardous Waste Management of Michigan's Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (ACT 451)

2.2 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.20 – 261.24, Identification and Listing of Hazardous Waste

2.3 Code of Federal Regulations, Title 49, *Environmental Protection Agency*, Chapter 1, Part 173.201 – 173.205, Shipping Papers

2.4 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.31, Hazardous Wastes from Non-Specific Sources

3.0 SUMMARY OF PROCEDURE

3.1 Laboratory waste is generated from unused client samples, analysis by-products, used acids, used solvents, used oils and expired chemicals.

3.2 Each waste type requires a specific disposal. Accurate laboratory waste characterization is essential for proper disposal and to prevent safety and/or environmental hazards.

3.3 Most of the laboratory waste is segregated, composited, stored, packed and disposed of by an approved and certified waste disposal company.

3.4 All hazardous waste is manifested by the disposal company, and is required by federal and state regulations.

4.0 PARAMETER OR COMPOUND LIST

4.1 There are no parameters or compounds lists directly associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-01-119, *Toxicity Characteristic Leaching Procedure (TCLP)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 There are no interferences or corrective procedures directly associated with this procedure.

7.0 SAFETY PRECAUTIONS

7.1 Caution must be used at all times when handling laboratory waste.

7.1.1 Most laboratory waste contains hazardous materials.

7.1.2 Most client samples may be corrosive, flammable, reactive or toxic from characteristic materials and/or from preservation chemicals.

7.1.3 Approved personal protective equipment (PPE) must be worn when handling any laboratory waste.

7.2 Personal protective equipment includes approved safety glasses, a laboratory coat and disposable gloves.

7.3 Packaging of individual waste containers into lab-packs for transport and disposal is performed only by the disposal company.

7.4 All TriMatrix employees must read and understand the TriMatrix Safety Manual and Chemical Hygiene Plan. A copy of these documents is given to each new employee at the safety orientation.

7.5 All TriMatrix employees have access to the Material Safety Data Sheet (MSDS) library located on the laboratory intranet.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Client sample disposal is to be done only after the scheduled disposal time in Element unless otherwise specified.

8.2 With pre-approval, extremely hazardous, highly concentrated and/or toxic samples must be returned to the client unless otherwise specified.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Laboratory Information Management System, Promium-ELEMENT™

9.2 Jars, "TCLP", 4-liter glass or plastic

9.3 ELEMENT "Samples Eligible For Disposal" report

9.4 Boxes and/or overpack drums, D.O.T. approved, drums are supplied by the waste hauler

9.5 Waste manifests, MDEQ, supplied by the waste hauler

9.6 Hazardous waste storage and accumulation labels

9.7 Hach EZ-COD Recycling Vessel, 20 gallon, part 2895420

9.8 pH paper, wide-range

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no routine preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Concentrated solutions of acid and sodium hydroxide for pH treatment.

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 ANALYTICAL PROCEDURE

13.1 Analyzed client samples that are ready for disposal are the largest source of laboratory waste. Identify, characterize and dispose of sample waste as follows:

13.1.1 Using ELEMENT™, print a list of all samples eligible for disposal (Attachment 1).

13.1.1.1 At the main menu select "Sample Control", "Disposal" and "Print Samples Eligible for Disposal Report". Refer to Attachment 1 for a report example.

13.1.1.2 Refer to the flow chart provided in Attachment 2.

13.1.2 The list printed in Section 13.1.1 only identifies samples logged into ELEMENT™. Samples not logged in such as those put on hold by a client will not be listed. Check the "hold shelves" but consult the project chemist for disposal instructions of any sample "on hold".

13.1.3 Water or wastewater sample waste can be disposed of by pouring down the drain after pH adjustment unless otherwise specified. Soil, oil, sludge and waste samples must follow strict disposal requirements and cannot be disposed of to the city sewer.

13.1.4 The ELEMENT™ disposal report flags all samples if a contaminant exceeds a regulatory disposal level. A default disposal level of 10,000 ppm will also trigger a flag for non-regulated

contaminants. No flagged sample is to be disposed of through the municipal sewer or landfill (drain or garbage bin).

13.1.5 The federal Environmental Protection Agency (EPA) has classified hazardous waste based on characteristic definitions and a list of toxic contaminants considered to be hazardous to the health and/or environment when present above certain concentrations. TriMatrix Laboratories, as a small quantity waste generator, is required to dispose of any laboratory waste as hazardous which has the characteristic definition of ignitability (D001), corrosivity (D002), or reactivity (D003). The laboratory is also required to dispose of laboratory waste as hazardous if compounds present on the TCLP toxicity characteristic list are present in the waste above the regulatory level. All hazardous waste disposals must be manifested. Disposal of D001-D003 hazardous waste is summarized below. Refer to Attachment 3, the Toxicity Characteristic List, to determine the characteristic of toxicity. A waste is considered hazardous whether it exhibits one characteristic or all. Any sample designated as hazardous is disposed of through the hazardous waste company or is returned to the client. The preference is to return any hazardous sample to the client. However, the project chemist must determine whether this action is appropriate. If a hazardous sample can not be returned to the client, it must be classified as waste and disposed of through the hazardous waste company. When determining whether sample waste is hazardous, check for ignitability, corrosivity and reactivity first since these wastes are specific classifications and are quickly determined. Then, determine characteristic toxicity.

13.1.5.1 Ignitability (D001): A sample must be returned to the client or manifested for disposal as ignitable hazardous waste if it meets any of the following criteria:

13.1.5.1.1 If it is a liquid, other than an aqueous solution containing less than 24 percent alcohol by volume and has a flashpoint less than 60 °C (140 °F).

13.1.5.1.2 It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and when ignited, burns so vigorously and persistently that it creates a hazard.

13.1.5.1.3 It is an ignitable compressed gas.

13.1.5.1.4 It is an ozidizer.

13.1.5.2 Corrosivity (D002): A sample must be returned to the client or manifested for disposal as corrosive hazardous waste if it meets any of the following criteria:

13.1.5.2.1 It is aqueous and has a pH that is less than 2.

13.1.5.2.2 It is aqueous and has a pH that is greater than 12.5.

13.1.5.2.3 It is a liquid and corrodes steel (SAE 1020) at a rate of more than 6.35 mm (0.250 in) per year at a test temperature of 55° C (130° F).

NOTE: The exception to the above is that wastewaters and many other aqueous samples can be treated to neutralize the pH. Disposal can then be through the municipal sewer if no other hazardous classification is found or unless otherwise specified.

13.1.5.3 Reactivity (D003): A sample must be returned to the client or manifested for disposal as reactive hazardous waste if it meets any of the following criteria:

13.1.5.3.1 It is normally unstable and readily undergoes violent change without detonating.

13.1.5.3.2 It reacts violently with water.

13.1.5.3.3 It forms potentially explosive mixtures with water.

13.1.5.3.4 When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

13.1.5.3.5 It is a cyanide or sulfide-bearing waste that, when exposed to pH conditions between 2.0 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

13.1.5.3.6 It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

13.1.5.3.7 It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

13.1.5.3.8 It is a forbidden explosive or it meets the definition of a class 1/division 1.1, 1.2 or 1.3 explosive.

13.1.5.4 Toxicity Characteristics (D004 – D043):

13.1.5.4.1 A sample must be returned to the client or manifested for disposal as toxic hazardous waste if it exhibits the corresponding "D" characteristic of toxicity. The characteristic of toxicity is determined based on the TCLP extract from a representative aliquot containing any TCLP contaminant equal to or greater than the regulatory limit given in Attachment 3. Refer to TriMatrix SOP GR-01-119 for details of the TCLP extraction.

13.1.5.4.1.1 Do not put soil samples confirmed to be D004-D043 hazardous waste into the non-hazardous soils composite drum or the municipal dumpster. The

hazardous classification does not go away by mixing with non-hazardous soils and the drum or dumpster will automatically take on the hazardous classification by association.

13.1.5.4.1.2 If a "totals" analysis is run on a soil sample and the result divided by 20 is less than the TCLP regulatory limit, the sample may be classified as not toxic for the TCLP parameter analyzed.

13.1.5.4.1.3 If a "totals" analysis is run on a soil sample and the result divided by 20 is greater than the regulatory level, the sample remains unclassified for toxicity for the parameter analyzed, but may be presumed as suspect for disposal purposes.

13.1.5.4.1.4 If a "totals" analysis is run on a liquid sample, the result may be presumed to be the TCLP result and compared to the TCLP regulatory limit.

13.1.5.3 Listed Waste (F, K, P, U): A sample must be returned to the client or manifested for disposal as listed hazardous waste if it meets any of the following criteria:

13.1.5.3.1 It is received by the laboratory as a hazardous waste sample from a non-specific source (F-waste).

13.1.5.3.2 It is received by the laboratory as a hazardous waste sample from a specific source (K-waste).

13.1.5.3.3 It is received by the laboratory as a hazardous waste sample from a discarded commercial chemical product or species(P-waste and U-waste).

13.1.5.4 Polychlorinated Biphenyls Waste (PCB): A sample must be returned to the client or manifested for disposal as PCB hazardous waste if it meets any of the following criteria:

13.1.5.4.1 It is received by the laboratory as a hazardous waste sample containing PCBs at any concentration.

13.1.5.4.2 It is analyzed and found to contain PCBs at any concentration.

13.1.5.4.3 Soil or oil waste composites found to contain PCBs at any concentration.

13.1.6 Samples considered non-hazardous may be disposed of by way of the city sewer or municipal dumpster if the following criteria are met:

13.1.6.1 The sample is not ignitable.

13.1.6.2 The sample is not corrosive.

13.1.6.3 The sample is not reactive.

13.1.6.4 The sample has been tested and is not toxic for all TCLP parameters or is verified to be non-toxic for all TCLP parameters through client, project chemist and/or analyst experience.

13.1.6.5 The sample was not received from the client as a listed hazardous waste.

13.1.7 Samples considered hazardous may be returned to the client as follows:

13.1.7.1 Return all samples in the original tagged sample container. Do not remove the sample tag.

13.1.7.2 Wrap sample containers in bubble wrap to prevent breakage, leaking, spilling or vaporization then package in a DOT-approved box or cooler. D.O.T approved containers are specified in 49CFR Ch. 1, Part 173.201-173.205 and generally identified as such on the container.

13.1.7.3 Assure that the following information accompanies the sample if it is not on the sample container label and/or tag:

13.1.7.2.1 The client's name, mailing address, and telephone number

13.1.7.2.2 The laboratory's name, mailing address, and telephone number

13.1.7.2.3 The approximate quantity of the sample.

13.1.7.2.4 The date of shipment.

13.1.7.2.5 A description of the sample.

13.1.7.4 Ship the package in compliance with DOT regulations as a laboratory sample.

13.1.8 Samples not returned to the client and considered or suspected as being hazardous must be disposed of through the hazardous waste disposal company as follows:

13.1.8.1 If a sample is confirmed to be hazardous by any TCLP parameter, D001-D003 characteristic, process listing or contains PCBs, store together with other "like" samples in TCLP jars in the waste storage room until a waste haul is scheduled. Label each TCLP jar with the appropriate internal hazardous waste storage label (Refer to Attachment 4).

13.1.8.2 Non-ignitable, non-corrosive and non-reactive soil sample waste that is presumed to be non-toxic based on the sample source and/or prior client/project chemist/analyst experience is to be compiled in a labeled drum and stored in the flammables cabinet area of the garage. However, before placing in the drum, each sample must be individually tested and found non-hazardous

for D001-D003 characteristics. Forward a copy of the results to the safety officer if not a part of the client-requested analysis. As the drum fills, homogenize the contents by mixing. When full, take a representative sampling and schedule for a full TCLP analysis. If TCLP results on the sampling indicate the contents are non-toxic, the drum may be disposed of through the hazardous waste disposal company as non-notification, non-hazardous material. Forward a copy of the TCLP results to the safety officer to file with the manifest.

13.1.8.3 Non-aqueous liquid sample waste will generally be classified as hazardous based on the client's testing request or sample identification. However, oils and solvents not testing as being hazardous must be tentatively classified based on client information and/or analyst experience before placing in the waste storage room. Oil samples must be checked for polychlorinated biphenyls (PCB) or confirmed by the client to not have PCBs. Forward a copy of the PCB analysis (if not an analysis requested by the client) to the safety officer. Label each sample appropriately for disposal by the hazardous waste disposal company.

13.1.8.4 Aqueous liquid sample waste will generally be classified as wastewater when not found to have a hazardous constituent and can be disposed of by way of the municipal sewer after neutralizing the pH.

13.2 Analysis By-Product Waste

13.2.1 Each analysis by-product waste requires individualized classification for disposal by the hazardous waste disposal company.

13.2.1.1 Chemical Oxygen Demand (COD) By-Products

13.2.1.1.1 COD by-product material contains sulfuric acid and compounds of silver, chromium, and mercury which can be recovered. As vials become available from the analysis, they are to be deposited in the Hach EZ-COD™ recycling vessel. The 20-gallon drum is located in the chemical storage area at the rear of the garage. Full containers of spent COD vials are then picked-up by Heritage Environmental for recycling. This material is not manifested as waste when sent to recycling.

13.2.1.2 Cyanide Distillation Waste

13.2.1.2.1 Cyanide distillation waste is comprised of sulfuric acid. The waste is collected in a 4-liter bottle labeled as sulfuric acid waste. A start date is put on the bottle. When the bottle becomes full, write the filled date on the label and store until the next waste haul. This waste is manifested as Waste Corrosive Liquid, Toxic, NOS, 8(6.1), UN2922, PG II (Sulfuric Acid).

13.2.1.3 Flash Point Waste

13.2.1.3.1 Liquid and solid flash point waste is tested remnants that flash at less than 140° F. Composite and store these remnants in the waste cabinet, in a glass TCLP jar with a PTFE-lined lid. Composite PCB-containing sample remnants separately.

13.2.1.3.2 Disposal will generally be manifested similar to "Waste Solid Containing Flammable Liquid, NOS, 4.1, UN3175, Pg II, (Methanol, Soil)".

13.2.1.4 pH Wastes

13.2.1.4.1 Complile aqueous remnants of pH testing as wastewater and dispose of according to Section 13.1.8.4.

13.2.1.4.2 Compile solid remnants of pH testing as soil and dispose of according to Section 13.1.8.2.

13.2.1.5 Solvent Wastes

13.2.1.5.1 Solvent wastes are generated in the individual lab areas through various processes. Solvents fall into two main categories, halogenated and non-halogenated and are composited as such. Identify the solvent contained in each bottle on the internal hazardous waste storage label. For example, a halogenated solvent bottle containing methylene chloride must be labeled "Halogenated – Methylene Chloride". A non-halogenated bottle containing hexane/toluene must be labeled "Non-Halogenated – Hexane/Toluene". Also label each bottle with a start and end date. The waste is manifested as "Waste Flammable Liquid, Toxic, NOS, 3(6.1), UN1992, PGII, (Acetone, Methylene Chloride)" and "Waste Flammable Liquid, NOS, UN1993, PGII, (Acetone, Toluene, Hexane)".

13.2.1.5.2 Methanol waste from soil extractions in the Volatile Organics laboratory are composited and stored in satellite jars. All containers are labeled as "MeOH waste" and must have an accumulation start and end date.

13.2.1.5.3 Solvent waste containing PCBs (except as specified in Section 13.2.1.7) or chloroform must be composited and stored in separate bottles for disposal. Do not mix PCB waste and chloroform however. Label each bottle with an internal hazardous waste storage label as PCB or Chloroform waste, including the start and end date. Store filled bottles in the flammables waste cabinet. The PCB waste is manifested as "Polychlorinated biphenyls, Liquid, 9, UN2315, PGII, (PCB Samples)" and the chloroform waste as "Waste Chloroform, 6.1, UN1888, Pg II".

13.2.1.6 Acid Wastes

13.2.1.6.1 Different types of acids are used in the laboratory. Collect each type in separate waste bottles and label appropriately (chromerge, sulfuric, or nitric). Also record the start and end date on each bottle. Full bottles must be disposed of through the hazardous waste disposal company. This waste is manifested as "Waste Corrosive Liquid, Toxic, NOS, 8(6.1), UN2922, PGII, (Constituent Acid, Chromic Acid).

13.2.1.7 PCB Wastes

13.2.1.7.1 Any material containing PCBs must be considered hazardous and disposed of as such. Label each bottle as PCB waste, including a start and end date. PCB wastes must only be composited with other PCB waste. ANY material with PCBs must be segregated from other waste and compiled based on compatibility and matrix. The waste is generally manifested as "Polychlorinated Biphenyls, Solid, NOS, 9, UN2315, PGII, (PCB: SOILS)".

13.2.1.8 Semi-Volatile Extracts

13.2.1.8.1 Extract vials are concentrated contaminants from samples analyzed in the extraction and semi-volatile laboratories. Vials are lab-packed and disposed of by the hazardous waste disposal company. However, vials containing any PCB concentration must be stored with the PCB waste (Section 13.2.1.7) for disposal as PCB waste. Non-PCB vials are manifested as "Waste Flammable Liquids, Toxic, NOS, 3, UN1992, PGII, (Methylene Chloride, Hexane)".

13.2.1.9 Outdated Chemicals and Standards

13.2.1.9.1 Outdated chemicals must remain in the original container and be lab-packed by the hazardous waste disposal company for disposal. When removed from the laboratory, store in the flammables cabinet for outdated chemicals until disposal. Manifesting will be at the discretion of the disposal company.

13.2.1.9.2 As an alternative, outdated chemicals may be donated to a credible institution or industrial laboratory without manifesting.

13.2.1.10 Chemical Spills

13.2.1.10.1 Chemical spills must be packed individually and labeled in accordance with the material and hazard class collected.

13.2.1.10.2 Manifesting will be at the discretion of the disposal company.

13.3 As the allotted waste storage space fills, arrangements for a waste haul must be made. TriMatrix uses a certified waste disposal company for all hazardous waste disposals. All hazardous waste manifests are completed by the disposal company as the waste is transferred to the disposal company's truck. Manifest copies are sent to the state of Michigan as required then maintained on file by the safety officer.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Manifesting is described in Section 13.0 and an example is presented as Attachment 5.

14.2 All analyses for the purpose of waste characterization and/or disposal must be copied to the laboratory safety officer for maintaining on file.

15.0 QUALITY ASSURANCE

15.1 There is no quality assurance directly associated with this procedure.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 There is no demonstration of capability study and/or method validation directly associated with this procedure.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

18.3 Follow all instructions in this procedure for laboratory waste disposal requirements.

Approved By: JM 5-22-06 QA Officer | Approved By: 5/22/06 Area Supervisor

19.0 REFERENCES

19.1 Hazardous Waste Management Program Administrative Rules Promulgated Pursuant To Part 111, Hazardous Waste Management of Michigan's Natural Resources and Environmental Protection Act, 1994 PA 451, as amended (ACT 451)

19.2 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.20 – 261.24, Identification and Listing of Hazardous Waste

19.3 Code of Federal Regulations, Title 49, *Environmental Protection Agency*, Chapter 1, Part 173.201 – 173.205, Shipping Papers

19.4 Code of Federal Regulations, Title 40, *Environmental Protection Agency*, Chapter 1, Part 261.31, Hazardous Wastes from Non-Specific Sources

20.0 ATTACHMENTS

20.1 Attachment 1: Samples Eligible for Disposal Report Example

20.2 Attachment 2: Sample Disposal Flowchart

20.3 Attachment 3: Toxicity Characteristic List

20.4 Attachment 4: Internal Hazardous Waste Storage label Example

20.7 Attachment 5: MDEQ Waste Manifest Example

ATTACHMENT 1
Samples Eligible for Disposal Report Example

TriMatrix Laboratories, Inc.

DISPOSAL REPORT -- Page 1 of 4

Printed 5/22/2006, 11:49:20AM

Samples Ready for disposal, received between Apr-24-06 and May-08-06

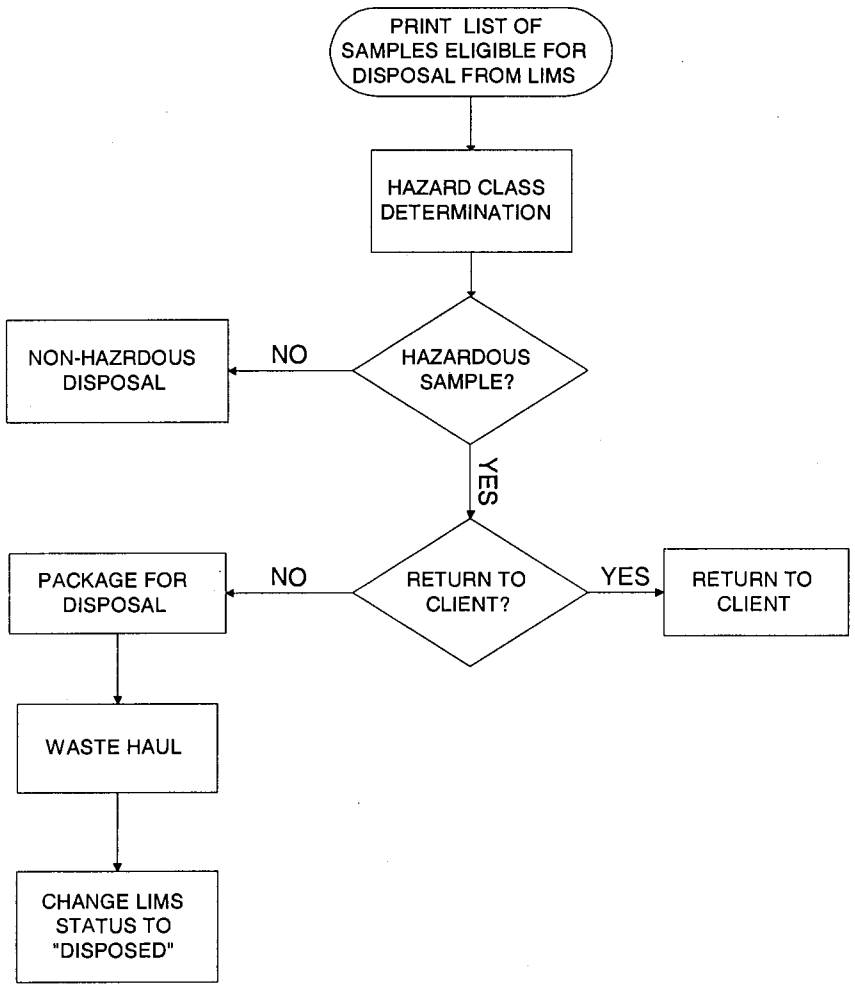
All clients; All projects; All matrices; All containers; All locations

Work Order	Sample	Matrix	Client / Hazard	Project	LabMgr	Status	Status Date
0604418			Taste Control Consultants	Waste Analysis	LMH	Completed	May-02-06 14:05
0604419			Smith & Jones Engineering	Beauford Oil - Emmett Street	LMH	Completed	May-08-06 17:44
0604420			Detroit Machinery - Plant-2	Chem Pond - Monitoring Point 009A	LMH	Completed	May-03-06 12:39
0604423	03	Waste	HJ - Metal Center **Cadmium TCLP [1.6mg/L]**	Waste Characterization	JLR	Completed	May-05-06 14:34
0604433			Smith & Jones Engineering	Fred Oil - Emmett Street	LMH	Completed	May-08-06 17:46
0604434			Smith & Jones Engineering	Yankee Springs	LMH	Completed	May-02-06 18:28
0604435			HJ - Engine Plant	Program 9 - Daily PCB Monitoring	JLR	Completed	Apr-27-06 10:37
0604437			Global Research & Development	PT-02 Quarterly Monitoring	JLR	Completed	May-01-06 10:53
0604438			Jones Associates, Inc.	Total Coliform	GLW	Completed	May-03-06 15:27
0604440			Detroit Machinery - RRPP	River Water Analysis	LMH	Completed	May-02-06 18:48
0604443			AAA Corporation	Wastewater Monitoring, Kentwood Facility	WJR	Completed	May-01-06 12:07
0604446			Hill Valley BPU	Wastewater Monitoring Program	GLW	Completed	May-04-06 14:48
0604447			Worthington Orchard Co.	Weekly Wastewater	LMH	Completed	May-04-06 19:49
0604448			GGH, Inc - Dublin, OH	New Waste Characterization	JLR	Completed	May-09-06 10:26
0604449			Oak Tree Semiconductor Corp.	Monthly Industrial Discharge	JLR	Completed	May-03-06 12:02
0604450			Jones Associates, Inc.	Total Coliform	GLW	Completed	May-03-06 15:27
0604451			Thomas County DPW	First Twp. - Phosphorus Study	JLR	Completed	May-01-06 10:52
0604452			Thomas County DPW	First Township; Permit #MI0036547	JLR	Completed	May-08-06 08:58
0604453			Edison Transmission Plant	Wastewater Monitoring	TLM	Completed	May-03-06 15:36
0604455			Alaska Proving Ground	Weekly 001 Monitoring	TLM	Completed	May-03-06 14:18
0604456			Sherman Clocks	Wastewater Monitoring	JLR	Completed	May-03-06 11:59

dsp_TriMatrix.rpt

Approved By: 5-22-06 **QA Officer** **Approved By:** 5/22/06 **Area Supervisor**

ATTACHMENT 2
Sample Disposal Flowchart



Approved By: 5-22-06 **Approved By:** 5/22/06
QA Officer **Area Supervisor**

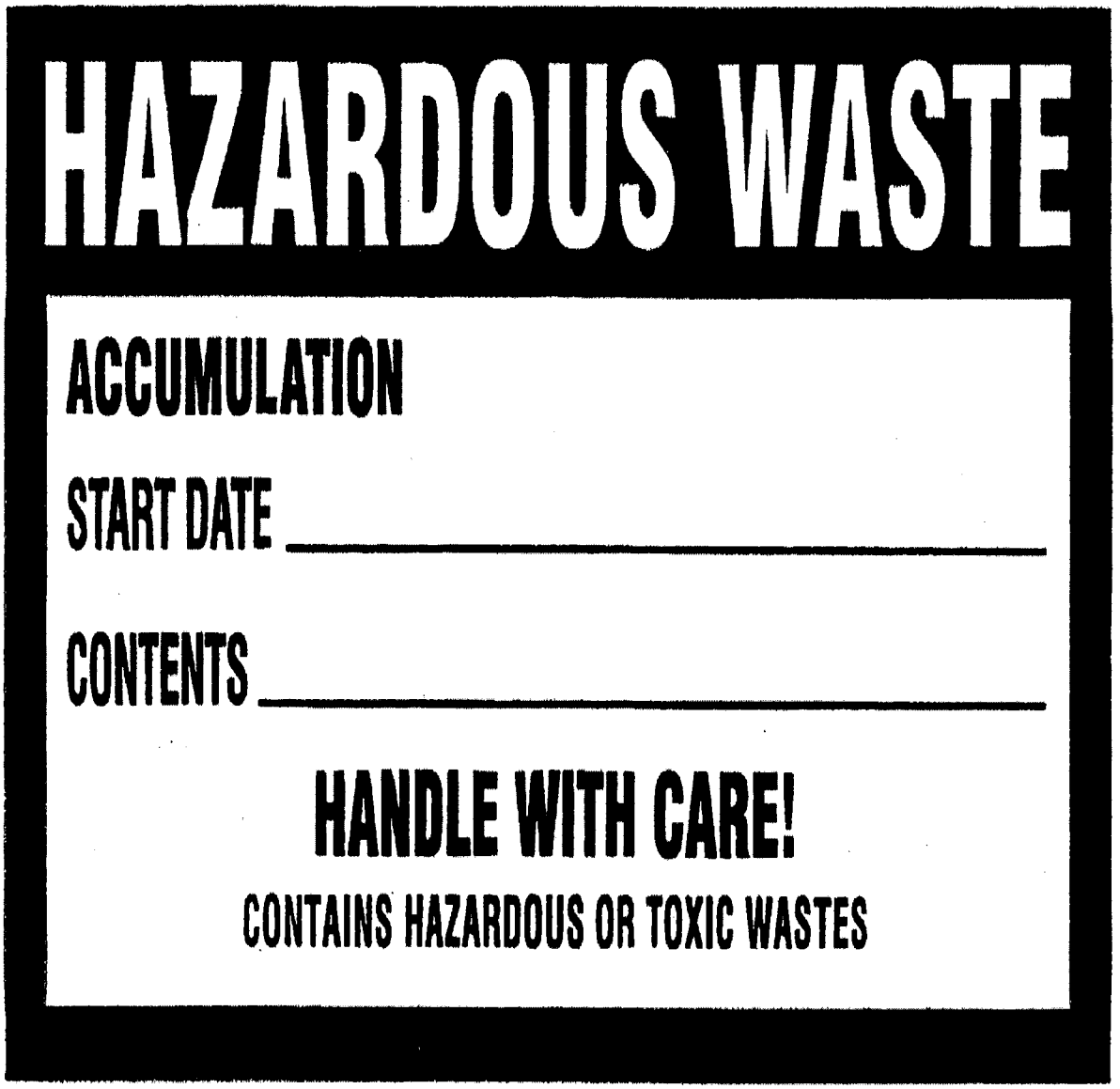
ATTACHMENT 3
Toxicity Characteristic List

EPA Hazardous Waste #	Contaminant	Total Regulatory Level (mg/kg)	TCLP Level (mg/L)
D004	Arsenic	100	5.0
D005	Barium	2000	100.0
D018	Benzene	10	0.5
D006	Cadmium	20	1.0
D019	Carbon Tetrachloride	10	0.5
D020	Chlordane	.60	0.03
D021	Chlorobenzene	2000	100.0
D022	Chloroform	120	6.0
D007	Chromium	100	5.0
D023	o-Cresol	4000	200.0
D024	m-Cresol	4000	200.0
D025	p-Cresol	4000	200.0
D026	Cresol	4000	200.0
D016	2,4,-D	200	10.0
D027	1,4-Dichlorobenzene	150	7.5
D028	1,2-Dichloroethane	10	0.5
D029	1,1-Dichloroethylene	14	0.7
D030	2,4-Dinitrotoluene	2.60	0.13
D012	Endrin	.40	0.02
D031	Heptachlor	.160	0.008
D032	Hexachlorobenzene	22.6	1.13
D033	Hexachlorobutadiene	10.0	0.5
D034	Hexachloroethane	60.0	3.0
D008	Lead	100.0	5.0
D013	Lindane	8.0	0.4
D009	Mercury	4.0	0.2
D014	Methoxychlor	200.00	10.0
D035	Methyl Ethyl Ketone	4000.00	200.0
D036	Nitrobenzene	40.0	2.0
D037	Pentachlorophenol	2000.0	100.0
D038	Pyridine	100.0	5.0
D010	Selenium	20.0	1.0
D011	Silver	100.0	5.0
D039	Tetrachloroethylene	14.0	0.7
D015	Toxaphene	10.0	0.5
D040	Trichloroethylene	10.0	0.5
D041	2,4,5-Trichlorophenol	8000	400.0
D042	2,4,6-Trichlorophenol	40.0	2.0
D017	2,4,5-TP (Silvex)	20.0	1.0
D043	Vinyl Chloride	4.0	0.2
	Cyanide	320	16
	PCBs (polychlorinated biphenyls)	<50 mg/kg from the source	

Approved By: 5-22-06 QA Officer

Approved By: 5/22/06 Area Supervisor

ATTACHMENT 4
Internal Hazardous Waste Storage Label Example



Lab Safety Supply Inc

Reorder No. 620

Approved By: TM 5-22-06
QA Officer

Approved By: 5/22/06
Area Supervisor

ATTACHMENT 5
MDEQ Waste Manifest Example

DEQ WASTE MANAGEMENT DIVISION
MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY

DO NOT WRITE IN THIS SPACE
ATT. ☐ DIS. ☐ REJ. ☐ PR. ☐

Required under authority of Part 111 and Part 121 of Act 451, 1994, as amended.

Failure to file may subject you to criminal and/or civil penalties under Sections 324.11151 or 324.12116 MCL.

Please print or type. Form Approved. OMB No. 2050-0039

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No.: MIR 000 041 632
Manifest Document No.: 02640
2. Page 1 of 1
Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address: TriMatrix Laboratories, Inc.
5560 Corporate Exchange Court
Grand Rapids, MI 49512
4. Generator's Phone ((616)) 975-4500

A. State Manifest Document Number: MI 10102640
B. State Generator's ID:

5. Transporter 1 Company Name: Drug & Laboratory Disposal, Inc.
6. US EPA ID Number: MID092947928
C. State Transporter's ID:
D. Transporter's Phone: (269) 685-9824

7. Transporter 2 Company Name:
8. US EPA ID Number:
E. State Transporter's ID:
F. Transporter's Phone:

9. Designated Facility Name and Site Address: Drug & Laboratory Disposal, Inc.
331 Broad Street
Plainwell, MI 49080-1439
10. US EPA ID Number: MID092947928
G. State Facility's ID:
H. Facility's Phone: (269) 685-9824

	11. US DOT Description (including Proper Shipping Name, Hazard Class, and ID NUMBER).	HM	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	I. Waste No.
a	WASTE FLAMMABLE LIQUID, n.o.s., 3, UN1993, PGII (ACETONITRILE, METHANOL)	X	1	DF	80	P	F003
b	WASTE FLAMMABLE LIQUID, TOXIC, n.o.s., 3(6.1), UN1992, PGII (ACETONE, METHYLENE CHLORIDE) RQ=D001	X	3	DF	320	P	F003
c	WASTE CHLOROFORM, 6.1, UN1888, PGIII RQ=10#	X	1	DF	50	P	D022
d	WASTE SOLID CONTAINING FLAMMABLE LIQUID, n.o.s., 4.1, UN3175, PGII (METHANOL, SOIL) RQ=D001	X	1	DF	130	P	F003

J. Additional Descriptions for Materials Listed Above
a) LP, D001
b) LP, F002, D001, D022
c) LP
d) LP, D001

K. Handling Codes for Wastes Listed Above
a.
b.
c.
d.

15. Special Handling Instructions and Additional Information
In case of accident, contain material and call (269) 685-9825. ERG guidebook on vehicle. DLD has the appropriate permits for, and will accept, the above wastes.

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations. If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR; if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

GENERATOR — Printed/Typed Name: Joshua Frank — Signature: Joshua Frank — Date: Month 01 Day 20 Year 06

TRANSPORTER — 17. Transporter 1 Acknowledgment of Receipt of Materials — Printed/Typed Name: Kyle Goodwin — Signature: Kyle Goodwin — Date: Month 01 Day 20 Year 06

18. Transporter 2 Acknowledgment of Receipt of Materials — Printed/Typed Name: — Signature: — Date: Month Day Year

FACILITY — 19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.
Printed/Typed Name: Kevin Jay Barghuis, CHMM — Signature: — Date: Month Day Year

ALL SPILLS MUST BE REPORTED TO THE MICHIGAN POLLUTION EMERGENCY ALERTING SYSTEM. IN MICHIGAN AT 1-800-292-4706 OR OUT OF STATE AT 517-373-7660 AND THE NATIONAL RESPONSE CENTER AT 1-800-424-8802 24 HOUR PER DAY.

EPA Form 8700-22 (Rev. 9/88) — To be mailed by Generator to: WASTE MANAGEMENT DIVISION, MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY, PO BOX 30038, LANSING MI 48909-7538 — EQP 5110 Rev. 7/03

Approved By: 5-22-06 QA Officer — Approved By: 5/22/06 Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Total and Amenable Cyanide Extraction and/or Macro-Distillation

SW-846 Method 9010C
SW-846 Method 9013A

APPROVALS:

Area Supervisor: Heather L. Brady Date: 11-13-09
Heather L. Brady

QA Officer: Date: 11-13-09
Tom C. Boocher

Operations Manager: Date: 11/13/09
Jeff P. Glaser

Procedure Number: GR-18-106
Revision Number: 3.2

Date Initiated: 5/12/93
Effective Date: 11/25/09

Date Revised: 11/12/09
Pages Revised: All

By: Tom C. Boocher

Total Number of Pages: 17

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure is for the extraction and/or macro-distillation of soluble cyanide salts and many insoluble cyanide complexes. An extraction for oily, solid and multiphasic samples containing so much solid or solids of such a size as to interfere with agitation and homogenization of the sample mixture in the distillation flask prior to the distillation is included.

1.2 The applicable concentration range is 0.05 to 10 mg/kg cyanide (dry weight).

1.3 The nominal reporting limit for soil and sludge samples is 0.05 mg/kg but varies based on percent solids. Waste samples are reported on an "as received" basis.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IIIB, Revision 3, November, 2004, Method 9010C, "Total and Amenable Cyanide Distillation"

2.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* other methods, Revision 1, November, 2004, Method 9013A, "Cyanide Extraction Procedure for Solids and Oils"

3.0 SUMMARY OF PROCEDURE

3.1 If a sample contains so much solid or solids of such a size as to interfere with agitation and homogenization of the sample mixture in the distillation flask or so much oil or grease as to interfere with the formation of a homogeneous emulsion, the sample is extracted with water at pH 10 or greater and the extract distilled and analyzed in accordance with TriMatrix SOP GR-05-122.

3.2 Samples of such a size as to interfere with agitation and homogenization of the sample mixture in the distillation flask or have so much oil or grease as to interfere with the formation of a homogeneous emulsion and that contain free water are filtered and separated into an aqueous component and a combined oil/solid component. The oil/solid component is extracted and an aliquot of the aqueous extract is combined with an aliquot of the filtrate in proportion to the sample composition. Alternatively, sample components may be analyzed separately and cyanide reported for each component.

3.3 If sample solids are known to contain sufficient levels of cyanide (≥50 mg/kg) as to be well above the method detection limit (MDL), the extraction step may be deleted and the solids distilled directly by diluting a small aliquot of the high-concentration solid (1-10 g) in 500 mL of water in the distillation flask and suspending the slurry during distillation.

3.4 Distillation of the sample or sample extract then releases cyanide as hydrogen cyanide gas (HCN) by the addition of sulfuric acid which is collected in a sodium hydroxide (NaOH) scrubber trap.

3.5 The NaOH trap solution is then analyzed in accordance with TriMatrix SOP GR-05-122.

4.0 PARAMETER OR COMPOUND LIST

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4.1 Total or Extractable Cyanide

4.2 Total or Extractable Amenable Cyanide

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-05-122, *Total Cyanide by Konelab UV Spectrophotometry*, latest revision

5.2 TriMatrix SOP GR-15-100, *Laboratory Waste Disposal*, latest revision

5.3 TriMatrix SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.4 TriMatrix SOP GR-09-128, *Homogenization, Grinding and Drying of Solid Samples*, latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Most interfering substances are removed by the distillation.

6.2 Sulfides adversely affect the colorimetric analysis. Sulfides are removed by the addition of bismuth nitrate prior to distillation.

6.3 Results are biased high for samples containing nitrate and/or nitrite without pre-treatment. During distillation, nitrate and nitrite forms nitrous acid which reacts with some organic compounds to form oximes. These compounds decompose to generate HCN. Nitrate and nitrite interference is eliminated by pre-treatment with sulfamic acid.

6.4 Oxidizing agents can cause negative interference by destroying cyanide-containing compounds. This interference is removed by treating with ascorbic acid.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 Heating mantles are hot during use. Avoid contact to prevent burns.

7.3 This procedure involves acidifying samples that potentially contain high levels of cyanide. The acidification and distillation must be carried out under a fume hood.

7.4 Avoid contact with laboratory chemicals and solutions. Especially avoid contact with sulfuric acid and sodium hydroxide.

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7.5 Approved safety glasses, disposable gloves and a laboratory coat are mandatory when working in the wet chemistry laboratory.

7.6 Potassium cyanide (KCN) is a violent poison. Avoid any type of contact. Do not breathe KCN dust or vapor. Always open the container and perform all dilutions in a fume hood. Keep potassium cyanide away from acids. If necessary, use a particle face mask when working with the chemical to further minimize exposure.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Waste, sludge and soil samples are to be collected in inert bottles with no preservative added.

8.2 Store all samples at 0 - 6° C until analysis.

8.3 Homogenize waste, sludge and soil samples thoroughly inside a laboratory hood, in accordance with TriMatrix GR-09-128, prior to sub-sampling for extraction and/or distillation.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Distillation apparatus is shown in Attachment 20.1 and contains parts as follows:

9.1.1 Distilling flask, 1 L in volume

9.1.2 Inlet tube

9.1.3 Condenser with cooling water

9.1.4 Hydrogen cyanide (HCN) scrubber trap

9.1.5 Low-vacuum source

9.2 Volumetric flasks, various sizes, type A

9.3 Volumetric pipets, various sizes, type A

9.4 Autopipetter, confirmed traceable to NIST standards at each volume measured

9.4 Balance, top-loading, readable to 0.01 g

9.5 Graduated cylinders, various sizes

9.6 TCLP Tumbler – This device sufficiently agitates a 1 L sealed glass container. For the purpose of this analysis, agitation is sufficient when:

9.6.1 All sample surfaces are continuously brought into contact with the extraction fluid.

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9.6.2 The agitation prevents stratification of the sample and extraction fluid.

9.7 Buchner funnel apparatus:

9.7.1 Buchner funnel, 500 mL capacity with 1-liter vacuum filtration flask.

9.7.2 Filters, 0.45 µm, glass fiber

9.7.3 Vacuum source

9.8 Separatory funnels, glass, 500 mL or larger

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 After each batch, wipe all distillation heating mantles and counter areas clean.

10.2 Carefully inspect each part of the distillation glassware before use. Cracked or chipped glassware must be repaired and/or replaced. Do not use cracked or chipped glassware.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory Reagent Water, ASTM Type II, Milli-Q water system

11.2 Sodium Hydroxide (NaOH), ACS grade pellets or better

11.3 Bismuth Nitrate pentahydrate ($Bi(NO_3)\cdot 5H_2O$), certified reagent grade powder or better

11.4 Concentrated Sulfuric Acid (H_2SO_4), technical grade or better

11.5 Potassium Cyanide (KCN), ACS grade crystals or better

11.6 Magnesium Chloride, hexahydrate ($MgCl_2\cdot 6H_2O$), ACS grade crystals or better

11.7 Sulfamic Acid (NH_2SO_3H), certified reagent grade crystalline powder or better

11.8 Potassium iodide/starch test paper

11.9 Prepare sodium hydroxide solution (1.25N) as follows:

11.9.1 Dissolve 50.0 g of sodium hydroxide pellets into a 1000 mL volumetric flask partially filled with laboratory reagent water.

11.9.2 Dilute to volume with laboratory reagent water after the solution cools.

11.9.3 Expiration is six months from the date made if stored tightly capped in an inert plastic or glass container.

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11.10 Prepare bismuth nitrate solution as follows:

11.10.1 Dissolve 30.0 g of $Bi(NO_3)_3 \cdot 5H_2O$ into a 1000 mL volumetric flask filled with approximately 100 mL reagent water. Stir until dissolved.

11.10.2 While stirring, add 250 mL of glacial acetic acid.

11.10.3 Dilute to volume with laboratory reagent water.

11.10.4 Expiration is six months from the date made if stored tightly capped in an inert plastic or glass container.

11.11 Prepare magnesium chloride solution as follows:

11.11.1 Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1000 mL volumetric flask partially filled with laboratory reagent water.

11.11.2 Stir to dissolve, dilute to volume with reagent water.

11.11.3 Expiration is six months from the date made if stored tightly capped in an inert plastic or glass container.

11.12 Prepare sulfamic acid solution as follows:

11.12.1 Dissolve 40 g of NH_2SO_3H in a 1000 mL volumetric flask partially filled with laboratory reagent water.

11.12.2 Dilute to volume with reagent water.

11.12.3 Expiration is six months from the date made if stored tightly capped in an inert plastic or glass container.

11.13 Glacial Acetic Acid ($C_2H_4O_2$), ACS grade or better

11.14 Anhydrous Sodium Acetate ($C_2H_3O_2Na$): ACS grade

11.15 Prepare acetate buffer solution (pH = 4.0) as follows:

11.15.1 Dissolve 7.3 g anhydrous sodium acetate in a 50 mL volumetric flask filled with approximately 20 mL laboratory reagent water.

11.15.2 Add 24 g glacial acetic acid then dilute to volume with laboratory reagent water.

11.15.3 Expiration is six months from the date made if stored tightly capped in an inert plastic or glass container.

11.16 Lead Acetate Paper

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11.17 Sodium hydroxide solution (50%, w/v), prepare as follows (in a fume hood):

11.17.1 Weigh out 150 g of sodium hydroxide (NaOH) pellets on a top-loading balance in a 1000 mL beaker.

11.17.2 Slowly add laboratory reagent water to the beaker until the pellets dissolve then add laboratory reagent water to the 300 mL mark on the beaker. Be careful not to add water so fast that splashing occurs from the heat of dissolution.

11.17.3 After cooling (on the bench and in the hood), transfer to a glass container and store at room temperature tightly capped.

11.17.4 Expiration is 6 months from the date prepared.

11.18 Hexane, pesticides grade or better

12.0 STANDARDS PREPARATION

12.1 Any sample treated with bismuth nitrate for sulfide interference (Section 13.5) must be quantitated using a calibration curve run with standards that have also been treated with bismuth nitrate.

12.2 Standards preparation is described in TriMatrix SOP GR-05-122, Section 12.0.

13.0 ANALYTICAL PROCEDURE

13.1 If a sample contains so much solid or solids of such a size as to interfere with agitation and homogenization of the sample mixture in the distillation flask or contains so much oil or grease as to interfere with the formation of a homogeneous emulsion, and does not contain any free aqueous phase, extract as follows:

13.1.1 Place the following in a 1 L wide-mouthed glass bottle:

13.1.1.1 500 mL laboratory reagent water

13.1.1.2 5 mL of 50% w/v sodium hydroxide (NaOH) solution

13.1.1.3 50 mL n-Hexane (only if a heavy grease is present)

13.1.1.4 A representative sample aliquot of 25 g (based on dry weight), determined as follows:

$$\text{grams needed} = \frac{0.005 \text{ mg/L} \times 250 \text{ mL} \times 100}{A \times B}$$

Where:

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A = Reporting limit, in mg/kg (dry weight)
B = Sample percent solids

13.1.2 Cap the bottle.

13.1.3 The extract pH must be maintained above 10 throughout the extraction and subsequent filtration. Since some samples may release acid, monitor the pH as follows:

13.1.3.1 Shake the extraction bottle and after one minute, check the pH.

13.1.3.2 If the pH is below 12, add 50% NaOH in 5 mL increments until it is at least 12.

13.1.3.3 Recap the bottle and repeat until the pH does not drop below 12.

13.1.4 Place the capped bottle in the TCLP tumbler, making sure there is enough foam insulation to cushion the bottle. Turn the tumbler on and allow the extraction to run for about 16 - 18 hours.

13.1.5 Prepare a Buchner funnel with a glass fiber filter and decant the extract to the Buchner funnel. Full recovery of the extract is not necessary. However, filter at least 250 mL unless the cyanide concentration is expected to be high.

13.1.6 If the extract contains an oil phase and/or hexane was added before extracting, separate the aqueous phase using a separatory funnel. Neither the separation nor the filtration are critical but are necessary to be able to measure the aliquot volume of the aqueous extract analyzed. Small amounts of suspended solids and oil emulsions will not interfere with the distillation or analysis.

13.1.7 Proceed to Section 13.5 for the distillation step.

13.2 If the sample is a homogeneous fluid or slurry that does not separate or settle in the distillation flask when mixing so the solids are entirely suspended then distill as a water without the solids extraction step by using 250 mL of sample and 250 mL of laboratory reagent water to distill a 500 mL total volume. Refer to Section 13.5 for the distillation step.

13.3 If the sample is multi-phasic and contains so much solid or solids of such a size as to interfere with agitation and homogenization of the sample mixture in the distillation flask or contains so much oil or grease as to interfere with the formation of a homogeneous emulsion, extract as follows:

13.3.1 Assemble a Buchner funnel apparatus. Insert a weighed glass fiber filter (weigh the pad then place it in the funnel). Turn the vacuum on and wet the pad with a known volume of water.

13.3.2 Transfer the sample to the Buchner funnel in small aliquots, first decanting the fluid. Rinse the sample container with known volumes of water and add the rinses to the Buchner funnel. When no free water remains in the funnel, slowly open the stopcock to allow air to enter the vacuum flask. A small amount of sediment may have passed through the glass fiber filter but this will not interfere with the analysis.

13.3.3 Transfer the solid and glass fiber filter to a tared weighing dish. Since most greases and oils will not pass through the filter, solids, oils and greases can be extracted together. If the filtrate

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includes an oil phase, transfer the filtrate to a separatory funnel. Collect and measure the volume of the aqueous phase. Transfer the oil phase to the weighing dish with the solid.

13.3.4 Weigh the dish containing solid, oil (if any) and filter. Subtract the mass of the dry filter pad. Calculate the net volume of water present in the original sample by subtracting the total volume of rinses used from the measured volume of the filtrate.

13.3.5 Place the following in a 1 L wide-mouthed glass bottle:

13.3.5.1 500 mL laboratory reagent water

13.3.5.2 5 mL 50% w/v NaOH

13.3.5.3 50 mL n-Hexane (if a heavy waste-insoluble grease is present)

13.3.5.4 If the mass of the solids is greater than 25 g, weigh out a representative aliquot of 25 g and add it to the bottle; otherwise add all of the solids and the filter. Cap the bottle tightly.

13.3.6 Maintain the extract pH above 10 throughout the extraction and subsequent filtration. Since some samples may release acid, monitor the pH as follows:

13.3.6.1 Shake the extraction bottle and after one minute, check the pH.

13.3.6.2 If the pH is below 12, add 50% w/v NaOH in 5 mL increments until it is at least 12.

13.3.6.3 Recap the bottle and repeat until the pH does not drop below 12.

13.3.7 Place the bottle in the TCLP tumbler, making sure there is enough cushioning foam insulation. Turn the tumbler on and allow the extraction to run for 16 – 18 hours.

13.3.8 Assemble a Buchner funnel with a glass fiber filter.

13.3.9 Decant the extract to the Buchner funnel. Full recovery of the extract is not necessary.

13.3.10 If the extract contains an oil phase, separate the aqueous phase using a separatory funnel. Neither the separation nor filtration are critical but are necessary to be able to measure the volume of the aqueous extract analyzed. Small amounts of suspended solids and oil emulsions will not interfere.

13.3.11 At this point, an aliquot of the filtrate from the original sample may be combined with an aliquot of the solids extract in volumes proportional and representative of the sample, as the following equation illustrates. The distillation step is in Section 13.5. Alternatively, each may be distilled and analyzed separately and concentrations reported for each phase if specified.

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$$\frac{\text{Liquid Sample Aliquot (mL)}}{\text{Extract Aliquot(mL)}} = \frac{\text{Solid Sample Mass extracted (g)}}{\text{(Solids \& oil phase) - (dry weight filter + tared dish) (g)}} \times \frac{\text{Filtrate Volume including rinses (mL)}}{\text{500 mL extraction fluid + NaOH (mL)}}$$

13.4 Pretreatment for cyanides amenable to chlorination if distilling for amenable cyanides is as follows:

13.4.1 The amenable cyanide pretreatment must be performed under amber light since $K_3[Fe\text{-}(CN)_6]$ decomposes under UV light and hence will test positive for cyanide amenable to chlorination if exposed to fluorescent lighting or sunlight. Two identical sample aliquots are required to determine cyanides amenable to chlorination. One treated and one untreated.

13.4.2 To 250 mL sample or solid/oil extract diluted to 500 mL, add calcium hypochlorite solution dropwise while agitating and maintaining the pH between 11 and 12 with 1.25M sodium hydroxide until an excess of chlorine is present as indicated by potassium iodide (KI) starch paper turning blue. The sample will be subjected to alkaline chlorination by this step.

Caution: The initial alkaline chlorination reaction product is cyanogen chloride. This reaction MUST be performed in a fume hood.

13.4.3 Test for excess chlorine with KI starch paper and maintain an excess for one hour with continuous agitation. A distinct blue color on the test paper indicates a sufficient chlorine level. If necessary, add additional calcium hypochlorite solution.

13.4.4 After one hour, add 0.2 g of L-ascorbic acid until KI-starch paper indicates no residual chlorine. Then add 0.2 g of L-ascorbic acid to ensure the presence of excess reducing agent. This is the chlorinated aliquot.

13.4.5 Distill then analyze for total cyanide in the chlorinated and the unchlorinated sample aliquots in accordance with TriMatrix SOP GR-05-122. The difference between the chlorinated and unchlorinated aliquots is equal to the cyanide amenable to chlorination concentration.

13.5 The distillation is as follows:

13.5.1 Place 250 mL of extract or up to 25 g sample diluted to 500 mL in the one liter distillation flask.

13.5.2 Pipet 50 mL of 1.25M sodium hydroxide into the gas scrubber. Connect the flask, condenser, gas scrubber and vacuum trap.

13.5.3 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately two bubbles of air per second enter the boiling flask through the air inlet tube.

13.5.4 Use potassium iodide starch test paper dampened with acetate buffer to test for oxidizing agents. If the test paper turns blue, add 0.2 ±0.1 g ascorbic acid powder then stir and test again. Repeat until the paper no longer turns blue then add 0.05 ±0.1 g of additional ascorbic acid powder.

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13.5.5 If a sample is known or suspected to contain sulfide, add 50 mL of 0.062M bismuth nitrate solution through the air inlet tube. Mix for three minutes. Use lead acetate paper to check the sample for the presence of sulfides. A positive test is indicated by a black color on the paper.

Note: Associated quality control samples must also be treated when this step is used.

13.5.6 If a sample is known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 50 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

Note: Associated quality control samples must also be treated when this step is used.

13.5.7 Slowly add 25 mL of concentrated sulfuric acid through the air inlet tube. Rinse the tube with water and allow the airflow to mix the flask contents for three minutes.

13.5.8 Add 20 mL of 2.5M magnesium chloride through the air inlet and wash the inlet tube with a stream of water.

13.5.9 Heat the solution to boiling then reflux for one hour. Turn off the heat and continue the airflow for at least 15 minutes. After cooling the flask and closing the vacuum source, disconnect the gas scrubber.

13.5.10 Transfer the scrubber solution to a 250-mL volumetric flask, rinse the scrubber into the volumetric flask and dilute to volume with laboratory reagent water.

13.5.11 Proceed to the cyanide analysis in accordance with TriMatrix SOP GR-05-122.

Note: If distillates are not analyzed immediately, store tightly sealed away from light.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Record all data in Element™ for each batch distilled, including the following.

14.1.1 Sample mass distilled, in g

14.1.2 Spiking solution concentrations, in mg/L

14.1.3 Spiking solution volume used, in mL

14.1.4 Analyst initials

14.1.5 Date of distillation

14.1.6 Batch number

14.2 Refer to Attachment 20.2 for an Element™ Preparation Batch Report example.

Approved By: pos 11-13-09 **QA Officer** **Approved By:** Heather L Brady **Area Supervisor**

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15.0 QUALITY ASSURANCE

15.1 Method and matrix QC must be distilled for each batch. A batch consists of a maximum of 20 samples per matrix and may be left open for up to 14 days.

15.1.1 Distillation QC consists of two Blank Spikes (BS), a Method Preparation Blank (BLK) each day samples are distilled and each time a new batch is opened, and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) for each batch of up to 20 samples.

15.1.1.1 The BS is an aliquot of stock standard spiked into up to 25 g of Ottawa sand and 500 mL of laboratory reagent water, distilled and analyzed like a sample.

15.1.1.1.1 To prepare the low-concentration BS, measure 25 g of Ottawa sand and 500 mL of laboratory reagent water into a 1 L distillation flask. Pipet 125 µL of 100 mg/L cyanide standard directly into the bottle then distill as a sample. The BS concentration is 0.5 mg/kg cyanide based on a 25 g sample distillation.

$$\frac{100\text{ mg}}{\text{L}}\ \frac{0.125\text{ mL}}{25\text{ g}} = 0.5\text{ mg/kg CN}^-$$

15.1.1.1.2 To prepare the high concentration blank spike, measure 25 g Ottawa sand and 500 mL of laboratory reagent water into a 1 L distillation flask. Pipet 250 µL of 100 mg/L cyanide standard directly into the flask then distill as a sample. The BS concentration is 1.0 mg/kg cyanide based on a 25 g sample distillation.

$$\frac{100\text{ mg}}{\text{L}}\ \frac{0.250\text{ mL}}{25\text{ g}} = 1.0\text{ mg/kg CN}^-$$

15.1.1.1.3 To prepare matrix spikes, measure up to 25 g of sample (using approximately the same amount for the MSD) and 500 mL of laboratory reagent water into a 1 L distillation flask. Pipet 250 µL of 100 mg/L cyanide standard directly into the flask then distill as a sample. The MS and/or MSD spiked concentration is 1.0 mg/kg cyanide based on 25 g sample distillation.

$$\frac{100\text{ mg}}{\text{L}}\ \frac{0.250\text{ mL}}{25\text{ g}} = 1.0\text{ mg/kg CN}^-$$

15.1.1.2 The BLK is 25 g Ottawa sand and 500 mL of laboratory reagent water distilled as a sample. Calculate the result based on a 25 g sample.

Note: Place QC randomly in the distillation setup. Do not use the same position each time.

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15.1.2 Extraction QC consists of Blank Spike (BS), a Method Preparation Blank (BLK) each day samples are extracted and each time a new batch is opened, and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) for each batch of up to 20 samples.

15.1.2.1 The BS is an aliquot of stock standard spiked into laboratory reagent water, extracted, filtered, distilled and analyzed like a sample.

15.1.2.1.1 To prepare the blank spike, measure 25 g of Ottawa sand and 500 mL of laboratory reagent water into a 1 L extraction bottle. Pipet 250 µL (or 50 µL for available cyanide) of 100 mg/L cyanide standard directly into the bottle then extract, filter and distill as a sample. The BS concentration is 1.0 mg/kg cyanide (or 0.2 mg/kg for available cyanide) based on a 25 g sample extraction and 250 mL extract distillation.

$$\frac{100\text{ mg}}{1\text{ L}} \Bigg| \frac{0.250\text{ mL}}{500\text{ mL}} = 0.05\text{ mg/L } CN^- \text{ at instrument}$$

$$\frac{0.05\text{ mg}}{1000\text{ mL}} \Bigg| \frac{250\text{ mL}}{250\text{ mL}} \Bigg| \frac{500\text{ mL}}{25\text{ g}} \Bigg| \frac{1000\text{ g}}{\text{kg}} = 1.0\text{ mg/kg } CN^- \text{ in sample}$$

15.1.2.2 The MS is an aliquot of stock standard spiked into no more than 25 g of sample and 500 mL of laboratory reagent water, extracted, and filtered, distilled and analyzed like a sample. The MSD is identical with approximately the same sample mass used.

15.1.2.2.1 To prepare the matrix spike, measure 25 g of sample and 500 mL of laboratory reagent water into a 1 L extraction bottle. Pipet 250 µL (or 50 µL for available cyanide) of 100 mg/L cyanide standard directly into the bottle then extract, filter and distill as a sample. The spiked concentration is 1.0 mg/kg cyanide (or 0.2 mg/kg for available cyanide) based on a 25 g sample extraction and 250 mL extract distillation.

$$\frac{100\text{ mg}}{1\text{ L}} \Bigg| \frac{0.250\text{ mL}}{500\text{ mL}} = 0.05\text{ mg/L } CN^- \text{ at instrument}$$

$$\frac{0.05\text{ mg}}{1000\text{ mL}} \Bigg| \frac{250\text{ mL}}{250\text{ mL}} \Bigg| \frac{500\text{ mL}}{25\text{ g}} \Bigg| \frac{1000\text{ g}}{\text{kg}} = 1.0\text{ mg/kg } CN^- \text{ in sample}$$

15.1.2.3 The BLK is 25 g of Ottawa sand and 500 mL of laboratory reagent water extracted, filtered and distilled as a sample. Calculate the result based on a 25 g sample extraction.

Note: If the sample being spiked has a high cyanide concentration, adjust the sample aliquot used to less than 25 g and/or spike at a higher concentration as necessary.

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15.2 Refer to TriMatrix SOP GR-10-106 for corrective action to be taken when quality control is unacceptable.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). Each analyst responsible for sample processing by this procedure must pass an IDC.

16.1.1 Initial Demonstration of Capability (IDC) Study

16.1.1.1 Prepare four blank spikes (using a stock standard separate from the calibration stock) at 0.5 mg/kg. Extract, filter, distill and analyze the four spikes following every step in this and the analytical procedure.

16.1.1.2 Input the four results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation.

16.1.1.3 Average percent recovery must fall within blank spike acceptance limits.

16.1.1.4 Relative standard deviation must be ≤20%.

16.1.1.5 If either criterion is not met, locate and correct the source of the problem and repeat the study. Repeated failure however, will indicate a problem with the techniques and/or the procedure. If this occurs, locate the problem, correct the techniques and/or procedure then repeat the study successfully.

16.1.1.6 Samples may not be processed by the analyst until an IDC study has been successfully completed.

16.1.1.7 Give a copy of all attempted IDC spreadsheets and raw data to Quality Assurance for training and documentation.

16.1.2 Continuing Demonstration of Capability (CDC) Study

16.1.2.1 Annually a CDC is required by each analyst.

16.1.2.2 The CDC may be accomplished by any of the following approaches:

16.1.2.2.1 By repeating the IDC study

16.1.2.2.2 By using the last four results from the annual method detection limit study if run exclusively by the analyst. Use only the last four results.

16.1.2.2.3 By using four consecutive blank spikes from routine sample analysis if run exclusively by the analyst.

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16.1.2.2.4 By successfully and exclusively running a PT study performed during the course of routine sample analysis.

16.1.2.3 Process CDC data using the same spreadsheet used for the IDC study.

16.2 A Method Detection Limit (MDL) study must be performed annually for this procedure in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IIIB, Revision 3, November, 2004, Method 9010C, "Total and Amenable Cyanide Distillation"

19.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* other methods, Revision 1, November, 2004, Method 9013A, "Cyanide Extraction Procedure for Solids and Oils"

20.0 ATTACHMENTS

20.1 Example Diagram of Apparatus for Cyanide Distillation

20.2 Element™ Preparation Batch Report Example

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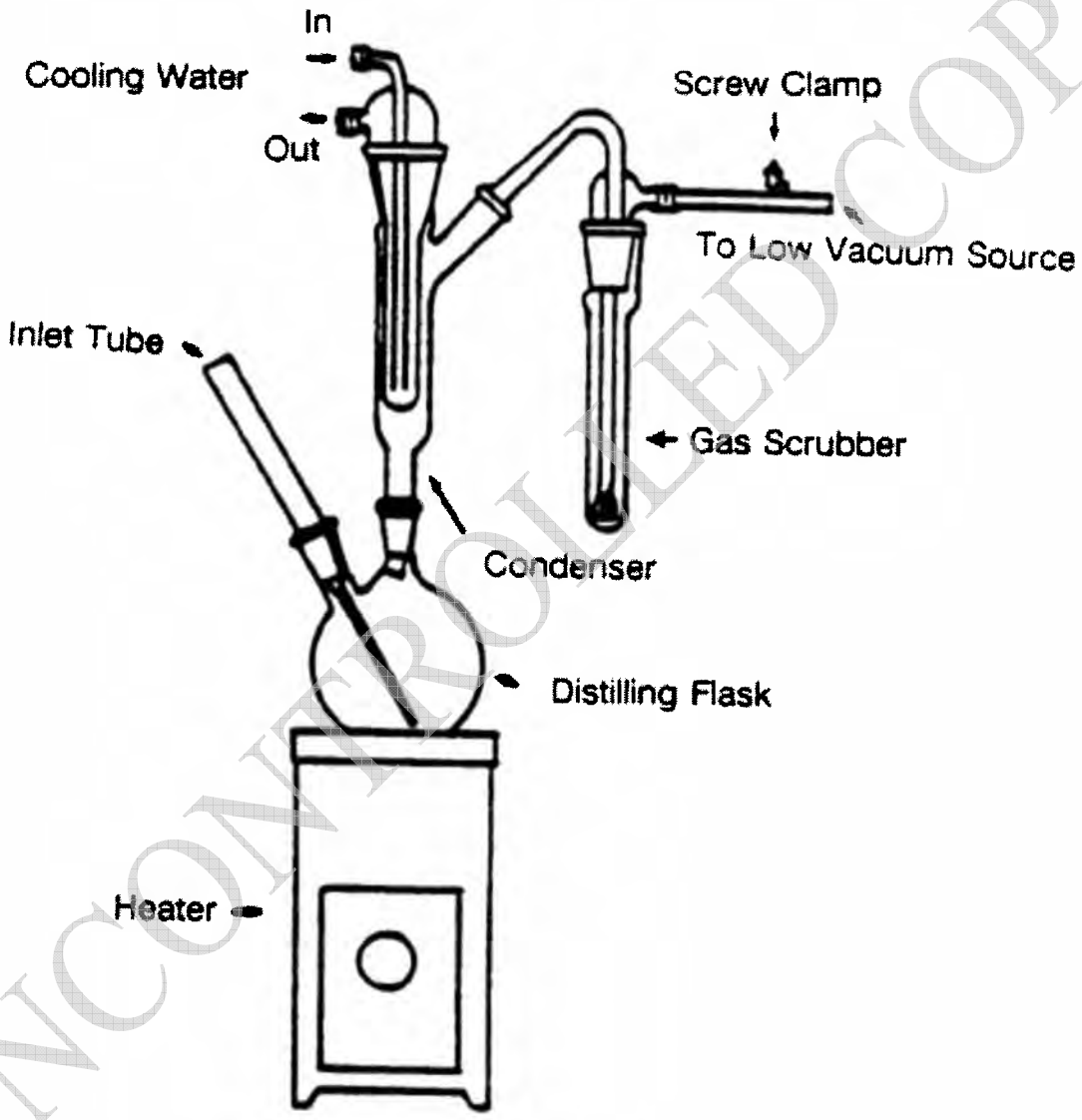
Attachment 20.1
Example Diagram of Apparatus for Cyanide Distillation



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Attachment 20.2
Element™ Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0903726 Page 1 of 1

Printed: 11/12/2009 12:04:00PM

Inorganic - Wet Chemistry, Soil, 9010B Cyanide Distillation

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0904051	Cyanide, Amenable 9014				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0903726-BLK1		Apr-06-09 07:00	LMA	25.33	250					BLANK	
0903726-DUP1		Apr-06-09 07:00	LMA	24.89	250		0904051-01			DUPLICATE	
0903726-BS1		Apr-06-09 07:00	LMA	25.87	250			9040185	250	LCS	
0903726-BS2		Apr-06-09 07:00	LMA	25.15	250			9040185	125	LCS	
0904051-01	A	Apr-06-09 07:00	LMA	24.75	250					[redacted]	

Comments:	Analyst Initials

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Approved By: 11-13-09 **QA Officer**

Approved By: Heather L. Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Midi-Distillation for Cyanide

Standard Methods 4500-CN⁻ B, Standard Methods 4500-CN⁻ C
SW-846 Method 9010C

APPROVALS:

Area Supervisor: Heather L. Brady Date: 11-13-09
Heather L. Brady

QA Officer: Date: 11-12-09
Tom C. Boocher

Operations Manager: Date: 11/13/09
Jeff P. Glaser

Procedure Number: GR-18-109
Revision Number: 3.4

Date Initiated: 7/23/93
Effective Date: 11/25/09

Date Revised: 11/12/09
Pages Revised: All

By: Lucy M. Asbury

Total Number of Pages: 13

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

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1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the midi-distillation of inorganic cyanides, present as simple soluble salts or complex radicals in water and wastewater.

1.2 The applicable concentration range is 0.005 mg/L to 0.2 mg/L.

1.3 The reporting limit is 0.005 mg/L.

2.0 PRINCIPAL METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, November 2004, Method 9010C, "Total and Amenable Cyanide Distillation"

2.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1995,* 4500-CN⁻ B, Cyanide, Preliminary Treatment of Samples

2.3 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1995,* 4500-CN⁻ C, Cyanide, Total Cyanide after Distillation

3.0 SUMMARY OF PROCEDURE

3.1 Cyanide present in a sample is released as a gas (HCN) upon addition of sulfuric acid (H_2SO_4). The HCN is collected in a sodium hydroxide (NaOH) scrubber. The NaOH solution from the scrubber is analyzed for cyanide in accordance with TriMatrix SOP GR-02-100 or GR-05-122.

3.2 Due to safety and waste considerations, the procedure uses bismuth nitrate as specified in method 9010C to eliminate sulfide interferences rather than powdered cadmium or lead carbonate as specified in methods 335.4 and 4500-CN⁻ C.

3.3 The procedure uses a midi-distillation block rather than the macro-distillation glassware specified in method 4500-CN⁻ C.

4.0 PARAMETER OR COMPOUND LIST

4.1 Total Cyanide

5.0 REFERENCED SOPs

5.1 TriMatrix Laboratories SOP GR-02-100, *Cyanide, Total, Lachat Autoanalyzer*, latest revision

5.2 TriMatrix Laboratories SOP GR-05-122, *Total Cyanide by Konelab UV Spectrophotometry*, latest revision

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5.3 TriMatrix Laboratories SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 Andrews Glass Company, *Midi-Distillation System*, Model 110-10, Instruction Manual

5.5 TriMatrix Laboratories SOP GR-10-106, *Inorganic and Metals Laboratories Corrective Actions*, latest revision

5.6 TriMatrix Laboratories SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Most interfering substances are removed by the distillation. However, sulfides, oxidizing agents, nitrates and nitrites are not removed. Detection of sulfides and oxidizing agents in a sample will require preliminary cleanup. All samples are treated to remove nitrate and nitrite interference.

6.2 Sulfides adversely affect the colorimetric determination and must be removed by addition of bismuth nitrate solution prior to distillation. Refer to Section 13.2.

6.3 Oxidizing agents such as residual chlorine cause a negative interference by destroying cyanide-containing compounds and must be removed by treating with ascorbic acid. Refer to Section 13.3.

6.4 High results are obtained for samples containing nitrates and/or nitrites. During the distillation, nitrates and nitrites form nitrous acid that reacts with some organic compounds to form oximes. These oximes decompose to generate HCN. Nitrate/nitrite interference must be removed by pre-treatment with sulfamic acid. Refer to Section 13.19.

7.0 SAFETY PRECAUTIONS

7.1 Comply with all instructions for health and safety as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.2 The heating block is hot during use. Precautions must be taken to avoid burns.

7.3 This procedure involves acidifying samples with potentially high cyanide levels. The distillation must be performed under an appropriate fume hood.

7.4 Avoid contact exposure with sulfuric acid and sodium hydroxide or any other chemical in the laboratory. Approved safety glasses, disposable gloves and a laboratory coat are required personal protective equipment (PPE) when working in the inorganic laboratory and while performing this procedure.

7.5 Potassium cyanide (KCN) is a violent poison. Precautions must be taken to avoid contact exposure, inhalation of dust or vapor and consequential ingestion. Always open containers and dilute all solutions in a fume hood wearing PPE. Keep potassium cyanide away from acids.

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8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Water and wastewater are collected in opaque brown, inert plastic bottles and preserved with sodium hydroxide to pH >12. Analysis must be performed within 14 days of sample collection.

8.2 All samples must be stored in the laboratory at 0 - 6° C until the time of analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Reflux distillation apparatus as shown in Attachment 20.1

9.2 Midi-distillation block

9.3 Volumetric flasks, glass, various sizes, type A

9.4 Volumetric pipets, glass, various sizes, type A

9.5 Balance, top-loading, readable to 0.01 g

9.6 Balance, analytical, readable to 0.0001 g

9.7 Graduated cylinders, various sizes

9.8 Boiling chips, clean and inert

9.9 Lead acetate paper, to check for sulfides

9.10 Specimen Cups, plastic, for scrubber/absorber solution storage

9.11 Micropipettes

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Clean the midi-distillation heating block after each use by wiping with a clean damp paper towel or cloth.

10.2 All glassware must be inspected for chips or cracks before use. Chipped or cracked glassware must be repaired or replaced.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory Reagent Water, ASTM Type II, Milli-Q system

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11.2 Sodium Hydroxide (NaOH), ACS grade or better

11.3 Bismuth Nitrate, 5-Hydrate ($Bi(NO_3)\cdot 5H_2O$), certified grade or better

11.4 Concentrated Sulfuric Acid (H_2SO_4), technical grade or better

11.5 Sulfuric Acid Solution (1:1 v/v) – Slowly and carefully add 500 mL of concentrated H_2SO_4 to 500 mL of laboratory reagent water. Cool to room temperature before use. Store in glass. Expiration is 6 months from the date made.

11.6 Potassium Cyanide (KCN), ACS grade or better

11.7 Magnesium Chloride, hexa-hydrate ($MgCl_2\cdot 6H_2O$), ACS grade or better

11.8 Sulfamic Acid (NH_2SO_3H), certified

11.9 L-Ascorbic Acid ($C_6H_8O_6$), ACS grade or better

11.10 Potassium iodide-starch test paper, to check for oxidizing agents

11.11 Sodium Hydroxide (0.25N)

11.11.1 Dissolve 10.0 g of sodium hydroxide pellets in a 1000 mL volumetric half-filled with reagent water. After all the material has dissolved, fill to the mark with reagent water.

11.11.2 Store tightly sealed in a labeled, inert polyethylene bottle at room temperature. Expiration is 6 months from the date made.

11.12 Bismuth Nitrate (0.026M)

11.12.1 Dissolve 30.0 g of $Bi(NO_3)\cdot 5H_2O$ in 100 mL laboratory reagent water, in a 1 L volumetric. While stirring, add 250 mL of glacial acetic acid. Stir until the material has dissolved. Dilute to 1 L with laboratory reagent water. Store tightly sealed in a labeled, inert polyethylene bottle at room temperature. Expiration is 6 months from the date made.

11.13 Magnesium Chloride (2.5M)

11.13.1 Dissolve 510 g of $MgCl_2\cdot 6H_2O$ in a 1 L volumetric half-filled with reagent water. After all the material has dissolved, fill to the mark with reagent water. Store tightly sealed in a labeled, inert polyethylene bottle at room temperature. Expiration is 6 months from the date made.

11.14 Sulfamic Acid Solution (0.4N)

11.14.1 Dissolve 40 g of NH_2SO_3H in a 1 L volumetric half-filled with reagent water. After all the material has dissolved, fill to the mark with reagent water. Store tightly sealed in a labeled, inert polyethylene bottle at room temperature. Expiration is 6 months from the date made.

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11.15 Glacial Acetic Acid (CH_3COOH), EM Omnitrace™ or equivalent

11.16 Anhydrous Sodium Acetate (CH_3COONa), ACS grade or better

11.17 Acetate Buffer Solution (pH = 4.0)

11.17.1 Dissolve 7.3 g of CH_3COONa in a 50 mL volumetric filled with about 20 mL of reagent water. Add 24 g of CH_3COOH (glacial) and swirl to mix. After all the material has dissolved, fill to the mark with reagent water. Store tightly sealed in a labeled, inert polyethylene bottle at room temperature. Expiration is 6 months from the date made.

Note: Check and record the solution pH after making.

11.18 Antifoam, silicone emulsifying agent

12.0 STANDARDS PREPARATION

12.1 Any samples treated with bismuth nitrate for sulfide interference (Section 13.18) must be quantitated using a calibration curve treated with bismuth nitrate.

12.2 Calibration standards preparation is covered in TriMatrix SOP GR-05-122, Section 12.0.

13.0 ANALYTICAL PROCEDURE

13.1 Water and wastewater samples must be homogenized by shaking just before removing a sample aliquot for analysis. After shaking well, measure 50 mL of sample with a graduated cylinder. Transfer the aliquot to a distillation tube containing 2 or 3 boiling chips.

13.2 Check each aliquot for sulfides with lead acetate paper. A black color on the paper indicates sulfides are present and the sample must be treated using bismuth nitrate (Section 13.18). If treated, a bismuth nitrate calibration must be used for quantitation.

13.3 Check each aliquot for oxidizing agents with potassium iodide starch paper (dampened with acetate buffer). A blue color on the paper indicates oxidizing agents are present. Add approximately 0.2 g of L-ascorbic acid. Stir and test again. Repeat until the paper no longer turns blue then add approximately 0.05 g more L-ascorbic acid powder.

13.4 Position the distillation tube containing the sample aliquot into the left rear position of the midi-distillation block.

13.5 Add 50 mL of 0.25N NaOH to a scrubber/absorber tube and place in the front left position of the block. If a sample appears foamy, add 1 to 2 drops of antifoam before starting the distillation.

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Note: If antifoam is used, the distillation blank (BLK) and blank spike (BS) must also use antifoam.

13.6 Place a reflux impinger with the gas exit tube facing the rear of the block into the distillation tube.

13.7 Place an absorber impinger with the gas inlet tube facing the rear of the block into the scrubber/absorber tube.

13.8 Connect the absorber impinger gas inlet tubing to the reflux impinger gas outlet.

13.9 Connect the absorber impinger vacuum tubing to the vacuum outlet.

13.10 Place a Cold-Finger Condenser, with water ports facing the rear of the block, into the reflux impinger.

13.11 Connect Cold-Finger Condenser water tubing to the water manifold quick-disconnect adapters on the rear of the block. The lower tubing connects to the left front quick-disconnect adapter (water outlet) and the upper tubing connects to the left rear quick-disconnect adapter (water inlet).

13.12 Repeat Sections 13.1 through 13.11 for each sample, moving from left to right across the block. Up to 10 samples may be loaded at one time.

13.13 Turn off all vacuum valves.

13.14 Check to make sure an excess gas trap is loaded and connected to the vacuum line attached to the block.

13.15 Turn on the cooling water and adjust to 6 gal/hr for each distilling position. For example, if 4 samples are being distilled, adjust the flow to 24 gal/hr (6 x 4 = 24 gal/hr).

Note: NEVER operate the cooling water unless cold-finger condensers are connected to the water manifold. Improper use may damage cold-finger condensers and/or rupture the tubing.

13.16 Turn on the vacuum and adjust each valve to give a flow of three bubbles per second in each reflux tube.

13.17 Let the vacuum draw for 5 minutes.

13.18 If a sample tested positive for sulfide (Section 13.2), add 10 mL bismuth nitrate solution at this time.

13.19 Add 5 mL of sulfamic acid through the air inlet of each reflux impinger.

13.20 Inject 5 mL of 1:1 (v/v) sulfuric acid through the air inlet of each reflux impinger.

13.21 Let the vacuum draw for 5 minutes more.

13.22 Inject 2 mL of magnesium chloride solution through the air inlet of each reflux impinger.

13.23 Turn the distillation block on. The red rocker switch light will glow while the unit is on.

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13.24 Turn on the timer. The green timer light and amber heat lights will glow. The timer is set for 15 minutes of heat-up and 90 minutes of distilling. The distilling block temperature is preset to 125° C.

13.25 The timer will automatically turn off the heating block when the time has expired. Vacuum draw and cooling water are unaffected by turning the heat off.

13.26 Let the distilling block cool for 15 minutes before turning off the cooling water and vacuum.

13.27 Close each valve and disconnect the absorber gas inlet tubing from the reflux impinger.

13.28 Starting from the right, open the valve to draw all solution from the absorber impinger frit. Close the valve and remove the impinger frit.

13.29 Disconnect the impinger vacuum tubing and remove the absorber impinger from the unit.

13.30 Pour the scrubber solution from the absorber tube into a plastic specimen cup. Label and tightly cap the specimen cup until analysis, which must be within the sample holding time.

13.31 Working from right to left, repeat Sections 13.28 through 13.30 for each distilling position.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Record the following data on the preparation batch report for each sample batch distilled:

14.1.1 Sample volume distilled, in mL

14.1.2 Final volume of diluted scrubber/absorber solution

14.1.3 Volume and concentration of all stock standards used as spikes

14.1.4 Analyst initials

14.1.5 Date

14.2 Refer to Attachment 20.2 for a preparation batch report/analysis sequence example.

15.0 QUALITY ASSURANCE

15.1 Method and matrix QC must be distilled with each sample analysis batch. A batch consists of up to 20 samples per matrix and may be left open for up to 14 days.

15.1.1 A blank spike (BS) is an aliquot of reagent water spiked from a stock standard, which is distilled and analyzed like a sample. Distill a low and high range BS with each batch.

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15.1.1.1 To prepare a high-concentration blank spike, measure and transfer 50 mL of reagent water into a distillation tube. Pipet 50 µL of the 100 mg/L stock standard (Refer to Section 12.7 of TriMatrix SOP GR-05-122) then proceed with the distillation and analysis. The high-range BS concentration is 0.1 mg/L cyanide.

15.1.1.2 To prepare a low-concentration BS, measure and transfer 50 mL of reagent water into a distillation tube. Pipet 20 µL of the 100 mg/L stock standard (Refer to section 12.7 of TriMatrix SOP GR-05-122) then proceed with the distillation and analysis. The low-range BS concentration is 0.04 mg/L cyanide.

15.1.2 A method blank (BLK) is 50 mL of reagent water distilled and analyzed like a sample. Measure and transfer 50 mL of reagent water to a distillation tube then proceed with the distillation and analysis.

15.1.3 At a minimum, a low-range blanks spike, a high-range blank spike and method blank are distilled at the start of every batch. Until a batch is closed (at 20 samples or in 14 days), a low-range BS and BLK <u>or</u> a high-range BS and BLK must also be distilled with each distillation set processed into the open batch. Consequently, BS/BLK data within each batch will vary with the number of times midi-distillation is performed before the batch is closed.

Note: Place QC samples in the Midi-block randomly. Do not use the same position to run QC for each batch.

15.1.4 Acceptance limits for quality control are listed in the laboratory information management system (Element™).

15.2 Matrix QC consists of replicate matrix spikes, labeled MS and MSD. Matrix spikes are performed at a frequency of one set per batch of up to 20 samples per matrix.

15.2.1 To prepare a matrix spike/matrix spike duplicate, measure and transfer two 50 mL sample aliquots into two separate distillation tubes. Pipet 50 uL of the 100 mg/L stock standard (Refer to Section 12.7 of TriMatrix SOP GR-05-122) then proceed with the distillation and analysis. The matrix spike concentration is 0.1 mg/L.

15.2.2 If sample concentration is high, sample dilution and adjustment to the spike quantity may be necessary.

15.2.3 Acceptance limits for matrix quality control are listed in the laboratory information management system (Element™).

15.3 All corrective action in association with out-of-control quality control must be in accordance with TriMatrix SOP GR-10-106.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

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16.1 Before actual sample analysis, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample distillation or analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare four aliquots of laboratory reagent water spiked with second-source stock standard. Distill and analyze the four prepared spikes following every step outlined in both distillation and analysis procedures. Use the IDC spreadsheet on the intranet library to calculate average percent recovery from the results obtained and relative standard deviation of the average. Recovery must be within 90 - 110% for drinking water samples and within blank spike acceptance limits for all other matrices. Relative standard deviation must be ≤20%.

16.1.1.2 If either criterion fails, locate and correct the source of the problem and repeat the study. Repeated failure however, will confirm a general problem with the procedure or techniques used. If this occurs, locate and correct the source of the problem, revise the procedure or techniques used and repeat the study. Samples may not be analyzed by any analyst or on any instrument until a demonstration of capability study has been successfully completed.

16.1.1.3 Copies of all (successful and unsuccessful) IDC/Method Validation spreadsheets and raw data must be submitted to Quality Assurance for training documentation.

16.1.2 Continuing Demonstration of Capability (CDC)

16.1.2.1 A CDC must be performed annually by all analysts distilling samples for cyanides analysis.

16.1.2.2 The CDC may be completed by one of the following approaches:

16.1.2.2.1 By repeating the IDC study

16.1.2.2.2 By distilling an acceptable PE sample during the course of routine sample analysis

16.1.2.2.3 By using the last four results from an MDL study distilled during the course of routine sample analysis.

Note: In each case, the work must have been done exclusively by one analyst.

16.2 A Method Detection Limit (MDL) Study must be completed annually in accordance with TriMatrix SOP GR-10-125.

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17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals. Material safety data sheets are available on the laboratory intranet library.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, November 2004, Method 9010C, "Total and Amenable Cyanide Distillation"

19.2 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1995,* 4500-CN⁻ B, Cyanide, Preliminary Treatment of Samples

19.3 *Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1995,* 4500-CN⁻ C, Cyanide, Total Cyanide after Distillation

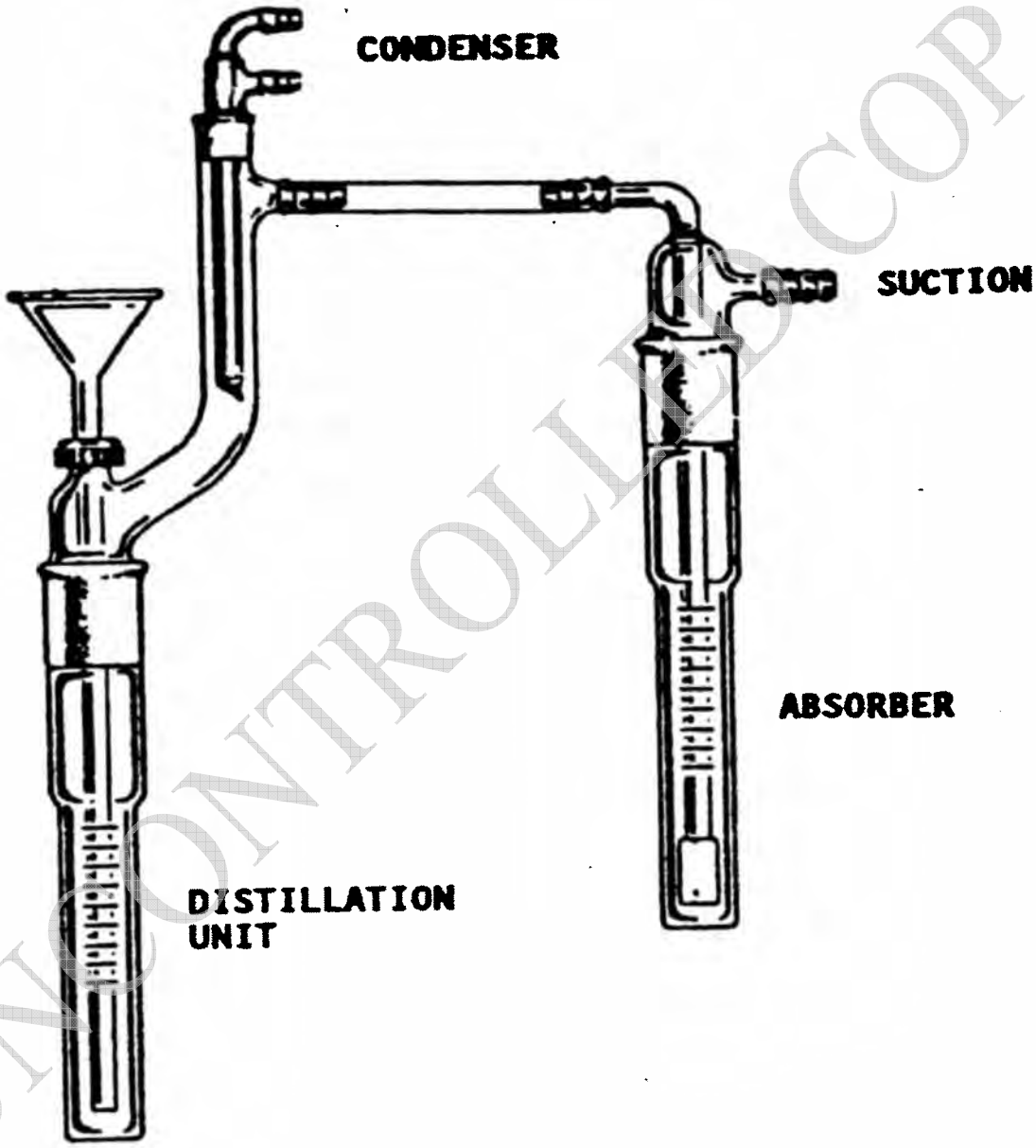
20.0 ATTACHMENTS

20.1 Reflux Distillation Apparatus

20.2 Preparation Batch Report Example

Approved By: 11-13-09 **QA Officer** **Approved By:** Heather L. Brady **Area Supervisor**

Attachment 20.1
Reflux Distillation Apparatus



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Attachment 20.2
Pre-treatment Benchsheet Example

TriMatrix Laboratories, Inc. **PREPARATION BATCH** 0911292 Page 1 of 1 Printed: 9/28/2009 2:55:22PM

Inorganic - Wet Chemistry, Waste Water, 4500-CN C Cyanide Distillation

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0909471	Cyanide, Total 335.4				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0911292-BLK1		Sep-25-09 06:00	LMA	50	50					BLANK	
0911292-DUP1		Sep-25-09 06:00	LMA	50	50		0909471-04			DUPLICATE	
0911292-BS1		Sep-25-09 06:00	LMA	50	50			9090699	50	LCS	
0911292-BS2		Sep-25-09 06:00	LMA	50	50			9090699	20	LCS	
0911292-MS1		Sep-25-09 06:00	LMA	50	50		0909471-04	9090699	50	MATRIX SPIKE	
0909471-01	B	Sep-25-09 06:00	LMA	50	50					[redacted]	
0909471-02	B	Sep-25-09 06:00	LMA	50	50					[redacted]	
0909471-03	B	Sep-25-09 06:00	LMA	50	50					[redacted]	
0909471-04	B	Sep-25-09 06:00	LMA	50	50					[redacted]	
0909471-05	B	Sep-25-09 06:00	LMA	50	50					[redacted]	

Comments:	Analyst Initials

bch_TriMatrix.rpt

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Approved By: 11-13-09 **QA Officer** **Approved By:** Heather L Brady **Area Supervisor**

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

REACTIVE SULFIDE

SOP has undergone an annual review. No revisions were required.
Date Reviewed: 9-12-06
Reviewed By: JLB
Review Expires: 9-12-07

SW-846 Chapter 7
Paragraph 7.3.4

APPROVALS:

Inorganic Manager: Jodi L. Blouw — Date: 11/7/01
Jodi L. Blouw

QA Manager: Rick D. Wilburn — Date: 11/7/01
Rick D. Wilburn

Laboratory Manager: Douglas E. Kriscunas — Date: 11/7/2001
Douglas E. Kriscunas

Procedure Number: GR-18-117
Revision Number: 3.0

Date Initiated: 3/29/94
Effective Date: 11/8/01

Date Revised: 11/07/01
Pages Revised: All

By: Heather Brady

Total Number of Pages: 11

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures.

1.2 This procedure releases only the evolved hydrogen sulfide at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 3, December 1996, Chapter 7, Section 7.7, "Interim Guidance for Reactive Sulfide".

3.0 SUMMARY OF PROCEDURE

3.1 An aliquot of sample is acidified to a pH of 2 in a closed system. The gas generated is swept into a scrubber. The analyte concentration is quantitated by titration.

3.2 Separation of reactive sulfide from the sample matrix is accomplished by acidifying the sample in a closed system. The generated gas is carried by a nitrogen stream into a sodium hydroxide filled scrubbing bottle. The solution in the scrubber is acidified to a pH of 2 and quantitated titrimetrically by Method 9034.

4.0 PARAMETER OR COMPOUND LIST

4.1 Reactive sulfide

5.0 REFERENCED SOPs

5.1 TriMatrix SOP number GR-06-108, *Titrimetric Procedure for Acid-Soluble and Reactive Sulfides*, most current version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences are undetermined

7.0 SAFETY PRECAUTIONS

7.1 The analyst must comply with all Standard Operating Procedures for Health and Safety as outlined in the TriMatrix Laboratories, Inc. Safety Manual.

7.2 As this test generates toxic hydrogen sulfide, this pre-treatment procedure must be conducted in a fume hood.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples should be collected in inert glass or plastic containers with a minimum of aeration. The container should be filled completely, excluding headspace.

8.2 Samples should be refrigerated at 4° C until the time of analysis. Analysis should commence as soon as possible, but both the pre-treatment and analysis must be completed within seven days of collection.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Round-bottom flask: 500-mL, three-neck, with 24/40 ground-glass joints

9.2 Magnetic stir plate and stir bars

9.3 Addition funnel, 250 mL, with 24/40 ground glass joints and pressure equalizing tube

9.4 Inlet adapter, 24/40 ground glass joint with 100 mm downtube and tubing connector (for introducing nitrogen below liquid surface)

9.5 Inlet adapter, 24/40 joint with tubing connector (for nitrogen exit)

9.6 Tygon tubing, R3603, 1/4" ID, 1/16" wall

9.7 Gas flow controller with flow meter

9.8 Scrubber assembly, 100 mL capacity, with fritted impinger

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Not applicable

11.0 CHEMICALS AND REAGENTS

11.1 Deionized water

11.2 Sodium sulfide ($Na_2S \bullet 9H_2O$), J.T. Baker reagent grade crystals or equivalent

11.3 Compressed pre-purified nitrogen

11.4 Sulfuric acid (0.01N), H_2SO_4

11.4.1 In a 1 liter volumetric flask partially filled with reagent water, add 28 mL concentrated H_2SO_4 and dilute to 1 L. This makes a 1.0N solution.

11.4.2 To a 1 liter volumetric flask partially filled with reagent water, add 10 mL of 1.0N solution and dilute to 1 L. This makes the 0.01N H_2SO_4 solution. This solution may be stored in an inert plastic bottle at room temperature for up to 6 months.

11.5 Sodium hydroxide, 0.25N

11.5.1 Add 10.0 g of sodium hydroxide pellets to a 1 L volumetric flask. Dilute to volume with DI H_2O. May be stored in an inert plastic bottle at room temperature for up to 6 months.

11.6 All reagents must be logged into the Reagent Logbook (Attachment 20.1). Unless otherwise indicated, all reagents have an expiration date of 1 year from the date of preparation.

12.0 STANDARDS PREPARATION

12.1 Stock sulfide standard

12.1.1 This standard is used for preparing the Laboratory Fortified Blank (LFB) and Matrix Spike (SPK). It must be prepared at a pH of between 9 and 11. Protect the standard from exposure to oxygen by preparing it without headspace.

12.1.2 Dissolve approximately 3.750 g $Na_2S \bullet 9H_2O$ in a 500 mL volumetric flask half full of reagent water. Adjust pH using NaOH. Dilute to volume with reagent water. 1 mL of this solution equals approximately 1.00 mg sulfide. This solution may be stored in an inert plastic bottle at room temperature for up to 6 months.

NOTE: This solution is unstable due to oxidative degradation and the evaporation of hydrogen sulfide. It also cannot be determined stoichiometrically because of the uncertain water content of the sodium sulfide crystals used to make it. Therefore, the concentration must be determined by iodine titration every day the standard is used, just prior to its use. Calculate the exact concentration using 2-4 mL of the standard, and follow the procedure in section 15 of the analytical SOP, TriMatrix SOP number GR-06-108.

12.2 The standard must be logged into the Standard Logbook (Attachment 20.2).

13.0 ANALYTICAL PROCEDURE

13.1 Add 50 mL of the 0.25N NaOH solution to the scrubber tube and dilute to adequate depth with reagent water.

13.2 Add 10 g of the waste sample to the three-neck round bottom flask.

13.3 Assemble the system as shown in Attachment 20.3.

13.4 Add 250 mL of 0.01 N sulfuric acid to the addition funnel.

13.5 Start the nitrogen flow into the flask. Adjust the flow rate to 60 mL/min.

13.6 With the nitrogen flowing, open the stopcock on the sulfuric acid addition funnel and begin adding the acid to the flask. Begin timing for 30 minutes at this point. Start stirring the solution in the flask, and continue adding acid until the flask is half full. The stirring rate must be slow enough to NOT create a vortex. The stirring speed must remain constant throughout the 30 minutes.

13.7 After 30 min shut off the nitrogen flow, stop stirring, and disconnect the scrubber. Determine the amount of reactive sulfide in the scrubber solution following method 9034, TriMatrix SOP number GR-06-108. Make sure the procedure for reactive sulfide is followed.

14.0 DATA REPORTING AND DELIVERABLES

14.1 The analyst processing a set of samples is responsible for the quality of the data and for correctly filling out the proper documentation. This is required for quality control purposes, and in order to provide the client with defensible data.

14.2 All benchsheets and logbooks must be filled in completely and correctly. All corrections are to be made in indelible ink. **Corrections are to be made with a single lineout, which is then dated and initialed. Write-overs are not acceptable.** The erroneous result must remain legible. The new result is placed near the incorrect result. Blank lines in the logbook should be Z'd out.

14.3 Attach the pre-treatment benchsheet (Attachment 20.4) to the analytical benchsheet.

14.4 The following data is to be put on the benchsheet for each batch turned in:

- analyst name
- date of analysis
- amounts of sample used
- reagent numbers
- standard numbers
- batch number

14.5 If internal chain of custody is required it is very important that the COC sheet is filled out correctly.

15.0 QUALITY ASSURANCE

15.1 Method and matrix QC must be prepared with each batch.

15.1.1 Method QC associated with this pre-treatment method consists of a Laboratory Fortified Blank (LFB), and a Method Preparation Blank (MPB).

15.1.1.1 A LFB and MPB should be prepared each day samples are prepped. The acceptance limits for the LFB are listed on the benchsheet used for the method 9034 analyses. The MPB result must be less than the reporting limit for the analysis.

15.1.1.2 The LFB is prepared by spiking 2-4 mL of the standard prepared in section 12.1 into 100 mL of reagent water, and pre-treating as described in this SOP.

15.1.1.3 The MPB is reagent water carried through the pre-treatment procedure.

15.1.2 Matrix QC associated with this pre-treatment method consists of a Matrix Spike (SPK) and sample duplicate (DUP).

15.1.2.1 A SPK and DUP must be pre-treated at a minimum of one set with every twenty samples. The acceptance limits for the SPK and DUP are listed on the benchsheet used for the method 9034 analyses.

15.1.2.2 The SPK is a sample aliquot spiked with 2-4 mL of the standard prepared in section 12.1, then pre-treated following this SOP.

15.1.2.3 The DUP is a second aliquot of sample carried through the pre-treatment process.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare four LFBs as described in section 15.1.1.2. Process the four samples following the procedures outlined in the SOP, and analyze following method 9034, SOP number GR-06-108. Calculate the average percent recovery and the standard deviation of the 4 recoveries. The average percent recovery must be within the LFB acceptability windows listed on the benchsheet. The standard deviation of the 4 recoveries must be ≤20%.

16.1.1.2 If either criteria are not met, locate and correct the source of the problem and repeat the test. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful analyst certifications/method validations spreadsheet and raw data should be given to the Quality Assurance Manager.

16.1.2 Continuing Demonstration of Capability

16.1.2.1 Annually a Continuing Demonstration of Capability (CDC) is required by each analyst. The CDC can be accomplished by repeating the IDC or processing four consecutive LFBs as an IDC. Process these samples using the same spreadsheet

used for the IDC study. Alternatively, the successful completion of a Performance Testing sample from that year may be used for a CDC.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve use of chemicals where applicable

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

18.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 3, December 1996, Chapter 7, Section 7.7, "Interim Guidance for Reactive Sulfide".

19.2 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 2, December 1996, Method 9030B, "Acid-Soluble and Acid-Insoluble Sulfides: Distillation".

19.3 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Revision 0, December 1996, Method 9034, "Titrimetric Procedure for Acid-Soluble and Acid Insoluble Sulfides".

20.0 ATTACHMENTS

20.1 Reagent Logbook Example

20.2 Standard Logbook Example

20.3 Diagram of Apparatus

20.4 Pre-Treatment Benchsheet Example

Attachment 20.1
Reagent Logbook Example

TriMatrix Laboratories, Inc.

Reagent Preparation Logbook

Reagent Name: Iodine Titrant (Sulfide) **Stock Reagent Number:** *INR1.61*

Prep Instructions: 20.25g KI + 3.2g Iodine / 1L DI H2O

Concentrations:

Working Reagent Number	Date Made	Expiration Date	Made By	Reagent(s)/Lot Number(s) Used	Solvent(s)/Lot Number(s) Used
INR1.61-1	4/5/00	4/5/01	CEM	KI (DEC00) 126578 I 292A (08906MF)	
INR1.61-2	4/20/00	4/20/01	CEM	↓ ↓	
INR1.61-3	5/12/00	5/12/01	CEM	126578/126578 ↓	
INR1.61-4	5/30/00	5/30/01	CEM	↓ ↓	
INR1.61-5	7/7/00	7/7/01	CEM	↓ ↓	
INR1.61-6	7/24/01	7/24/02	CEM	↓ ↓	
INR1.61-7	8/9/01	8/9/02	HLB	↓ ↓	
INR1.61-8	10/10/01	10/10/02	HLB	↓ ↓	
INR1.61-9	10/10/01	10/10/02	HLB	↓ ↓	
INR1.61-10					
INR1.61-11					
INR1.61-12					
INR1.61-13					
INR1.61-14					
INR1.61-15					
INR1.61-16					
INR1.61-17					
INR1.61-18					

file: in1reagent page: 61 of 100 revision: 0.0

Approved By: 11/7/01
QA Manager

Approved By: 11/7/01
Area Manager

Attachment 20.2
Standard Logbook Example

TriMatrix Laboratories, Inc.

Row #	Standard Number	Standard Description	Analyte(s) (and/or Stock Standard Number for dilutions)	Manufacturer and Lot Numbers	Exp. Date	Ampule or Stock Standard Concentration	Initial Weight/ Volume	Solvent Used	Final Volume	Final Concentration	Made or Opened By	Date Made or Opened	Date Expires	Math Check By	Checked Against Standard #
1	IN1.21.1	orthophos	IN1.8.8			1000ppm	10ml	H_2O ~~100ml~~ SAJ 11/3/00	100ml	100ppm	SAJ	11/3/00	11/4/00		
2	IN1.21.2	↓	IN1.8.9			↓	↓	↓	↓	↓	↓	↓	↓		
3	IN1.21.3	Chloride	264J.37			1000ppm	40ml	H_2O	100ml	400ppm	SAJ	11/3/00	12/3/00		
4	IN1.21.4	↓	↓			1000ppm	15ml	↓	100ml	150ppm	↓	↓	↓		
5	IN1.21.5		SAJ			1000ppm	25ml		250ml	100ppm					
6	IN1.21.6		↓			1000ppm	6ml		100ml	60ppm					
7	IN1.21.7	SAJ	264J.37			1000ppm	2.0ml		100ml	20ppm		SAJ	SAJ		
8	IN1.21.8		IN1.21.5			100ppm	10ml	SAJ	100ml	10ppm	SAJ				
9	IN1.21.9		↓			100ppm	5ml		100ml	5.0ppm					
10	IN1.21.10		SAJ			100ppm	2ml	↓	100ml	2.0ppm	↓	↓	↓		
11	IN1.21.11	Chloride	IN1.21.5			100ppm	2ml	H_2O	200ml	1.0ppm	SAJ	11/3/00	12/3/00		
12	IN1.21.12	TOC	IN1.18.12			10ppm	1ml	H_2O	100ml	1.0ppm	JMB	11/6/00	11/6/00		
13	~~IN1.21.13~~ 11/6/00	Sulfite				1.5768g Sodium Sulfite	1000ml	H_2O	1,000ml	1,000ppm	JMB	10/30/00	10/30/00		
14	IN1.21.14	Sulfate	225B54		3/1/01	~~10ml~~ 1000mg/L	20ml	H_2O	100ml	200mg/L	JLB	11/6/00	12/6/00 ~~11/20/00~~ 11/6/00		
15	IN1.21.15	↓	↓			↓	15ml	↓	100ml	150		↓	↓		
16	IN1.21.16						20ml		200ml	100					
17	IN1.21.17	↓	↓			↓	16ml	↓	200ml	80		↓			
18	IN1.21.18	Sulfate	225B54			1000mg/L	5ml	H_2O	100ml	50		11/6/00	12/6/00 ~~11/20/00~~ 11/6/00		

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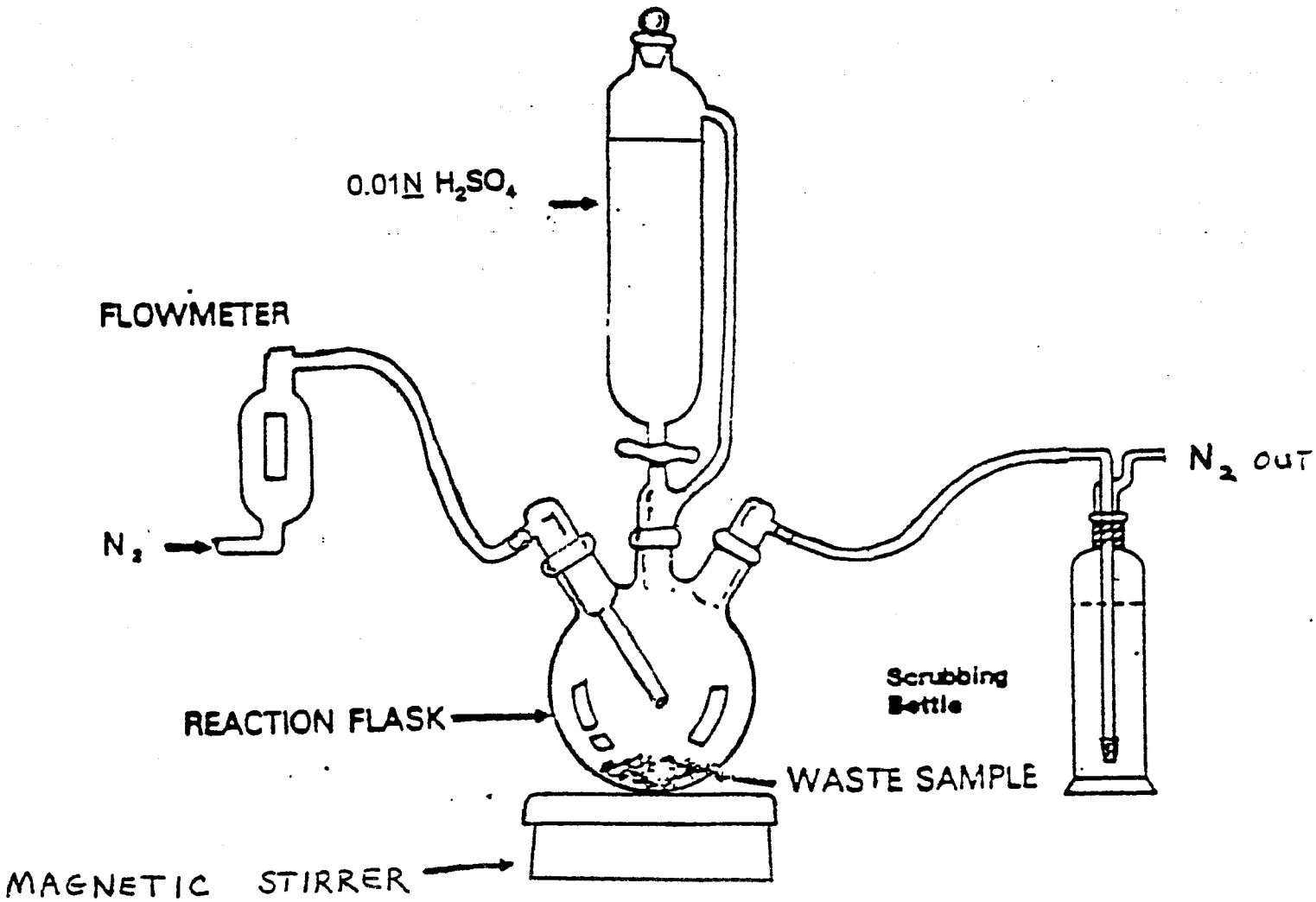
page: 21 of 50

revision: 1.0

Approved By: RF 11/7/01
QA Manager

Approved By: GB 11/7/01
Area Manager

Attachment 20.3
Diagram of Apparatus



Approved By: _____ QA Manager

Approved By: _____ Area Manager

Attachment 20.4
Pre-treatment Benchsheet Example

24-OCT-01 WET CHEM PRETREATMENT BENCHSHEET PAGE 1

Test #: 394.01- 374.01
Parameter: INORGANIC PRETREATMENT
Method: H2S/REACT/WST
Ref. cit.: USEPA-7.3.4.2

Thermometer #: ____ | ____
Initial Deg C: ____ | ____
Final Deg C: ____ | ____

Est anal hrs: 2
Act anal hrs: _____
Stock std #: _____

Owner: HLB
Date run: 10-25-01
Supervisor: JLB
QC Batch #: TM1838-125

Comments: _____

Client / Submittal Location	Sample	COC	QC	Anl.	Date Treated	Initial wt/vol	Final wt/vol	Conc. Spiking Solution	SPK STK #	Volume Spiked	Final Spike Conc.	Qu
Kent County DPW SL-01	33280- 20 290628		LV2			10g	10g					
Environmental Services S. Wall of	32148- 22 291100 * !		LV3			10g	10g					
MPB:						10	10					
LFB: Stk IN4.55.16						10	10	700ppm		5mls	380 mg/kg	
SPK1: Stk IN4.55.16 Smp 291100						10g	10g	700ppm		5mls	380mg/kg	
SPK2: Stk _____ Smp _____												
DUP: _____ Smp 291100						10g	10g					

TRIMATRIX LABORATORIES, INC

Approved By: 11/7/01
QA Manager

Approved By: GB 11/7/01
Area Manager

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Setaflash Closed-Cup Flash point

SW-846 Method 1020A

APPROVALS:

Area Supervisor: Heather L. Brady — Date: 12-17-08

QA Officer: Tom C. Boocher — Date: 12-16-08

Operations Manager: Jeff P. Glaser — Date: 12/16/08

Procedure Number: GR-18-124
Revision Number: 1.2

Date Initiated: 10/1/99
Effective Date: 12/31/08

Date Revised: 12/16/08
Pages Revised: All

By: Shelly A. Jewell

Total Number of Pages: 10

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable for the determination of the flash point of fuel oils, lube oils, suspension of solids, and liquids. The procedure, while not technically applicable, is also used to determine the flash point of solid and waste samples. Flash point data reported from the analysis of solid and waste samples will be qualified. If a waste sample is difficult to homogenize, and a larger amount of sample needs to be used to show a more representative sample, Method 1010 should be used.

1.2 The applicable range is 68° F - 200° F.

1.3 The reporting limit for this analysis is 68° F.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 1, July, 1992, Method 1020A, "Setaflash Closed-Cup Method for Determining Ignitability".

3.0 SUMMARY OF PROCEDURE

3.1 The sample is heated at a low, constant rate while a flame is directed into the cup at regular intervals. The flash point is the lowest temperature at which application of the flame ignites vapors above the sample.

4.0 PARAMETER OR COMPOUND LIST

4.1 Flash point

5.0 REFERENCED SOPs

5.1 See TriMatrix SOP number GR-18-108, *Flash point – Pensky/Marten Closed-Cup,* latest version.

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Ambient pressure must be that of the laboratory at the time of testing and not that of weather stations and airports which are pre-corrected to give sea level readings.

6.2 Sample non-homogeneity may give erroneous readings. Care must be taken to not open containers unnecessarily or make sample transfers at temperatures within 30° F of the flash point. Excessive mixing can also lead to loss of volatile materials.

6.3 Drafts must be kept to a minimum by use of a draft-free room or compartment.

6.4 Operator bias can affect flash point. Care must be taken to assure that all testing conditions are nominal and consistent in the flame exposure device. These items include the size of the test flame, the rate of temperature increase, and the rate of dipping the flame into the sample vapor.

7.0 SAFETY PRECAUTIONS

7.1 Analysts must comply with all standard operating procedures for health and safety as outlined in the TriMatrix Laboratories, Inc. Laboratory Safety Manual.

7.2 All flash point testing must be performed in a fume hood, and away from ignitable solvents.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples shall not be stored in plastic bottles since volatile materials may diffuse through the walls of the bottle.

8.2 Samples are to be stored at 4° C until time of analysis.

8.3 Waters and wastewaters must be homogenized by shaking just prior to removing a sample aliquot. Wastes, sludges, and soils must be homogenized using an appropriate mixing procedure prior to analysis.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Koehler Rapid Tester Model K16502, Closed-cup

9.2 ASTM Thermometer No. 9F Range 20 – 230° F, 287 mm

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Routine preventive maintenance items include thoroughly cleaning and drying all parts of the cup. Additional cleaning, when necessary can be accomplished with solvents such as methylene chloride which are capable of removing oily and stubborn material.

10.2 A visual inspection of the flash point apparatus components should be performed each day prior to use, to insure proper connections and mechanical functionality.

11.0 CHEMICALS AND REAGENTS

11.1 p-Xylene is used as the reference standard. Store p-xylene under a hood.

11.2 The p-xylene reference standard may be stored at room temperature for up to 2 years.

12.0 STANDARDS PREPARATION

12.1 Not applicable

13.0 SAMPLE PREPARATION

13.1 Samples of viscous materials may be warmed until they are reasonably fluid prior to testing. No sample shall be heated any higher than necessary, and no sample shall be heated to a temperature that exceeds 30° F below the sample's expected flash point.

14.0 CALIBRATION PROCEDURES

14.1 Use the thermometer reading, not the LED readout for the temperature of the testing unit.

14.2 Determine the p-xylene flash point by adding 2.0 mL (using provided syringe) through the leakproof entry port. When the unit is properly operating, the flash point of p-xylene will be at 81° F ± 2° F. If the value lies outside of this window, re-verify the operating conditions, flash point unit, and the sample itself and repeat test. If the value still lies outside the excepted window, contact the area supervisor.

15.0 ANALYTICAL PROCEDURE

15.1 Thoroughly clean and dry all parts of the cup and the cover assembly before starting the test. Be sure to remove any solvent that was used to clean the apparatus.

15.2 Add 2 mL of liquid sample through injection port of tester. For soils or wastes use approximately 2 g of sample.

15.3 To adjust end temperature in which you want the tester to ramp to, depress the red Temperature Preset switch while turning the black knob. This can be helpful for checking the calibration, or if the flash point temperature is suspected.

15.4 To check for a flash, turn on the power switch and allow temperature to rise 9° F. Apply test flame for one second. If no flash occurs repeat test flame every 9° F. If flash occurs, fresh sample should be added. A repeat determination is then made, starting the test at the temperature of the last interval before the original flash was observed.

15.5 When there is enough sample available, each sample must be run in duplicate. The result of the duplicate analysis must be within 3° F of the initial analysis. If this criteria is not met, the samples must be re-analyzed (if possible), or the result qualified as estimated.

15.6 Record as the flash point, the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. DO NOT confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash. The actual flash will have occurred when a large flame propagates itself over the surface of the sample.

15.7 If the flash point is expected to be at a certain temperature, you may hold off applying the test flame until you have reached no less than 30° F of the true flash point.

15.8 For the characteristic of ignitability, the analysis should be carried to approximately 210° F for samples that do not flash. The pressure compensated result must be greater than 200° F, and should be recorded as: >200° F.

16.0 CALCULATIONS AND DATA HANDLING

16.1 Basic rules apply for significant figure reporting: report 3 figures over 100 mg/L, report 2 figures below 100 mg/L, and report only 1 place to the right of the decimal for <10 mg/L.

16.2 Observe and record the ambient barometric pressure at the time of the test in mm of mercury. When the pressure differs from 760 mm, correct the flash point as follows:

$$[F + [0.06\,(760 - P)]] = \text{Corrected Flashpoint}$$

Where:

F = observed flash point, ° F
P = ambient barometric pressure, mm mercury

Note: The barometric pressure used in this calculation must be the ambient pressure for the laboratory. The final flash point temperature needs to be adjusted only if the corrected flash point is >1° F from the uncorrect value.

16.3 Record the corrected flash point to the nearest 1° F. The final result is reported as the average of the two duplicate analyses, in ° F. Samples that do not flash are to be reported as >200° F.

17.0 DATA REPORTING AND DELIVERABLES

17.1 The following data is to be put on the benchsheet for each batch turned in:

- Standard log number of primary stock standard.
- Reported flash point in units of ° F.
- Average of the duplicate analyses.

17.2 All flash point results reported from the analysis of solid or waste samples, regardless of whether the result reported is above or below the reporting limit, must be qualified with LIMS qualifier number 94. This qualifier reads:

"SW-846 method 1020 was not designed to be used as the flash point analysis method for solid/solid waste matrices. The positive result or reporting limit reported for this solid sample should not be used to define the characteristic of ignitability."

17.3 See attached copies of batch and data reports (Attachments 23.1, 23.2, and 23.3).

18.0 QUALITY ASSURANCE

18.1 When there is enough sample available, each sample must be run in duplicate. The results from both analyses must be within 3° F of each other. If this criteria is not met, the sample and duplicate must be re-analyzed (if possible), or the result qualified as estimated.

18.2 A Laboratory Control Sample (LCS) of p-xylene from a source other than the reference standard must be analyzed in duplicate with each batch. The average value must be 81° ± 2° F. If this criteria is not met, all samples analyzed in the batch must be re-analyzed or qualified as estimated. Contact the area supervisor if the p-xylene fails this criteria and the samples cannot be re-analyzed. It is advisable to perform the LCS analysis at the beginning of the analytical batch, to identify potential problems and avoid the potential re-analysis of the entire batch of samples.

18.3 All flash point thermometers must be calibrated against a NIST certified thermometer prior to their initial use, and thereafter on an annual basis.

18.4 Solid and solid waste samples results must be qualified as defined in paragraph 17.2.

19.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

19.1 Before the analysis of any actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by running an Initial Demonstration of Capability (IDC). While IDCs are not instrument dependent, one is required on each instrument used in sample analysis, to demonstrate the instrument's ability to generate acceptable accuracy and precision. Annually, a Continuing Demonstration of Capability (CDC) is required.

19.1.1 Initial Demonstration of Capability

19.1.1.1 Analyze four 2 mL aliquots of p-xylene. The average value must meet the criteria of the LCS (81° ± 2° F).

19.1.1.2 If this criteria is not met, locate and correct the source of the problem and repeat the test. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test. Samples may not be analyzed by any analyst or on any instrument until the analyst certification has been successfully completed. Copies of the successful IDC/Method Validation spreadsheet and raw data should be given to the Quality Assurance Manager.

19.1.2 Continuing Demonstration of Capability (CDC)

19.1.2.1 A CDC must be performed annually by all analysts running samples by this SOP. A CDC may be accomplished by repeating the IDC procedure using specific CDC samples, by processing four consecutive LCSs following the IDC procedure, or by

submitting an acceptable PT sample on the appropriate paperwork for inclusion in the analyst's training file.

20.0 POLLUTION PREVENTION

20.1 Maintain an inventory of all chemicals used in the laboratory and monitor their use.

20.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

20.3 Conserve use of chemicals where applicable.

20.4 Comply with all environmental laws associated with chemicals in the laboratory.

21.0 WASTE MANAGEMENT

21.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

21.2 To minimize the environmental impact and costs associated with the disposal of chemicals, order and use only the minimum amount of material required.

21.3 Follow all procedures in TriMatrix Laboratory SOP number GR-15-102, *Laboratory Waste Disposal*, most recent revision, for laboratory waste disposal requirements.

22.0 REFERENCES

22.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition, Final Update III, Revision 1, July, 1992, Method 1020A, "Setaflash Closed-Cup Method for Determining Ignitability".

23.0 ATTACHMENTS

23.1 Preparation Batch Report Example

23.2 Analysis Sequence Report Example

23.3 Data Review Report Example

Attachment 23.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0800584 Page 1 of 1

Printed: 12/16/2008 3:26:18PM

Inorganic - Wet Chemistry, Waste, Method-Specific Preparation

(No Surrogate)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0801032	FlashPoint 1020A	0801059	FlashPoint 1020A	0801108	FlashPoint 1020A
0801156	FlashPoint 1020A	0801158	FlashPoint 1020A	0801159	FlashPoint 1020A
0801218	FlashPoint 1020A				

Lab Number	*Contain*	*Prepared*	*By*	*Initial (g)*	*Final (mL)*	*uL Surrogate*	*Source ID*	*Spike ID*	*uL Spike*	*Client / QC Type*	*Extraction Comments*
0800584-BLK1		Jan-17-08 08:01	INR	2	2					BLANK	
0800584-DUP1		Jan-17-08 08:01	INR	2	2		0801032-02			DUPLICATE	
0800584-BS1		Jan-17-08 08:01	INR	2	2			7120232	2000	LCS	
0801032-02	C	Jan-17-08 08:01	INR	2	2					[redacted]	
0801059-01	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801059-02	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801108-01	B	Jan-17-08 08:01	INR	2	2					[redacted]	
0801156-01	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801158-01	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801158-02	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801159-01	A	Jan-17-08 08:01	INR	2	2					[redacted]	if there are VOCs, they get sample 1st
0801218-01	A	Jan-17-08 08:01	INR	2	2					[redacted]	

Approved By: GM 12-16-08
QA Officer

Approved By: Heather L. Brady
Area Supervisor

Attachment 23.2
Analysis Sequence Report Example

TriMatrix Laboratories, Inc. ANALYSIS SEQUENCE 8011708 Page 1 of 1 Printed: 12/16/2008 3:29:52PM

Inorganic - Wet Chemistry, Waste, Jan-17-08

Instrument = 186, Calibration = UNASSIGNED

Sequence Analyses:
FlashPoint 1020A

Lab Number	*Analysis*	*Contain*	*STD ID*	*ISTD ID*	*Client / QC Type*	*Extraction Comments*
0801032-02	FlashPoint 1020A	C			[redacted]	
0800584-DUP1	QC				DUPLICATE	
0801059-01	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801059-02	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801108-01	FlashPoint 1020A	B			[redacted]	
0801156-01	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801158-01	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801158-02	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801159-01	FlashPoint 1020A	A			[redacted]	if there are VOCs, they get sample 1st
0801218-01	FlashPoint 1020A	A			[redacted]	
0800584-BS1	QC				LCS	
8011708-SCV1	QC		7120233		SECONDARY CAL CHECK	
0800584-BLK1	QC				BLANK	

Attachment 23.3
Data Review Report Example

TriMatrix Laboratories, Inc.

Data Review Report -- Wet Chem Lab

Sequence = 8011708 Page 1 of 1

on 12/16/2008 at 3:33

SampleID	Analysis	IResult	Diln	FResult	FMRL	Qualifier	Recovery	RPD	Analyzed	Analyst	Batch
0801032-02	FlashPoint 1020A	999	1	999	68	GN014,WC015			1/17/2008 1:01	INR	0800584
0800584-DUP1	FlashPoint 1020A	999	1	999	68	WC015		0	1/17/2008 1:01	INR	0800584
0801059-01	FlashPoint 1020A	999	1	999	68	GN014,WC015			1/17/2008 1:01	INR	0800584
0801059-02	FlashPoint 1020A	999	1	999	68	GN014,WC015			1/17/2008 1:01	INR	0800584
0801108-01	FlashPoint 1020A	999	1	999	68	GN014,WC015			1/17/2008 1:01	INR	0800584
0801156-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801158-01	FlashPoint 1020A	171	1	171	68				1/17/2008 1:01	INR	0800584
0801158-02	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801159-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0801218-01	FlashPoint 1020A	999	1	999	68	WC015			1/17/2008 1:01	INR	0800584
0800584-BS1	FlashPoint 1020A	81	1	81	68		100		1/17/2008 1:01	INR	0800584
8011708-SCV1	FlashPoint 1020A	81	1	81			100		1/17/2008 1:01	INR	8011708
0800584-BLK1	FlashPoint 1020A	0	1	0	68	WC015			1/17/2008 1:01	INR	0800584

Approved By: 12-16-08
QA Officer

Approved By: Heather L. Brady
Area Supervisor

The American Association for Laboratory Accreditation

World Class Accreditation

Accredited DoD ELAP Laboratory

A2LA has accredited

TRIMATRIX LABORATORIES, INC.

Grand Rapids, MI

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (QSM v4.1); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

Presented this 25th day of January 2010.

President & CEO
For the Accreditation Council
Certificate Number 675.01
Revised January 25, 2010
Valid to April 30, 2011

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TRIMATRIX LABORATORIES INC.
5560 Corporate Exchange Court SE
Grand Rapids, MI 49512
Rick Wilburn Phone: 616 975 4500

ENVIRONMENTAL

Valid To: April 30, 2011 Certificate Number: 0675.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1)) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a) as it pertains to the A2LA Kentucky Underground Storage Tank Laboratory Accreditation Program:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, Cold Vapor Atomic Fluorescence Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, F^-, O_2), Hazardous Waste Characteristic Tests, Spectrophotometry (Visible), Spectrophotometry (Automated) and Titrimetry.

Parameter/Analyte	**Nonpotable Water**	**Solid Hazardous Waste**	
		Aqueous	**Solid**
Metals			
Aluminum	EPA 200.7	EPA 6010C	EPA 6010C
Antimony	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Arsenic	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Barium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Beryllium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Boron	EPA 200.7	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Cadmium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Calcium	EPA 200.7	EPA 6010C	EPA 6010C

Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Chromium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Cobalt	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Copper	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Iron	EPA 200.7	EPA 6010C	EPA 6010C
Lead	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Lithium	EPA 200.7	EPA 6010C	EPA 6010C
Magnesium	EPA 200.7	EPA 6010C	EPA 6010C
Manganese	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Mercury	EPA 245.1 EPA 1631E	EPA 7470A	EPA 7471A
Molybdenum	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Nickel	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Potassium	EPA 200.7	EPA 6010C	EPA 6010C
Selenium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Silicon	EPA 200.7	EPA 6010C	EPA 6010C
Silver	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Sodium	EPA 200.7	EPA 6010C	EPA 6010C
Strontium	EPA 200.7	EPA 6010C	EPA 6010C
Thallium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Tin	EPA 200.7 Tri GR-01-129 ICPMS	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Titanium	EPA 200.7	EPA 6010C	EPA 6010C
Vanadium	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Zinc	EPA 200.7 EPA 200.8	EPA 6010C EPA 6020A	EPA 6010C EPA 6020A
Nutrients			
Ammonia (as N)	SM 4500 NH_3 G	SM 4500 NH_3 G	SM 4500 NH_3 G
Nitrate (as N)	EPA 300.0 SM 4500 NO_3 F	EPA 9056A	EPA 9056A
Nitrate-nitrite (as N)	EPA 300.0 SM 4500 NO_3 F	EPA 9056A	EPA 9056A
Nitrite (as N)	EPA 300.0 SM 4500 NO_2 B SM 4500 NO_3 F	EPA 9056A	EPA 9056A
Orthophosphate (as P)	SM 4500 PE	- - - - - - - - - -	- - - - - - - - - -
Total phosphorus	SM 4500 PE	SM 4500 PE	SM 4500 PE

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Total kjeldahl nitrogen	EPA 351.2	- - - - - - - - - -	- - - - - - - - - -
Demands			
Biochemical oxygen demand	SM 5210 B	- - - - - - - - - -	- - - - - - - - - -
Carbonaceous BOD	SM 5210 B	- - - - - - - - - -	- - - - - - - - - -
Chemical oxygen demand	SM 5220 D	- - - - - - - - - -	- - - - - - - - - -
Total organic carbon	SM 5310 C	EPA 9060	Walkley Black (MSA 29-3.5-2)
Wet Chemistry			
Acidity	SM 2310 B	- - - - - - - - - -	- - - - - - - - - -
Alkalinity	SM 2320 B	- - - - - - - - - -	- - - - - - - - - -
Bromide	EPA 300.0	EPA 9056A	EPA 9056A
Chloride	EPA 300.0 SM 4500 Cl E	EPA 9056A	EPA 9056A
Chromium (VI) Cr 6+	SM 3500 Cr B	EPA 7196A	EPA 7196A
Cyanide	SM 4500 CN E	EPA 9014	EPA 9014
Cyanide Amenable to Chlorination	SM 4500 CN G	EPA 9014	EPA 9014
Extractable Cyanide	- - - - - - - - - -	EPA 9010B	EPA 9010B
Fluoride	EPA 300.0 SM 4500 F C	EPA 9056A	EPA 9056A SM 4500 F C
Hardness (total and calcium)	SM 2340 B SM 2340 C	- - - - - - - - - -	- - - - - - - - - -
Oil and Grease / TPH	EPA 1664A	EPA 1664A EPA 9070A	EPA 9071B
pH	SM 4500 H B	EPA 9040B	EPA 9045C
Phenolics	EPA 420.4	EPA 9065	EPA 9065
Conductivity	SM 2510 B	EPA 9050A	- - - - - - - - - -
Sulfate	EPA 300.0 ASTM D 516-90 (02)	EPA 9038 EPA 9056A	EPA 9038 EPA 9056A
Sulfide	SM 4500 S_2 D, F	EPA 9030B EPA 9034	EPA 9030B EPA 9034
Sulfite	SM 4500 SO_3 B	- - - - - - - - - -	- - - - - - - - - -
Surfactants (MBAS)	SM 5540 C	- - - - - - - - - -	- - - - - - - - - -
Total Residue	SM 2540 B Tri GR-07-115 Grav	- - - - - - - - - -	- - - - - - - - - -
Dissolved Residue (TDS)	SM 2540 C	- - - - - - - - - -	- - - - - - - - - -
Suspended Residue (TSS)	SM 2540 D	- - - - - - - - - -	- - - - - - - - - -
Turbidity	SM 2130 B	- - - - - - - - - -	- - - - - - - - - -
Volatile Residue	EPA 160.4	- - - - - - - - - -	- - - - - - - - - -
Color	EPA 2120B	- - - - - - - - - -	- - - - - - - - - -
Available Cyanide	OIC-1677	- - - - - - - - - -	- - - - - - - - - -
Purgeable Organics			
Acetone	- - - - - - - - - -	EPA 8260B	EPA 8260B
Acetonitrile	EPA 624	EPA 8260B	EPA 8260B
Acrolein	EPA 624	EPA 8260B	EPA 8260B
Acrylonitrile	EPA 624	EPA 8260B	EPA 8260B
Ally Chloride)3-chchloropropene)	- - - - - - - - -	EPA 8260B	EPA 8260B

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Benzene	EPA 602 EPA 624	EPA 8260B EPA 8021B	EPA 8260B
Bromobenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
Bromochloromethane	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
Bromodichloromethane	EPA 601 EPA 624	EPA 8260B EPA 8021B	EPA 8260B
Bromoform	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Bromomethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Carbon disulfide	- - - - - - - - - -	EPA 8260B	EPA 8260B
Carbon tetrachloride	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Chlorobenzene	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Chloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Chloroform	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Chloromethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Chloroprene	- - - - - - - - - -	EPA 8260B	EPA 8260B
1-Chlorohexane	- - - - - - - - -	EPA 8260B	EPA 8260B
Cyclohexane	- - - - - - - - -	EPA 8260B	EPA 8260B
cis-1,2-Dichloroethylene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
cis-1,3-Dichloropropene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Dibromochloromethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Dibromomethane	- - - - - - - - - -	EPA 8260B	EPA 8260B
Dichlorodifluoromethane	EPA 601	EPA 8021B EPA 8260B	EPA 8260B
Diethyl ether	- - - - - - - - - -	EPA 8260B	EPA 8260B
Ethanol	- - - - - - - - - -	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Ethyl Acetate	- - - - - - - - - -	EPA 8260B	EPA 8260B
Ethylmethacrylate	- - - - - - - - - -	EPA 8260B	EPA 8260B
Ethybenzene	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Gasoline Range Organics	- - - - - - - - - -	EPA 8015B	EPA 8015B
Hexachlorobutadiene	- - - - - - - - - -	EPA 8260B	EPA 8260B
Hexachloroethane	- - - - - - - - -	EPA 8260B	EPA 8260B
Iodomethane	- - - - - - - - - -	EPA 8260B	EPA 8260B

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Isobutyl Alcohol	- - - - - - - - - -	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Isopropyl Alcohol (2-Propanol)	- - - - - - - - - -	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
Isopropyl ether (DIPE)	- - - - - - - - -	EPA 8260B	EPA 8260B
Ethane	RSK-175	- - - - - - - - - -	- - - - - - - - - -
Ethylene	RSK-175	- - - - - - - - - -	- - - - - - - - - -
Methane	RSK-175	- - - - - - - - - -	- - - - - - - - - -
Isopropylbenzene	- - - - - - - - - -	EPA 8260B	EPA 8260B
Methacrylonitrile	- - - - - - - - - -	EPA 8260B	EPA 8260B
Methanol	- - - - - - - - - -	EPA 8015B	EPA 8015B
Methylcyclohexane	- - - - - - - - -	EPA 8260B	EPA 8260B
Methylene chloride	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Methyl ethyl ketone (MEK) (2 Butanone)	- - - - - - - - - -	EPA 8260B	EPA 8260B
Methyl methacrylate	- - - - - - - - - -	EPA 8260B	EPA 8260B
Methyl tert-butyl ether (MTBE)	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
2-Nitropropane	- - - - - - - - -	EPA 8260B	EPA 8260B
Naphthalene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
n-Butyl Alcohol	- - - - - - - - - -	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
n-Butylbenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
n-Proppyl Alcohol	- - - - - - - - -	EPA 8015B EPA 8260B	EPA 8015B EPA 8260B
n-Propylbenzene	- - - - - - - - - -	EPA 8260B	EPA 8260B
Paraldehyde	- - - - - - - - - -	EPA 8015B	EPA 8015B
Propionitrile	- - - - - - - - - -	EPA 8260B	EPA 8260B
Sec-Butylbenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
Styrene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,1,1,2-Tetrachloroethane	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,1,1-Trichloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Freon-113 (1,1,2-Trichloro-1,2,2,-Trifluoroethane)	EPA 624	EPA 8260B	EPA 8260B
1,1,2-Trichloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B

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		Aqueous	Solid
1,1-Dichloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,1-Dichloroethene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,1-Dichloropropene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,2,3-Trichlorobenzene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,2,3-Trichloropropane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,2,4-Trichlorobenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,2,4-Trimethylbenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dibromo-3-Chloropropane (DBCP)	- - - - - - - - - -	EPA 8021B EPA 8260B EPA 8011	EPA 8260B
1,2-Dibromoethane (EDB)	- - - - - - - - - -	EPA 8021b EPA 8260B EPA 8011	EPA 8260B
1,2-Dichlorobenzene	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dichloroethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,2-Dichloropropane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,3,5-Trimethylbenzene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,3-Dichlorobenzene	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,3-Dichloropropane	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
1,4-Dichlorobenzene	EPA 601 EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
1,4-Dioxane (p-Dioxane)	- - - - - - - - - -	EPA 8260B EPA 8270C	EPA 8260B
2,2-Dichloropropane	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
2-Chloroethyl vinyl ether	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
2-Chlorotoluene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
2-Hexanone	- - - - - - - - - -	EPA 8260B	EPA 8260B
4-Chlorotoluene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
4-Isopropyltoluene	- - - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
4-Methyl-2-Pentanone (MIBK)	- - - - - - - - - -	EPA 8260B	EPA 8260B
tert-amyl methyl ether (tAME)	- - - - - - - - - -	EPA 8260B	EPA 8260B
tert-butyl alcohol	- - - - - - - - -	EPA 8260B	EPA 8260B
tert-butyl benzene	- - - - - - - - -	EPA 8021B EPA 8260B	EPA 8260B
Tetrachloroethene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Toluene	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
trans-1,2-Dichloroethene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
trans-1,3-Dichloropropene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
trans-1,4-Dichloro-2-Butene	- - - - - - - - - -	EPA 8260B	EPA 8260B
Trichloroethylene	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Trichlorofluoromethane	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Vinyl acetate	- - - - - - - - - -	EPA 8260B	EPA 8260B
Vinyl chloride	EPA 601 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
m-Xylene	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
o-Xylene	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
p-Xylene	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Xylenes Total	EPA 602 EPA 624	EPA 8021B EPA 8260B	EPA 8260B
Extractable Organics (Semivolatiles)			
Acenaphthene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Acenaphthylene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Acetophenone	- - - - - - - - -	EPA 8270C	EPA 8270C
Acetaldehyde	- - - - - - - - -	EPA 8315A	- - - - - - - - -
2-Acetylaminofluorene	- - - - - - - - -	EPA 8270C	- - - - - - - - -
4-Aminobiphenyl	- - - - - - - - -	EPA 8270C	EPA 8270C
2-Amino-4,6-Dinitrotoluene	- - - - - - - - -	EPA 8330	EPA 8330
4-Amino-2,6-Dinitrotoulene	- - - - - - - - - -	EPA 8330	EPA 8330
Aniline	- - - - - - - - - -	EPA 8270C	EPA 8270C
Anthracene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Aramite	- - - - - - - - - -	EPA 8270C	- - - - - - - - -
Benzidine	EPA 625	EPA 8270C	- - - - - - - - -

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Benzoic acid	- - - - - - - - - -	EPA 8270C	EPA 8270C
Benzaldehyde	- - - - - - - - - -	EPA 8270C	EPA 8270C
Benzo(a)anthracene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(b)fluoranthene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(k)fluoranthene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(ghi)perylene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzo(a)pyrene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Benzyl alcohol	- - - - - - - - - -	EPA 8270C	EPA 8270C
Biphenyl	- - - - - - - - - -	EPA 8270C	EPA 8270C
Bis (2-Chloroethoxy) methane	EPA 625	EPA 8270C	EPA 8270C
Bis (2-Chloroethyl) ether	EPA 625	EPA 8270C	EPA 8270C
Bis (2-Chloroisopropyl) ether	EPA 625	EPA 8270C	EPA 8270C
Bis (2-Ethylhexyl) phthalate	EPA 625	EPA 8270C	EPA 8270C
4-Bromophenyl phenyl ether	EPA 625	EPA 8270C	EPA 8270C
Butanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Butyl benzyl phthalate	EPA 625	EPA 8270C	EPA 8270C
Caprolactam	- - - - - - - - - -	EPA 8270C	EPA 8270C
Carbazole	- - - - - - - - - -	EPA 8270C	EPA 8270C
4-Chloroaniline	EPA 625	EPA 8270C	EPA 8270C
2-Chloronaphthalene	EPA 625	EPA 8270C	EPA 8270C
2-Chlorophenol	EPA 625	EPA 8270C	EPA 8270C
Chrysene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
4-Chloro-3-methylphenol	EPA 625	EPA 8270C	EPA 8270C
4-Chlorophenyl phenyl ether	EPA 625	EPA 8270C	EPA 8270C
Crotonaldehyde	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Cyclohexanone	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Decanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Dibenzo(a,h)anthracene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Dibenzofuran	- - - - - - - - - -	EPA 8270C	EPA 8270C
1,2-Dichlorobenzene	EPA 625	EPA 8270C	EPA 8270C
1,3-Dichlorobenzene	EPA 625	EPA 8270C	EPA 8270C
1,4-Dichlorobenzene	EPA 625	EPA 8270C	EPA 8270C
3,3-Dichlorobenzidine	EPA 625	EPA 8270C	EPA 8270C
2,4-Dichlorophenol	EPA 625	EPA 8270C	EPA 8270C
2,6-Dichlorophenol	- - - - - - - - - -	EPA 8270C	EPA 8270C
Diethyl Phthalate	EPA 625	EPA 8270C	EPA 8270C
Diesel Range Organics (DRO)	- - - - - - - - - -	EPA 8015B	EPA 8015B
Dimethoate	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
4-Dimethylaminoazobenzene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
7,12-Dimethylbenz(a)anthracene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
3,3-Dimethylbenzidine	- - - - - - - - - -	EPA 8270C	EPA 8270C

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
2,4-Dimethylphenol	EPA 625	EPA 8270C	EPA 8270C
Dimethyl Phthalate	EPA 625	EPA 8270C	EPA 8270C
Di-n-butyl phthalate	EPA 625	EPA 8270C	EPA 8270C
Di-n-octyl phthalate	EPA 625	EPA 8270C	EPA 8270C
Dinoseb (2-sec-Butyl-46-Dinitrophen)	- - - - - - - - - -	EPA 8270C EPA 8151A	EPA 8151A
2,4-Dinitrophenol	EPA 625	EPA 8270C	EPA 8270C
2,4-Dinitrotoluene	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330
2,6-Dinitrotoluene	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330
1,3-Dinitrobenzene	- - - - - - - - - -	EPA 8270C EPA 8330	EPA 8270C EPA 8330
1,2-Diphenylhydrazine	- - - - - - - - - -	EPA 8270C	EPA 8270C
Diphenylamine	EPA 625	EPA 8270C	EPA 8270C
Disulfonton	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Ethyl methane sulfonate	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Famphur	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Fluoranthene	EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Fluorene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Formaldehyde	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Heptanal	- - - - - - - - - -	EPA 8315A	
Hexachlorobenzene	EPA 625	EPA 8270C	EPA 8270C
Hexachlorobutadiene	EPA 625	EPA 8270C	EPA 8270C
Hexachlorocyclopentadiene	EPA 625	EPA 8270C	EPA 8270C
Hexachloroethane	EPA 625	EPA 8270C	EPA 8270C
Hexachloropropene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Hexanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
HMX	- - - - - - - - - -	EPA 8330	EPA 8330
Indeno (1,2,3-cd) Pyrene	EPA 610 EPA 625	EPA 8270 EPA 8310	EPA 8270 EPA 8310
Isophorone	EPA 625	EPA 8270C	EPA 8270C
Isosafrole	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Kepone	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Methapyrilene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
3-Methylcholanthrene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
2-Methyl-4,6-dinitrophenol	EPA 625	EPA 8270C	EPA 8270C
Methyl methanesSulfonate	- - - - - - - - - -	EPA 8270C	EPA 8270C
1-Methylnaphthalene	- - - - - - - - - -	EPA 8270C	EPA 8270C
2-Methylnaphthalene	- - - - - - - - - -	EPA 8270C	EPA 8270C
2-Methylphenol	- - - - - - - - - -	EPA 8270C	EPA 8270C
3-Methylphenol	- - - - - - - - - -	EPA 8270C	EPA 8270C
4-Methylphenol	- - - - - - - - - -	EPA 8270C	EPA 8270C
Naphthalene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
1,4-Naphthoquinone	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
1-Naphthylamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
2-Naphthylamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
2-Nitroaniline	- - - - - - - - - -	EPA 8270C	EPA 8270C
3-Nitroaniline	- - - - - - - - - -	EPA 8270C	EPA 8270C
4-Nitroaniline	- - - - - - - - - -	EPA 8270C	EPA 8270C
Nitrobenzene	EPA 625	EPA 8270C EPA 8330	EPA 8270C EPA 8330
Nitroquinoline-1-Oxide	- - - - - - - - - -	EPA 8270C	EPA 8270C
2-Nitrophenol	EPA 625	EPA 8270C	EPA 8270C
4-Nitrophenol	EPA 625	EPA 8270C	EPA 8270C
n-Nitrosodiethylamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
n-Nitroso-di-n-butylamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
n-Nitroso-di-methylamine	EPA 625	EPA 8270C	EPA 8270C
n-Nitroso-di-n-propylamine	EPA 625	EPA 8270C	EPA 8270C
n-Nitroso-di-Phenylamine	EPA 625	EPA 8270C	EPA 8270C
n-Nitrosomethylethylamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
n-Nitrosomorpholine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
n-Nitrosopiperidine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
n-Nitrosopyrrolidine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
2-Nitrotoluene	- - - - - - - - - -	EPA 8330	EPA 8330
3-Nitrotoluene	- - - - - - - - - -	EPA 8330	EPA 8330
4-Nitrotoluene	- - - - - - - - - -	EPA 8330	EPA 8330
5-Nitro-o-toluidine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Nonanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Octanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
ooo-Tiethyl phosphorothioate	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
o-Toluidine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Parathion ethyl	- - - - - - - - - -	EPA 8270C	EPA 8270C
Parathion methyl	- - - - - - - - - -	EPA 8270C	EPA 8270C
Pentachlorobenzene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Pentachloronitrobenzene	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Pentachlorophenol	EPA 625	EPA 8270C	EPA 8270C
Pentanal	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Phenacetin	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Phenanthrene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Phenol	EPA 625	EPA 8270C	EPA 8270C
1,4-Phenylenediamine	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Phorate	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
2-Picoline	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Pronamide	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Propanal (Propionaldehyde)	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
Pyrene	EPA 610 EPA 625	EPA 8270C EPA 8310	EPA 8270C EPA 8310
Pyridine	- - - - - - - - - -	EPA 8270C	EPA 8270C
RDX	- - - - - - - - - -	EPA 8330	EPA 8330

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Safrole	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Sulfotepp	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
Tetryl	- - - - - - - - - -	EPA 8330	EPA 8330
1,2,4,5-Tetrachlorobenzene	EPA 625	EPA 8270C	EPA 8270C
Thionazin	- - - - - - - - - -	EPA 8270C	- - - - - - - - - -
1,3-Tolualdehyde	- - - - - - - - - -	EPA 8315A	- - - - - - - - - -
1,2,4-Trichlorobenzene	EPA 625	EPA 8270C	EPA 8270C
2,3,4, 6-Tetrachlorophenol	- - - - - - - - -	EPA 8270C	EPA 8270C
1,3,5-Trinitrobenzene	- - - - - - - - - -	EPA 8330	EPA 8330
2,4,6-Trinitrotoluene	- - - - - - - - - -	EPA 8330	EPA 8330
2,4,5-Trichlorophenol	- - - - - - - - - -	EPA 8270C	EPA 8270C
2,4,6-Trichlorophenol	EPA 625	EPA 8270C	EPA 8270C
Nitroglycerin	- - - - - - - - - -	EPA 8332	EPA 8332
PETN	- - - - - - - - - -	EPA 8332	EPA 8332
Pesticides-Herbicides-PCBs			
Aldrin	EPA 608	EPA 8081A	EPA 8081A
a-BHC	EPA 608	EPA 8081A	EPA 8081A
b-BHC	EPA 608	EPA 8081A	EPA 8081A
d-BHC	EPA 608	EPA 8081A	EPA 8081A
g-BHC	EPA 608	EPA 8081A	EPA 8081A
Chlorobenzilate	- - - - - - - - - -	EPA 8270C	EPA 8270C
Chlordane	EPA 608	EPA 8081A	EPA 8081A
2,4-D	- - - - - - - - - -	EPA 8151A EPA 8270C	EPA 8151A
Dalapon	- - - - - - - - - -	EPA 8151A	EPA 8151A
2,4-DB	- - - - - - - - - -	EPA 8151A	EPA 8151A
2,4,5-T	- - - - - - - - - -	EPA 8151A	EPA 8151A
Silvex (2,4,5-TP)	- - - - - - - - - -	EPA 8151A	EPA 8151A
4,4”-DDD	EPA 608	EPA 8081A	EPA 8081A
4,4” DDE	EPA 608	EPA 8081A	EPA 8081A
4,4”-DDT	EPA 608	EPA 8081A	EPA 8081A
Diallate	- - - - - - - - - -	EPA 8270C	EPA 8270C
Dicamba	- - - - - - - - - -	EPA 8151A	EPA 8151A
Dichlorprop	- - - - - - - - - -	EPA 8151A	EPA 8151A
Dieldrin	EPA 608	EPA 8081A	EPA 8081A
Dinoseb	- - - - - - - - - -	EPA 8151A	EPA 8151A
Endosulfan I (alpha)	EPA 608	EPA 8081A	EPA 8081A
Endosulfan II (beta)	EPA 608	EPA 8081A	EPA 8081A
Endosulfan sulfate	EPA 608	EPA 8081A	EPA 8081A
Endrin	EPA 608	EPA 8081A	EPA 8081A
Endrin aldehyde	EPA 608	EPA 8081A	EPA 8081A
Endrin ketone	EPA 608	EPA 8081A	EPA 8081A
a-Chlordane	EPA 608	EPA 8081A	EPA 8081A
g-Chlordane	EPA 608	EPA 8081A	EPA 8081A
Heptachlor	EPA 608	EPA 8081A	EPA 8081A

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Parameter/Analyte	Nonpotable Water	Solid Hazardous Waste	
		Aqueous	Solid
Heptachlor epoxide	EPA 608	EPA 8081A	EPA 8081A
Isodrin	- - - - - - - - - -	EPA 8270C	EPA 8270C
Kepone	- - - - - - - - - -	EPA 8270C	EPA 8270C
Lindane	EPA 608	EPA 8081A	EPA 8081A
Methoxychlor	EPA 608.2	EPA 8081A	EPA 8081A
MCPA	- - - - - - - - - -	EPA 8151A	EPA 8151A
MCPP	- - - - - - - - - -	EPA 8151A	EPA 8151A
Toxaphene	EPA 608	EPA 8081A	EPA 8081A
PCB 1016	EPA 608	EPA 8082	EPA 8082
PCB 1221	EPA 608	EPA 8082	EPA 8082
PCB 1232	EPA 608	EPA 8082	EPA 8082
PCB 1242	EPA 608	EPA 8082	EPA 8082
PCB 1248	EPA 608	EPA 8082	EPA 8082
PCB 1254	EPA 608	EPA 8082	EPA 8082
PCB 1260	EPA 608	EPA 8082	EPA 8082
PCB 1262	EPA 608	EPA 8082	EPA 8082
PCB 1268	EPA 608	EPA 8082	EPA 8082
Hazardous Waste Characteristics			
Corrosivity	- - - - - - - - - -	EPA 9040B	EPA 9045C
Ignitability	- - - - - - - - - -	- - - - - - - - - -	EPA 1020A
Paint Filter Liquid Test	- - - - - - - - - -	- - - - - - - - - -	EPA 9095A
TCLP	- - - - - - - - - -	- - - - - - - - - -	EPA 1311
SPLP	- - - - - - - - - -	- - - - - - - - - -	EPA 1312
Total Organic Halides	- - - - - - - - - -	EPA9020B	- - - - - - - - - -

Kentucky UST Program

Gasoline Range Organics	- - - - - - - - - -	- - - - - - - - - -	EPA 8015B
Diesel Range Organics (DRO)	- - - - - - - - - -	- - - - - - - - - -	EPA 8015B
Oil and Grease / TPH	- - - - - - - - - -	- - - - - - - - - -	EPA 9071B
TCLP Metals			
Arsenic	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Barium	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Cadmium	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Chromium	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Lead	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Mercury	- - - - - - - - - -	- - - - - - - - - -	EPA 7471A
Selenium	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A
Silver	- - - - - - - - - -	- - - - - - - - - -	EPA 6010C EPA 6020A

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Preparation Methods

Fraction	Analytical Method	Preparation Method
Metals by ICP	EPA 200.7 EPA 6010C	EPA 3005A EPA 3010A EPA 3020A EPA 3050B EPA 200.2
Metals by ICP/MS	EPA 200.8 EPA 6020A	EPA 3005A EPA 3010A EPA 3020A EPA 3050B EPA 200.2
Hexavalent Chromium	EPA 7196A	EPA 3060A
Organochlorine Pesticides	EPA 8081B	EPA 3510C EPA 3550B EPA 3545
Polychlorinated Biphenyls (PCBs)	EPA 8082	EPA 3510C EPA 3550B EPA 3545
BNAs	EPA 8270C	EPA 3510C EPA 3550B EPA 3545
Volatiles	EPA 8260B	EPA 5030B EPA 5035A
Diesel Range Organics	EPA 8015B	EPA 3510C EPA 3550B EPA 3545
Gasoline Range Organics	EPA 8015B	EPA 5030B EPA 5035A
Sulfide	EPA 9034	EPA 9030B
Cyanide	EPA 9010B	EPA 9013A

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STATE OF NEW YORK
DEPARTMENT OF HEALTH

Wadsworth Center The Governor Nelson A. Rockefeller Empire State Plaza P.O. BOX 509 Albany, New York 12201-0509

RICHARD F. DAINES, M.D.
Commissioner

WENDY E. SAUNDERS
Executive Deputy Commissioner

LAB ID: 11776

April 01, 2009

MR. JEFF GLASER
TRIMATRIX LABORATORIES, INC
5560 CORPORATE EXCHANGE CT SE
GRAND RAPIDS, MI 49512-5503

Certificate Expiration Date:
April 01, 2010

Dear Mr. Glaser,

Enclosed are the ELAP and/or NELAP Certificate(s) of Approval issued to your environmental laboratory for the current permit year. The Certificate(s) supersede any previously issued and is(are) in effect through the expiration date listed above. Please carefully examine the Certificate(s) to insure that the categories, subcategories, analytes and methods for which your laboratory is approved are listed correctly, as well as verifying your laboratory's name, address, lead technical director and identification number.

Pursuant to regulation (Part 55-2 NYCRR), original certificates must be posted conspicuously in the laboratory and copies shall be made available to any client of the laboratory upon request. Certificates remain the property of the New York State Department of Health and must be surrendered promptly on demand.

Please note, pursuant to Section 55-2.6 NYCRR, any misrepresentation of the Fields of Accreditation (Matrix - Method - Analyte) for which your laboratory is approved may result in denial, suspension, or revocation of your certification. Any use of the ELAP or NELAP name, reference to the laboratory's approval status and/or using the NELAC/NELAP logo in any catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports or other materials must include the laboratory's ELAP identification number, and must distinguish between proposed testing for which the laboratory is approved and the proposed testing for which the laboratory is not approved.

Please notify the ELAP office of any changes you feel need to be made to your Certificate(s). We may be reached via email to elap@health.state.ny.us or by calling (518) 485-5570.

Sincerely,

Joyce Reilly
Program Administrator
Environmental Laboratory
Approval Program

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
RICHARD F. DAINES, M.D.

Expires 12:01 AM April 01, 2010
Issued April 01, 2009

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JEFF GLASER
TRIMATRIX LABORATORIES, INC
5560 CORPORATE EXCHANGE CT SE
GRAND RAPIDS, MI 49512-5503

NY Lab Id No: 11776
EPA Lab Code: MI00005

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 624
	EPA 8260B
Acrylonitrile	EPA 624
	EPA 8260B
Ethyl methacrylate	EPA 8260B
Methyl acrylonitrile	EPA 8260B
Methyl methacrylate	EPA 8260B
Amines	
1,4-Phenylenediamine	EPA 8270C
1-Naphthylamine	EPA 8270C
2-Naphthylamine	EPA 8270C
2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
5-Nitro-o-toluidine	EPA 8270C
Aniline	EPA 8270C
Carbazole	EPA 8270C
Methapyrilene	EPA 8270C
Pronamide	EPA 8270C
Propionitrile	EPA 8260B
Pyridine	EPA 8270C
Benzidines	
3,3'-Dichlorobenzidine	EPA 625
	EPA 8270C
3,3'-Dimethylbenzidine	EPA 8270C
Benzidine	EPA 625

Benzidines	
Benzidine	EPA 8270C
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
	EPA 8081A
4,4'-DDE	EPA 608
	EPA 8081A
4,4'-DDT	EPA 608
	EPA 8081A
Aldrin	EPA 608
	EPA 8081A
alpha-BHC	EPA 608
	EPA 8081A
alpha-Chlordane	EPA 8081A
beta-BHC	EPA 608
	EPA 8081A
Chlordane Total	EPA 608
	EPA 8081A
Chlorobenzilate	EPA 8270C
delta-BHC	EPA 608
	EPA 8081A
Diallate	EPA 8081A
	EPA 8270C
Dieldrin	EPA 608
	EPA 8081A
Endosulfan I	EPA 608
	EPA 8081A
Endosulfan II	EPA 608
	EPA 8081A

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Chlorinated Hydrocarbon Pesticides

Endosulfan sulfate	EPA 608
	EPA 8081A
Endrin	EPA 608
	EPA 8081A
Endrin aldehyde	EPA 608
	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Heptachlor	EPA 608
	EPA 8081A
Heptachlor epoxide	EPA 608
	EPA 8081A
Isodrin	EPA 8270C
Kepone	EPA 8270C
Lindane	EPA 608
	EPA 8081A
Methoxychlor	EPA 8081A
PCNB	EPA 8270C
Toxaphene	EPA 608
	EPA 8081A

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8021B
	EPA 8260B
1,2,4,5-Tetrachlorobenzene	EPA 8270C
1,2,4-Trichlorobenzene	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270C

Chlorinated Hydrocarbons

2-Chloronaphthalene	EPA 625
	EPA 8270C
Hexachlorobenzene	EPA 625
	EPA 8270C
Hexachlorobutadiene	EPA 625
	EPA 8260B
	EPA 8270C
Hexachlorocyclopentadiene	EPA 625
	EPA 8270C
Hexachloroethane	EPA 625
	EPA 8270C
Hexachloropropene	EPA 8270C
Pentachlorobenzene	EPA 8270C

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
	EPA 8270C

Demand

Biochemical Oxygen Demand	SM 18-20 5210B (01)
Carbonaceous BOD	SM 18-20 5210B (01)
Chemical Oxygen Demand	SM 18-20 5220D (97)

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Fuel Oxygenates

Di-isopropyl ether	EPA 8260B
Ethanol	EPA 8015 B
	EPA 8260B
Methyl tert-butyl ether	EPA 8021B
	EPA 8260B
tert-Butyl alcohol	EPA 8260B

Haloethers

4-Bromophenylphenyl ether	EPA 625
	EPA 8270C
4-Chlorophenylphenyl ether	EPA 625
	EPA 8270C
Bis (2-chloroisopropyl) ether	EPA 625
	EPA 8270C
Bis(2-chloroethoxy)methane	EPA 625
	EPA 8270C
Bis(2-chloroethyl)ether	EPA 625
	EPA 8270C

Low Level Polynuclear Aromatics

Acenaphthene	EPA 610
	EPA 8310
Acenaphthylene	EPA 610
	EPA 8310
Anthracene	EPA 610
	EPA 8310
Benzo(a)anthracene	EPA 610
	EPA 8310
Benzo(a)pyrene	EPA 610

Low Level Polynuclear Aromatics

Benzo(a)pyrene	EPA 8310
Benzo(b)fluoranthene	EPA 610
	EPA 8310
Benzo(g,h,i)perylene	EPA 610
	EPA 8310
Benzo(k)fluoroanthene	EPA 610
	EPA 8310
Chrysene	EPA 610
	EPA 8310
Dibenzo(a,h)anthracene	EPA 610
	EPA 8310
Fluoranthene	EPA 610
	EPA 8310
Fluorene	EPA 610
	EPA 8310
Indeno(1,2,3-cd)pyrene	EPA 610
	EPA 8310
Naphthalene	EPA 610
	EPA 8310
Phenanthrene	EPA 610
	EPA 8310
Pyrene	EPA 610
	EPA 8310

Microextractables

1,2-Dibromo-3-chloropropane	EPA 8021B
	EPA 8260B
1,2-Dibromoethane	EPA 8021B
	EPA 8260B

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Mineral

Alkalinity	SM 18-21 2320B (97)
Chloride	EPA 300.0 Rev. 2.1
	EPA 9056
	SM 18-20 4500-Cl- E (97)
Fluoride, Total	EPA 300.0 Rev. 2.1
	EPA 9056
	SM 18-21 4500-F C (97)
Hardness, Total	SM 18-20 2340B (97)
	SM 18-20 2340C (97)
Sulfate (as SO4)	ASTM D516-90 & 02
	EPA 300.0 Rev. 2.1
	EPA 9056

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8270C
	EPA 8330
1,3-Dinitrobenzene	EPA 8270C
	EPA 8330
1,4-Naphthoquinone	EPA 8270C
2,4,6-Trinitrotoluene	EPA 8330
2,4-Dinitrotoluene	EPA 625
	EPA 8270C
	EPA 8330
2,6-Dinitrotoluene	EPA 625
	EPA 8270C
	EPA 8330
2-Amino-4,6-dinitrotoluene	EPA 8330
2-Nitrotoluene	EPA 8330
3-Nitrotoluene	EPA 8330

Nitroaromatics and Isophorone

4-Amino-2,6-dinitrotoluene	EPA 8330
4-Nitrotoluene	EPA 8330
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330
Isophorone	EPA 625
	EPA 8270C
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330
Nitrobenzene	EPA 625
	EPA 8270C
	EPA 8330
Octahydro-tetranitro-tetrazocine	EPA 8330

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270C
N-Nitrosodimethylamine	EPA 625
	EPA 8270C
N-Nitrosodi-n-butylamine	EPA 8270C
N-Nitrosodi-n-propylamine	EPA 625
	EPA 8270C
N-Nitrosodiphenylamine	EPA 625
	EPA 8270C
N-nitrosopiperidine	EPA 8270C
N-Nitrosopyrrolidine	EPA 8270C

Nutrient

Ammonia (as N)	SM 18 4500-NH3 F or G
Kjeldahl Nitrogen, Total	EPA 351.2 Rev. 2.0
Nitrate (as N)	EPA 300.0 Rev. 2.1
	EPA 9056
	SM 18-21 4500-NO3 F (00)

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Nutrient

Nitrite (as N)	EPA 300.0 Rev. 2.1
	EPA 9056
	SM 18-21 4500-NO2 B (00)
	SM 18-21 4500-NO3 F (00)
Orthophosphate (as P)	SM 18-21 4500-P E
Phosphorus, Total	SM 18-21 4500-P F

Organophosphate Pesticides

Dimethoate	EPA 8270C
Disulfoton	EPA 8270C
Famphur	EPA 8270C
Parathion ethyl	EPA 8270C
Parathion methyl	EPA 8270C
Phorate	EPA 8270C

Phthalate Esters

Benzyl butyl phthalate	EPA 625
	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 625
	EPA 8270C
Diethyl phthalate	EPA 625
	EPA 8270C
Dimethyl phthalate	EPA 625
	EPA 8270C
Di-n-butyl phthalate	EPA 625
	EPA 8270C
Di-n-octyl phthalate	EPA 625
	EPA 8270C

Polychlorinated Biphenyls

PCB-1016	EPA 608
	EPA 8082
PCB-1221	EPA 608
	EPA 8082
PCB-1232	EPA 608
	EPA 8082
PCB-1242	EPA 608
	EPA 8082
PCB-1248	EPA 608
	EPA 8082
PCB-1254	EPA 608
	EPA 8082
PCB-1260	EPA 608
	EPA 8082

Polynuclear Aromatics

3-Methylcholanthrene	EPA 8270C
7,12-Dimethylbenzyl (a) anthracene	EPA 8270C
Acenaphthene	EPA 625
	EPA 8270C
Acenaphthylene	EPA 625
	EPA 8270C
Anthracene	EPA 625
	EPA 8270C
Benzo(a)anthracene	EPA 625
	EPA 8270C
Benzo(a)pyrene	EPA 625
	EPA 8270C
Benzo(b)fluoranthene	EPA 625

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Polynuclear Aromatics

Benzo(b)fluoranthene	EPA 8270C
Benzo(ghi)perylene	EPA 625
	EPA 8270C
Benzo(k)fluoranthene	EPA 625
	EPA 8270C
Chrysene	EPA 625
	EPA 8270C
Dibenzo(a,h)anthracene	EPA 625
	EPA 8270C
Fluoranthene	EPA 625
	EPA 8270C
Fluorene	EPA 625
	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 625
	EPA 8270C
Naphthalene	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270C
Phenanthrene	EPA 625
	EPA 8270C
Pyrene	EPA 625
	EPA 8270C

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270C
2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 625
	EPA 8270C

Priority Pollutant Phenols

2,4-Dichlorophenol	EPA 625
	EPA 8270C
2,4-Dimethylphenol	EPA 625
	EPA 8270C
2,4-Dinitrophenol	EPA 625
	EPA 8270C
2,6-Dichlorophenol	EPA 8270C
2-Chlorophenol	EPA 625
	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 625
	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 625
	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 625
	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 625
	EPA 8270C
Pentachlorophenol	EPA 625
	EPA 8270C
Phenol	EPA 625
	EPA 8270C

Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA 8260B
1,2-Dichlorobenzene	EPA 601
	EPA 602

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER
RICHARD F. DAINES, M.D.

Expires 12:01 AM April 01, 2010
Issued April 01, 2009

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JEFF GLASER
TRIMATRIX LABORATORIES, INC
5560 CORPORATE EXCHANGE CT SE
GRAND RAPIDS, MI 49512-5503

NY Lab Id No: 11776
EPA Lab Code: MI00005

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER All approved analytes are listed below:

Purgeable Aromatics

1,2-Dichlorobenzene	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270C
1,3,5-Trimethylbenzene	EPA 8260B
1,3-Dichlorobenzene	EPA 601
	EPA 602
	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270C
1,4-Dichlorobenzene	EPA 601
	EPA 602
	EPA 624
	EPA 625
	EPA 8021B
	EPA 8260B
	EPA 8270C
Benzene	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Chlorobenzene	EPA 601
	EPA 602
	EPA 624
	EPA 8021B

Purgeable Aromatics

Chlorobenzene	EPA 8260B
Ethyl benzene	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Isopropylbenzene	EPA 8260B
n-Butylbenzene	EPA 8021B
	EPA 8260B
n-Propylbenzene	EPA 8260B
p-Isopropyltoluene (P-Cymene)	EPA 8021B
	EPA 8260B
sec-Butylbenzene	EPA 8260B
Styrene	EPA 8021B
	EPA 8260B
tert-Butylbenzene	EPA 8021B
	EPA 8260B
Toluene	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B
Total Xylenes	EPA 602
	EPA 624
	EPA 8021B
	EPA 8260B

Purgeable Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8021B
	EPA 8260B
1,1,1-Trichloroethane	EPA 601

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Purgeable Halocarbons

1,1,1-Trichloroethane	EPA 624
	EPA 8021B
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
1,1,2-Trichloroethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
1,1,2-Trifluoro-1,2,2-Trichloroethane	EPA 8260B
1,1-Dichloroethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
1,1-Dichloroethene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
1,1-Dichloropropene	EPA 8021B
	EPA 8260B
1,2,3-Trichloropropane	EPA 8021B
	EPA 8260B
1,2-Dichloroethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B

Purgeable Halocarbons

1,2-Dichloropropane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
1,3-Dichloropropane	EPA 8021B
	EPA 8260B
2,2-Dichloropropane	EPA 8021B
	EPA 8260B
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260B
2-Chloroethylvinyl ether	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
3-Chloropropene (Allyl chloride)	EPA 8260B
Bromochloromethane	EPA 8021B
	EPA 8260B
Bromodichloromethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Bromoform	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Bromomethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B

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Purgeable Halocarbons

Carbon tetrachloride	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Chloroethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Chloroform	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Chloromethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
cis-1,2-Dichloroethene	EPA 8021B
	EPA 8260B
cis-1,3-Dichloropropene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Dibromochloromethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Dibromomethane	EPA 8021B
	EPA 8260B

Purgeable Halocarbons

Dichlorodifluoromethane	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Methylene chloride	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Tetrachloroethene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
trans-1,2-Dichloroethene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
trans-1,3-Dichloropropene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B
Trichloroethene	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B
Trichlorofluoromethane	EPA 601
	EPA 624
	EPA 8021B

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Purgeable Halocarbons

Trichlorofluoromethane	EPA 8260B
Vinyl chloride	EPA 601
	EPA 624
	EPA 8021B
	EPA 8260B

Purgeable Organics

1,4-Dioxane	EPA 8260B
2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Acetonitrile	EPA 8260B
Carbon Disulfide	EPA 8260B
Ethyl Acetate	EPA 8260B
Isobutyl alcohol	EPA 8015 B
	EPA 8260B
Methyl iodide	EPA 8260B
o-Toluidine	EPA 8270C
Vinyl acetate	EPA 8260B

Residue

Settleable Solids	SM 18-20 2540 F
Solids, Total	SM 18-20 2540B (97)
Solids, Total Dissolved	SM 18-21 2540C (97)
Solids, Total Suspended	SM 18-20 2540D (97)

Semi-Volatile Organics

2-Methylnaphthalene	EPA 8270C

Semi-Volatile Organics

4-Amino biphenyl	EPA 8270C
Acetophenone	EPA 8270C
Benzoic Acid	EPA 8270C
Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C
Ethyl methanesulfonate	EPA 8270C
Isosafrole	EPA 8270C
Methyl methanesulfonate	EPA 8270C
O,O,O-Triethyl phosphorothioate	EPA 8270C
p-Dimethylaminoazobenzene	EPA 8270C
Phenacetin	EPA 8270C
Safrole	EPA 8270C

Wastewater Metals I

Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Copper, Total	EPA 200.7 Rev. 4.4

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All approved analytes are listed below:

Wastewater Metals I

Copper, Total	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Iron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Lead, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Magnesium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Potassium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Sodium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Strontium, Total	EPA 6010B

Wastewater Metals II

Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Antimony, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Chromium VI	EPA 7196A
	SM 20 3500-Cr B (01)
Mercury, Total	EPA 1631E
	EPA 245.1 Rev. 3.0
	EPA 7470A
Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Zinc, Total	EPA 200.7 Rev. 4.4

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Wastewater Metals II

Zinc, Total	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020

Wastewater Metals III

Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Thallium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
	EPA 6010B
	EPA 6020
Tin, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Titanium, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Uranium (Mass)	EPA 200.8 Rev. 5.4
	EPA 6020

Wastewater Miscellaneous

Boron, Total	EPA 200.7 Rev. 4.4
	EPA 6010B
Color	SM 18-21 2120B (01)
Cyanide, Total	OIA-1677

Wastewater Miscellaneous

Cyanide, Total	SM 18-21 4500-CN E (99)
	SM 18-21 4500-CN G (99)
Hydrogen Ion (pH)	SM 18-21 4500-H B (00)
Oil & Grease Total Recoverable (HEM)	EPA 1664A
	EPA 9070 (Solvent:Hexane)
Organic Carbon, Total	SM 18-21 5310C (00)
Phenols	EPA 420.1 Rev. 1978
Silica, Dissolved	EPA 200.7 Rev. 4.4
	EPA 6010B
Specific Conductance	SM 18-21 2510B (97)
Sulfide (as S)	SM 18-20 4500-S D (00)
Surfactant (MBAS)	SM 18-21 5540C (00)
Total Recoverable Petroleum Hydrocarb	EPA 1664A
Turbidity	SM 18-21 2130 B (01)

Sample Preparation Methods

EPA 200.2
EPA 3010A
EPA 3020A
EPA 3510C
EPA 5030B
SM 18-20 4500-CN C

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260B
Acrylonitrile	EPA 8260B
Ethyl methacrylate	EPA 8260B
Methyl acrylonitrile	EPA 8260B
Methyl methacrylate	EPA 8260B

Amines

1,2-Diphenylhydrazine	EPA 8270C
1,4-Phenylenediamine	EPA 8270C
1-Naphthylamine	EPA 8270C
2-Naphthylamine	EPA 8270C
2-Nitroaniline	EPA 8270C
3-Nitroaniline	EPA 8270C
4-Chloroaniline	EPA 8270C
4-Nitroaniline	EPA 8270C
5-Nitro-o-toluidine	EPA 8270C
Aniline	EPA 8270C
Carbazole	EPA 8270C
Diphenylamine	EPA 8270C
Methapyrilene	EPA 8270C
Pronamide	EPA 8270C

Benzidines

3,3'-Dichlorobenzidine	EPA 8270C
3,3'-Dimethylbenzidine	EPA 8270C
Benzidine	EPA 8270C

Characteristic Testing

Ignitability	EPA 1020

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081A
4,4'-DDE	EPA 8081A
4,4'-DDT	EPA 8081A
Aldrin	EPA 8081A
alpha-BHC	EPA 8081A
alpha-Chlordane	EPA 8081A
beta-BHC	EPA 8081A
Chlordane Total	EPA 8081A
Chlorobenzilate	EPA 8081A
	EPA 8270C
delta-BHC	EPA 8081A
Diallate	EPA 8081A
	EPA 8270C
Dieldrin	EPA 8081A
Endosulfan I	EPA 8081A
Endosulfan II	EPA 8081A
Endosulfan sulfate	EPA 8081A
Endrin	EPA 8081A
Endrin aldehyde	EPA 8081A
Endrin Ketone	EPA 8081A
gamma-Chlordane	EPA 8081A
Heptachlor	EPA 8081A
Heptachlor epoxide	EPA 8081A
Kepone	EPA 8081A
	EPA 8270C
Lindane	EPA 8081A
Methoxychlor	EPA 8081A
Pentachloronitrobenzene	EPA 8270C

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Chlorinated Hydrocarbon Pesticides

Toxaphene	EPA 8081A

Chlorinated Hydrocarbons

1,2,4,5-Tetrachlorobenzene	EPA 8270C
1,2,4-Trichlorobenzene	EPA 8021B
	EPA 8260B
	EPA 8270C
2-Chloronaphthalene	EPA 8270C
Hexachlorobenzene	EPA 8270C
Hexachlorobutadiene	EPA 8260B
	EPA 8270C
Hexachlorocyclopentadiene	EPA 8270C
Hexachloroethane	EPA 8270C
Hexachloropropene	EPA 8270C
Pentachlorobenzene	EPA 8270C

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A

Haloethers

4-Bromophenylphenyl ether	EPA 8270C
4-Chlorophenylphenyl ether	EPA 8270C
Bis (2-chloroisopropyl) ether	EPA 8270C
Bis(2-chloroethoxy)methane	EPA 8270C
Bis(2-chloroethyl)ether	EPA 8270C

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene	EPA 8310
Acenaphthylene	EPA 8310
Anthracene	EPA 8310
Benzo(a)anthracene	EPA 8310
Benzo(a)pyrene	EPA 8310
Benzo(b)fluoranthene	EPA 8310
Benzo(g,h,i)perylene	EPA 8310
Benzo(k)fluoroanthene	EPA 8310
Chrysene	EPA 8310
Dibenzo(a,h)anthracene	EPA 8310
Fluoranthene	EPA 8310
Fluorene	EPA 8310
Indeno(1,2,3-cd)pyrene	EPA 8310
Naphthalene	EPA 8310
Phenanthrene	EPA 8310
Pyrene	EPA 8310

Metals I

Barium, Total	EPA 6010B
	EPA 6020
Cadmium, Total	EPA 6010B
	EPA 6020

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Metals I

Calcium, Total	EPA 6010B
Chromium, Total	EPA 6010B
	EPA 6020
Copper, Total	EPA 6010B
	EPA 6020
Iron, Total	EPA 6010B
Lead, Total	EPA 6010B
	EPA 6020
Magnesium, Total	EPA 6010B
Manganese, Total	EPA 6010B
	EPA 6020
Nickel, Total	EPA 6010B
	EPA 6020
Potassium, Total	EPA 6010B
Silver, Total	EPA 6010B
	EPA 6020
Sodium, Total	EPA 6010B
Strontium, Total	EPA 6010B

Metals II

Aluminum, Total	EPA 6010B
Antimony, Total	EPA 6010B
	EPA 6020
Arsenic, Total	EPA 6010B
	EPA 6020
Beryllium, Total	EPA 6010B
	EPA 6020
Chromium VI	EPA 7196A
Lithium, Total	EPA 6010B

Metals II

Mercury, Total	EPA 7471A
Selenium, Total	EPA 6010B
	EPA 6020
Vanadium, Total	EPA 6010B
	EPA 6020
Zinc, Total	EPA 6010B
	EPA 6020

Metals III

Cobalt, Total	EPA 6010B
	EPA 6020
Molybdenum, Total	EPA 6010B
	EPA 6020
Silica, Dissolved	EPA 6010B
Thallium, Total	EPA 6010B
	EPA 6020
Tin, Total	EPA 6010B
Titanium, Total	EPA 6010B

Minerals

Chloride	EPA 9056
Fluoride, Total	EPA 9056
Sulfate (as SO4)	EPA 9038
	EPA 9056

Miscellaneous

Boron, Total	EPA 6010B
Cyanide, Total	EPA 9012A
	EPA 9014

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WADSWORTH CENTER
RICHARD F. DAINES, M.D.

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Issued April 01, 2009

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JEFF GLASER
TRIMATRIX LABORATORIES, INC
5560 CORPORATE EXCHANGE CT SE
GRAND RAPIDS, MI 49512-5503

NY Lab Id No: 11776
EPA Lab Code: MI00005

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Miscellaneous

Hydrogen Ion (pH)	EPA 9040B
	EPA 9045C
Phenols	EPA 9065
Sulfide (as S)	EPA 9034
Total Organic Halides	EPA 9020

Nitroaromatics and Isophorone

1,3,5-Trinitrobenzene	EPA 8330
1,3-Dinitrobenzene	EPA 8330
1,4-Naphthoquinone	EPA 8270C
2,4,6-Trinitrotoluene	EPA 8330
2,4-Dinitrotoluene	EPA 8270C
	EPA 8330
2,6-Dinitrotoluene	EPA 8270C
	EPA 8330
2-Amino-4,6-dinitrotoluene	EPA 8330
2-Nitrotoluene	EPA 8330
3-Nitrotoluene	EPA 8330
4-Amino-2,6-dinitrotoluene	EPA 8330
4-Dimethylaminoazobenzene	EPA 8270C
4-Nitrotoluene	EPA 8330
Hexahydro-1,3,5-trinitro-1,3,5-triazine	EPA 8330
Isophorone	EPA 8270C
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330
Nitrobenzene	EPA 8270C
	EPA 8330
Nitroquinoline-1-oxide	EPA 8270C
Octahydro-tetranitro-tetrazocine	EPA 8330
Pyridine	EPA 8270C

Nitrosoamines

N-Nitrosodiethylamine	EPA 8270C
N-Nitrosodimethylamine	EPA 8270C
N-Nitrosodi-n-butylamine	EPA 8270C
N-Nitrosodi-n-propylamine	EPA 8270C
N-Nitrosodiphenylamine	EPA 8270C
N-nitrosomethylethylamine	EPA 8270C
N-nitrosomorpholine	EPA 8270C
N-nitrosopiperidine	EPA 8270C
N-Nitrosopyrrolidine	EPA 8270C

Nutrients

Nitrate (as N)	EPA 9056
Nitrite (as N)	EPA 9056

Organophosphate Pesticides

Dimethoate	EPA 8270C
Famphur	EPA 8270C
Parathion ethyl	EPA 8270C
Parathion methyl	EPA 8270C
Phorate	EPA 8270C
Sulfotepp	EPA 8270C
TEPP	EPA 8270C
Thionazin	EPA 8270C

Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015 B
Gasoline Range Organics	EPA 8015 B
Oil & Grease Total Recoverable (HEM)	EPA 9071 (Solvent:Hexane)

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Phthalate Esters

Benzyl butyl phthalate	EPA 8270C
Bis(2-ethylhexyl) phthalate	EPA 8270C
Diethyl phthalate	EPA 8270C
Dimethyl phthalate	EPA 8270C
Di-n-butyl phthalate	EPA 8270C
Di-n-octyl phthalate	EPA 8270C

Polychlorinated Biphenyls

PCB-1016	EPA 8082
PCB-1221	EPA 8082
PCB-1232	EPA 8082
PCB-1242	EPA 8082
PCB-1248	EPA 8082
PCB-1254	EPA 8082
PCB-1260	EPA 8082

Polynuclear Aromatic Hydrocarbons

3-Methylcholanthrene	EPA 8270C
7,12-Dimethylbenzyl (a) anthracene	EPA 8270C
Acenaphthene	EPA 8270C
Acenaphthylene	EPA 8270C
Anthracene	EPA 8270C
Benzo(a)anthracene	EPA 8270C
Benzo(a)pyrene	EPA 8270C
Benzo(b)fluoranthene	EPA 8270C
Benzo(ghi)perylene	EPA 8270C
Benzo(k)fluoranthene	EPA 8270C
Chrysene	EPA 8270C
Dibenzo(a,h)anthracene	EPA 8270C

Polynuclear Aromatic Hydrocarbons

Fluoranthene	EPA 8270C
Fluorene	EPA 8270C
Indeno(1,2,3-cd)pyrene	EPA 8270C
Naphthalene	EPA 8260B
	EPA 8270C
Phenanthrene	EPA 8270C
Pyrene	EPA 8270C

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270C
2,4,5-Trichlorophenol	EPA 8270C
2,4,6-Trichlorophenol	EPA 8270C
2,4-Dichlorophenol	EPA 8270C
2,4-Dimethylphenol	EPA 8270C
2,4-Dinitrophenol	EPA 8270C
2,6-Dichlorophenol	EPA 8270C
2-Chlorophenol	EPA 8270C
2-Methyl-4,6-dinitrophenol	EPA 8270C
2-Methylphenol	EPA 8270C
2-Nitrophenol	EPA 8270C
3-Methylphenol	EPA 8270C
4-Chloro-3-methylphenol	EPA 8270C
4-Methylphenol	EPA 8270C
4-Nitrophenol	EPA 8270C
Pentachlorophenol	EPA 8270C
Phenol	EPA 8270C

Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA 8021B

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Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA 8260B
1,2-Dichlorobenzene	EPA 8021B
	EPA 8260B
	EPA 8270C
1,3,5-Trimethylbenzene	EPA 8021B
	EPA 8260B
1,3-Dichlorobenzene	EPA 8021B
	EPA 8260B
	EPA 8270C
1,4-Dichlorobenzene	EPA 8021B
	EPA 8260B
	EPA 8270C
2-Chlorotoluene	EPA 8260B
4-Chlorotoluene	EPA 8260B
Benzene	EPA 8021B
	EPA 8260B
Bromobenzene	EPA 8021B
	EPA 8260B
Chlorobenzene	EPA 8021B
	EPA 8260B
Ethyl benzene	EPA 8021B
	EPA 8260B
Isopropylbenzene	EPA 8260B
n-Butylbenzene	EPA 8021B
	EPA 8260B
n-Propylbenzene	EPA 8260B
p-Isopropyltoluene (P-Cymene)	EPA 8260B
sec-Butylbenzene	EPA 8021B

Purgeable Aromatics

sec-Butylbenzene	EPA 8260B
Styrene	EPA 8021B
	EPA 8260B
tert-Butylbenzene	EPA 8021B
	EPA 8260B
Toluene	EPA 8021B
	EPA 8260B
Total Xylenes	EPA 8021B
	EPA 8260B

Purgeable Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8021B
	EPA 8260B
1,1,1-Trichloroethane	EPA 8021B
	EPA 8260B
1,1,2,2-Tetrachloroethane	EPA 8021B
	EPA 8260B
1,1,2-Trichloroethane	EPA 8021B
	EPA 8260B
1,1,2-Trifluoro-1,2,2-Trichloroethane	EPA 8260B
1,1-Dichloroethane	EPA 8021B
	EPA 8260B
1,1-Dichloroethene	EPA 8021B
	EPA 8260B
1,1-Dichloropropene	EPA 8260B
1,2,3-Trichloropropane	EPA 8260B
1,2-Dibromo-3-chloropropane	EPA 8260B
1,2-Dibromoethane	EPA 8260B
1,2-Dichloroethane	EPA 8021B

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Purgeable Halocarbons

1,2-Dichloroethane	EPA 8260B
1,2-Dichloropropane	EPA 8021B
	EPA 8260B
1,3-Dichloropropane	EPA 8260B
2,2-Dichloropropane	EPA 8260B
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260B
2-Chloroethylvinyl ether	EPA 8021B
	EPA 8260B
3-Chloropropene (Allyl chloride)	EPA 8260B
Bromochloromethane	EPA 8260B
Bromodichloromethane	EPA 8021B
	EPA 8260B
Bromoform	EPA 8021B
	EPA 8260B
Bromomethane	EPA 8021B
	EPA 8260B
Carbon tetrachloride	EPA 8021B
	EPA 8260B
Chloroethane	EPA 8021B
	EPA 8260B
Chloroform	EPA 8021B
	EPA 8260B
Chloromethane	EPA 8021B
	EPA 8260B
cis-1,2-Dichloroethene	EPA 8021B
	EPA 8260B
cis-1,3-Dichloropropene	EPA 8021B
	EPA 8260B

Purgeable Halocarbons

Dibromochloromethane	EPA 8021B
	EPA 8260B
Dibromomethane	EPA 8021B
	EPA 8260B
Dichlorodifluoromethane	EPA 8021B
	EPA 8260B
Methylene chloride	EPA 8021B
	EPA 8260B
Tetrachloroethene	EPA 8021B
	EPA 8260B
trans-1,2-Dichloroethene	EPA 8021B
	EPA 8260B
trans-1,3-Dichloropropene	EPA 8021B
	EPA 8260B
trans-1,4-Dichloro-2-butene	EPA 8260B
Trichloroethene	EPA 8021B
	EPA 8260B
Trichlorofluoromethane	EPA 8021B
	EPA 8260B
Vinyl chloride	EPA 8021B
	EPA 8260B

Purgeable Organics

1,4-Dioxane	EPA 8260B
2-Butanone (Methylethyl ketone)	EPA 8260B
2-Hexanone	EPA 8260B
4-Methyl-2-Pentanone	EPA 8260B
Acetone	EPA 8260B
Acetonitrile	EPA 8260B

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Purgeable Organics

Carbon Disulfide	EPA 8260B
Ethyl Acetate	EPA 8260B
Isobutyl alcohol	EPA 8015 B
	EPA 8260B
Methyl tert-butyl ether	EPA 8021B
	EPA 8260B
o-Toluidine	EPA 8270C
Propionitrile	EPA 8260B
tert-Butyl alcohol	EPA 8260B
Vinyl acetate	EPA 8260B

Semi-Volatile Organics

2-Methylnaphthalene	EPA 8270C
4-Amino biphenyl	EPA 8270C
Acetophenone	EPA 8270C
Aramite	EPA 8270C
Benzoic Acid	EPA 8270C
Benzyl alcohol	EPA 8270C
Dibenzofuran	EPA 8270C
Ethyl methanesulfonate	EPA 8270C
Isosafrole	EPA 8270C
Methyl methanesulfonate	EPA 8270C
O,O,O-Triethyl phosphorothioate	EPA 8270C
Phenacetin	EPA 8270C
Safrole	EPA 8270C

Sample Preparation Methods

	EPA 1311
	EPA 3010A

Sample Preparation Methods

	EPA 3020A
	EPA 3050B
	EPA 3545
	EPA 3550B
	EPA 3580
	EPA 3585
	EPA 5030B
	EPA 5035
	EPA 9030B

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TriMatrix Laboratories, Inc.

5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record

COC No.

Page ____ of ____

For Lab Use Only

Cart

VOA Rack/Tray

Receipt Log No.

Project Chemist

Laboratory Project No.

Client Name

Address

Phone

Fax

Project Name

Client Project No. / P.O. No.

Invoice To Client

Other (comments)

Contact/Report To

Analyses Requested

Container Type (corresponds to Container Packing List)

Number of Containers Submitted

Test Group	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	COMP	GRAB	Matrix											Total	Sample Comments
			1																			
			2																			
			3																			
			4																			
			5																			
			6																			
			7																			
			8																			
			9																			
			10																			

^ PRESERVATIVES

A NONE pH~7
B HNO_3 pH<2
C H_2SO_4 pH<2
D 1+1 HCl pH<2
E NaOH pH>12
F ZnAc/NaOH pH>9
G MeOH
H Other (note below)

Sampled By (print)

Sampler's Signature

Company

How Shipped? Hand Carrier_____

Tracking No.

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 For Lab By Date Time

APPENDIX C

DEXSIL L2000DX USER'S MANUAL AND SOIL EXTRACTION PROCEDURE

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http://www.dexsil.com

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Introduction to the L2000DX Analysis System

Congratulations on your purchase of the L2000DX Analyzer, a versatile analysis system suitable for the analysis of a wide variety of chlorinated organic compounds in a variety of matrices. The basic principal of the L2000DX system is to measure the total organic chlorine content of a sample and equate that to an equivalent concentration of the target or expected analyte. If all of the organic chlorine present is assumed to be derived from the target analyte, then an upper limit is established for the compound in question. (If other organic chlorines are present, in addition to the target analyte, they will be counted as the target analyte.) To accomplish this, all of the organically bound chlorine must be converted into inorganic chloride and the resulting chloride quantified. Once the total chlorine content of the sample is known, a conversion factor is used to convert the chloride concentration into an equivalent concentration of the target analyte. If the contaminant is known, the resulting concentration estimates will accurately correlate with the actual concentration of the analyte in the sample as determined by GC analysis in the lab. If the contaminant is unknown, a conservative or worst case conversion factor is chosen to provide an upper limit for the concentration of the target analyte in the sample.

There are three basic steps involved in the chemical analysis for total halogen by the L2000DX:

- Sample Preparation
- Conversion to Inorganic Chloride
- Quantification

The sample preparation step determines the type of chlorine detected, i.e., organic, inorganic, or total, and is matrix dependent. The sample preparation can be as simple as collecting a transformer oil sample, or can involve the extraction of a soil or water sample. In the case of a wipe sample, the surface is wiped and the wipe-gauze is extracted. The steps involved in the conversion to inorganic chloride reaction and the chloride quantification are the same for all matrices. The steps in the conversion to inorganic chloride involves the reaction of the sample with metallic sodium and the extraction of the resulting chloride into an aqueous buffer system. A chloride specific electrode is used to quantify the extracted chloride. Using stored conversion factors, the chloride value is then converted to an equivalent concentration of analyt

Sample Preparation

The L2000DX can be used to analyze the following matrices:

- Transformer Oil
- Soil
- Water
- Surface Wipe

Each matrix requires different preparation prior to the conversion reaction step and subsequent quantification. Each of the matrix preparation steps are described in detail under **Sample Preparation.**

The routine analysis of transformer oil requires no sample preparation other than to collect a clean sample without introducing any extraneous sources of chloride into the sample, such as perspiration or road salt. This is important because, for transformer oil, there is no sample cleanup procedures to remove inorganic chloride contamination. Once collected the sample is reacted and the resulting chloride is extracted and quantified.

The chlorine quantified in this case is the total chlorine contained in the sample. Transformer oil is typically free of inorganic chlorine eliminating the need for any sample cleanup procedure. In special cases where transformers have failed due to water contamination or have been removed for service and stored in areas near seawater or road salt, inorganic chloride may cause an elevated reading.

Before a soil sample can be analyzed, the organic contaminates must be extracted using an organic solvent. Because soil samples invariably contain inorganic chlorine, the soil extract is cleaned up to remove all traces of the inorganic chloride. The cleaned extract is then reacted and the resulting chloride is quantified. For soil analyses, therefore, only the organic chlorine content is ever quantified. Extraneous sources of chloride contamination such as road salt or sea salt are **not** detected.

As with soil samples, a water sample must also be extracted prior to final analysis. The ratio of the solvent volume to the sample volume determines the sensitivity of the test. The extract is reacted and the chloride is quantified as above. As with soil samples, only the organic chlorine is quantified in water samples.

Wipe tests require that a specific area be wiped using a hexane-soaked gauze. The gauze is extracted with an organic solvent, reacted, and the chlorine content determined. For wipe samples, the standard procedure eliminates most all inorganic chlorine contamination. Areas with very high surface concentration of salts may need to be prepared differently.

Chloride Conversion Reaction

Once the sample has been prepared, the remaining chloride conversion steps are the same for all sample types. The conversion step is a reaction of the sample with an excess of metallic sodium in the presence of a catalyst to convert the covalently bound organic chlorine into free chloride ions. This reaction of metallic sodium with organo-chlorine compounds is vigorous and goes to completion converting all of the organic chlorine to chloride.

Quantification

Upon the completion of the conversion reaction, the resulting chloride ions are extracted into an aqueous buffer. The chloride content in the final extract/buffer is then quantified using a chloride specific electrode and converted to an equivalent analyte concentration using the conversion factors programed into the

analysis method of the instrument. The conversion factor is made up of the percent chlorine, the sample size multiplier, and the extraction efficiency multiplier. The analyte concentration is determined by first subtracting the blank from the raw chloride reading (except when the Blank Subtraction has been turned off), and the resulting corrected chloride value is multiplied by the size and extraction multipliers and divided by the chloride fraction (percent chlorine divided by 100):

$$[\text{Analyte}] = ([Cl^-]_{raw} - [Cl^-]_{blank})(\text{size multiplier})(\text{extraction multiplier})/(\text{chlorine fraction})$$

The L2000DX analyzer comes preprogramed with 28 methods providing for analysis of a large number of chloride compounds in most matrices. Correction factors are preprogramed to account for all of the steps necessary to run samples using the standard prepackaged reagents. If the procedures are modified or other analytes are expected, up to 22 custom methods can easily be built.

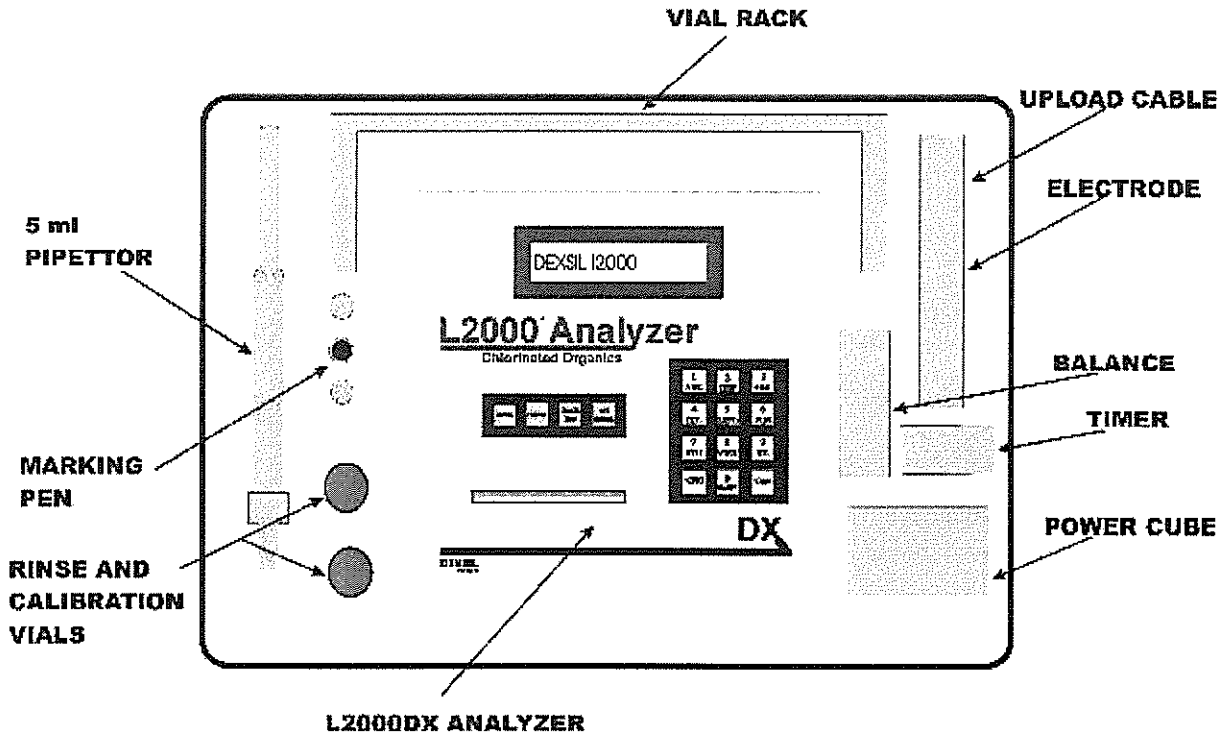
The operational program of the L2000DX has been designed to provide the most versatile data reduction platform possible for the analysis of chlorinated organic compounds in a variety of matrices. At the same time, the program is easy to use for routine analysis and does not require custom method development. The operating program is divided into an **Analysis Loop** and the **OPTIONS Menu**. Preprogramed methods are available for use in routine analyses and are easily selected from within the **Analysis Loop**. The software automatically cycles back to critical points in the analysis loop to prompt the user for **"Calibration"**, **"Blank Subtraction"** and **"Sample ID"** inputs. Measurements can be initiated without requiring any additional programing by simply choosing the **Method** and following the on-screen instructions,. Custom methods can be constructed by selecting the **OPTIONS Menu** and modifying existing methods or creating a new method (see **Creating and Editing a Method**).

Unpacking

The L2000DX is shipped in its own carrying case, complete with all the hardware necessary for operation. Upon receiving the L2000DX Analyzer, please verify that all of the items listed below are present and in good working order.

The carrying case for the L2000DX Analyzer should contain the following items:

L2000DX Electronic PCB/Chloride Analyzer
Power cube AC-DC transformer
PC Upload Cable
Portable electronic balance & 100 gm Calibration weight
5 mL pipettor
Vial rack
Timer
Marking pen
Two empty 20 mL glass vials for RINSE and CALIBRATION solutions
L2000DX Data Manager Software CD
Instruction Manual, Certificate of Calibration and MSDS



In addition to the aforementioned items in the carrying case, the following should be included in the same outer shipping box:

Chloride-ion specific electrode[1]
Packet of polishing strips[1]
Test tube rack

To prevent damage to the instrument in the case of a leak, the sample preparation reagents are shipped separately. The exact makeup of the reagent pack will depend on the specific reagent option chosen at the time of ordering. The options available for the L2000DX and the components contained in each are the following:

Option 1: Reagents for 40 Oil Tests

1 - 250 mL bottle of EXTRACT solution
1 - 250 mL bottle of RINSE solution
1 - 250 mL bottle of CALIBRATION solution
1 - 60 mL bottle of Electrode Filling Solution
1 - tray of 40 empty 20 mL glass vials
1 - box of tissue wipes
1 - **shelf-pack** containing:

[1] These items are shipped in their original packaging from Orion. Once they have been unpacked, they should be carried in the above-labeled slot in the carrying case.

40 - filters
40 - pipettes
40 - reaction tubes (black dispenser caps)

Reorder Part No: (LP-ORK)

Option 2: Reagents for 20 Soil Tests (Standard Procedure)

1 - 250 mL bottle of EXTRACT solution
1 - 250 mL bottle of RINSE solution
1 - 250 mL bottle of CALIBRATION solution
1 - 60 mL bottle of Electrode Filling Solution
1 - box of tissue wipes
1 - **40-cell box** containing:
    20 - empty 20 mL glass vials
    20 - bottles containing Soil Extraction Solvent
1 - **shelf-pack** containing:
    20 - filters
    20 - pipettes
    20 - reaction tubes (black dispenser caps)
    20 - metal scoops for obtaining soil samples
    20 - 10 mL plastic syringes
    20 - drying columns (foil packed)
    20 - empty test tubes with white caps

Reorder Part No: (LP-SRK)

Option 2a: Reagents for 20 Soil Tests (Two-Step Extraction Procedure)

1 - 250 mL bottle of EXTRACT solution
1 - 250 mL bottle of RINSE solution
1 - 250 mL bottle of CALIBRATION solution
1 - 60 mL bottle of Electrode Filling Solution
1 - box of tissue wipes
1 - set of instructions
1 - **40-cell box** containing:
    20 - empty 20 mL glass vials
    20 - 6 mL black-capped water vials
1 - **shelf-pack** containing (in a heat sealed foil bag):
    20 - empty 25 mL soil tubes
    20 - filter funnels
    20 - pipettes
    20 - reaction tubes with black dispenser caps
    20 - break-top vials containing Soil Extraction Solvent
    20 - metal scoops for obtaining soil samples
    20 - syringe filters (in a heat sealed foil bag)
1 - set of instructions

Reorder Part No: (LP-SR2)

Option 3: Reagents for 20 Water Tests

1 - 250 mL bottle of EXTRACT solution
1 - 250 mL bottle of RINSE solution
1 - 250 mL bottle of CALIBRATION solution
1 - 60 mL bottle of Electrode Filling Solution
1 - box of laboratory wipes
1 - **40-cell box** containing:
    20 - empty 20 mL glass vials
    20 - bottles of Isooctane
1 - **shelf-pack** containing:
    20 - filters
    20 - pipettes
    20 - reaction tubes (black dispenser caps)
    20 empty test tubes with white caps
1 - set of instructions

Reorder Part No: (LP-WTR)

Option 4: Reagents for 20 Wipe Tests

1 - 250 mL bottle of EXTRACT solution
1 - 250 mL bottle of RINSE solution
1 - 250 mL bottle of CALIBRATION solution
1 - 60 mL bottle of Electrode Filling Solution
1 - box of tissue wipes
2 - pair of safety goggles (1 pair/10 tests)
2 - pair of neoprene gloves (1 pair/10 tests)
1 - **shelf-pack** containing:
    20 - filters
    20 - pipettes
    20 - reaction tubes (black dispenser caps)
    20 - hexane ampules
    20 - empty test tubes with white dispensing caps
1 - **box** containing:
    20 - gauze pads in 20 mL glass vials
    20 - 10 mL glass bottles containing Extraction Solvent (isooctane)
1 - **box** containing:
    20 - empty 20 mL glass vials
    20 - forceps
1 - set of instructions

Reorder Part No: (LP-WIP)

NOTE: DO NOT STORE REAGENTS IN THE INSTRUMENT CARRYING CASE, AS ANY LEAKS WILL DAMAGE THE INSTRUMENT.

Initial Set Up

Once the L2000DX has been unpacked, the components checked and the contents of the reagent pack verified, the L2000DX can be set up for analysis. Locate the vial rack, the two empty 20 mL vials and the bottles of RINSE and CALIBRATION solutions. Remove the caps from both vials and label one 20 mL vial "RINSE" and the other "CAL". Fill each vial approximately ½ full with the appropriate solution and set in vial rack. (NOTE: It is helpful to label the rack with the location of the RINSE and CAL vials.) These solutions will be used for the initial setup and calibration of the electrode. They will also be used periodically to re-calibrate the electrode and check the electrode output. Once set up, the electrode should remain in the RINSE solution when not in use.

Prior to running the Analyzer on battery power, it should be charged overnight using the power cube supplied with the instrument. The Analyzer can be operated from line power while the battery is charging and should be left plugged in whenever AC power is available. Turn on the Analyzer by pressing and holding the <ENTER/ON> key until the printer advances one line and the display reads:

```
Dexsil L2000DX
Version 1.24
```

This screen is the **First Screen** in the operating program. The Analyzer is ready to accept instructions.

Restoring the Electrode After Extended Storage

The electrode is shipped empty and should be stored empty whenever it will be left for an extended period of time (see **Electrode Care and Maintenance**). To restore the electrode to operating condition:

1. Remove protective cap from the tip of the electrode.

2. Fill the electrode, up to the filling hole in the side, with the Orion Electrode Filling Solution supplied with each lot of reagents. To fill, place the nozzle of the fill solution bottle into the hole on the side of the plastic body and gently squeeze the fill bottle.

3. Drain the electrode, while holding it upright over the waste beaker, by grasping the body of the electrode firmly in one hand and pushing down on the black cap where the cord enters the electrode. The filling solution will drain out of the bottom of the electrode.

4. Refill the electrode and make sure that the fill solution is making contact between the black cone and the plastic shell at the bottom of the electrode. If it is not making contact at all points, drain the electrode again and refill.

5. Connect the electrode to the BNC connector labeled "ELECTRODE" on the back of the L2000DX Analyzer and check electrode output.

6. To check the electrode, turn the Analyzer on by pressing and holding the <ENTER/ON> key

until the printer advances one line and the display screen activates. Select the **OPTIONS Menu** from the **First Screen** by the pressing <OPTION> key. At the **OPTIONS Menu**, go to "DIAGNOSTICS" by pressing the <6> key. This display:

```
DIAGNOSTICS X.X
XXX.X mV XX.X °C
```

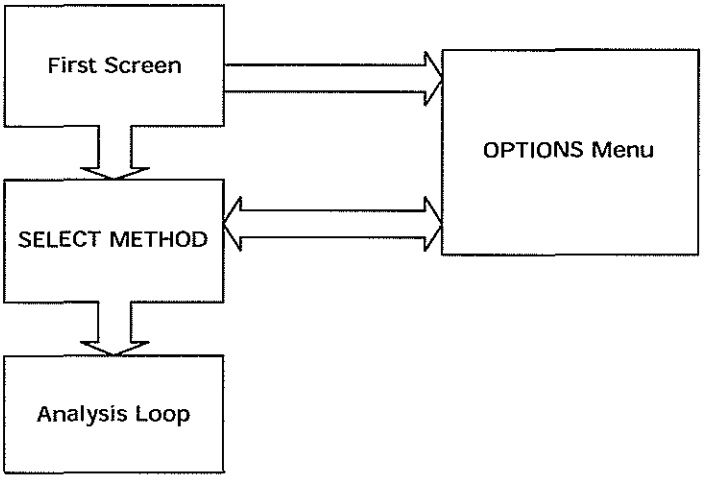
will indicate the current temperature and then continuously update the electrode output in millivolts (mV). Place the electrode in fresh RINSE solution, gently swirl the solution with the electrode and allow to sit. The electrode output should reach 140 mV or greater within 1 minute. If the electrode output does not reach at least 140 mV, empty the RINSE solution and refill with fresh RINSE solution. If this does not improve the electrode output, drain the electrode, refill it with filling solution and check the output again. Once the output is greater than 140 mV, the electrode is functioning correctly and it is safe to proceed with measurements. If the electrode output does not reach 140 mV, see under **Trouble Shooting** for remedies. NOTE: The update rate for the "**Diagnostics**" display is 6 seconds, any keys pressed will not register until the end of the current measurement cycle.

Basic Operation of the L2000DX

General Information

The L2000DX meter is equipped with an internal gel cell battery and can be operated with or without the wall adaptor. A fully charged battery should be sufficient to operate for 3 full 8-hour days of sample analyses, printing each data point. To conserve battery power, the printer can be turned off and the stored data printed or uploaded at a later time. To preserve the battery, the unit should be plugged into an AC outlet whenever possible.

The operation of the L2000DX is controlled by a program organized into a series of "screens". A screen prompts the user for appropriate input or displays output from the processor. There are two screens at the beginning of the program that will be referred to throughout the text. They are, the **First Screen** which appears when the analyzer is turned on and is returned to when turning the meter off manually, and the **SELECT METHOD Screen** which is the next screen in sequence. From the **SELECT METHOD Screen** the desired analytical method is chosen prior to analysis. From this screen the program enters a series of screens that step the user through the analysis of prepared samples. This series includes the calibration steps and blank determination and will loop back for periodic re-calibration as necessary. This section of the program is referred to the **Analysis Loop**. (See below for a block diagram of the program..)



Once in the **Analysis Loop**, the analyzer must be calibrated. The calibration procedure is a single point calibration using a 50 ppm chloride CALIBRATION standard supplied with each batch of reagents. This calibration must be repeated approximately every 20 measurements or one hour, whichever comes first. A timer, a counter, and a thermistor have been built into the instrument to prompt the user for recalibration when necessary. During the calibration procedure, the function of the electrode will also be evaluated. If the electrode potential for the calibration solution is not within the acceptable limits, a warning will be displayed and the program will jump to the diagnostics mode. (See below under **Diagnostics**.)

All other functions of the program are accessed by pressing the <OPTION> key from the **First Screen** or the **SELECT METHOD Screen**. This section of the program is referred to in the manual as the **OPTIONS Menu**. Although only one choice can be displayed at a time, options are organized into a sequence of choices, functioning much the same as a menu typical of most computer programs.

Turning the Analyzer On and Navigating the Operating Program

Pressing and holding the <ENTER/ON> key until the printer advances one line and the display activates will turn the Analyzer on and the program will be in the **First Screen**. In the **First Screen** the display will read:

```
Dexsil L2000DX
 Version X.XX
```

indicating the current software version running on the L2000DX. The version number should match the version number indicated on the cover of this manual.

From the **First Screen** pressing the <ENTER/ON> key will move the program to the **SELECT METHOD Screen**. In the **SELECT METHOD Screen**:

```
SELECT METHOD
method id
```

pressing the <<NO> and <YES>> keys will scroll through the stored methods. When the desired method is displayed, pressing the <ENTER/ON> key will select it. The program will then enter the **Analysis Loop** and the program will begin prompting one through the operations necessary to begin the analysis of samples using the chosen method.

Calibration

The first step required before any measurements can be made is to calibrate the instrument. The L2000DX operating program will jump to the **CALIBRATION** screen when a new method is selected, when the temperature has changed by more than 5 • C, or when the recalibration timer is triggered. The display will read:

```
CALIBRATION FOR
method name
```

indicating the method that is currently selected. Press the <ENTER/ON> or <YES>> keys and the display will indicate that the electrode should be placed in clean fresh CALIBRATION solution:

```
IS CALIBRATION
SOLUTION READY
```

Remove the electrode from the RINSE solution, gently wipe the body of the electrode with a clean wipe and insert the tip into the CALIBRATION solution. NOTE: DO NOT WIPE THE TIP OF THE ELECTRODE AS THIS MAY DAMAGE IT. Swirl the electrode gently a few times and press the <ENTER/ON> or <YES>> key. Allow the electrode to remain in the calibration solution, the meter will briefly display the message:

```
MEASUREMENT IN
PROGRESS
```

Provided the electrode output is within the acceptable range and the ambient temperature is also within the acceptable range, the calibration procedure is complete when the program display briefly:

```
CAL TEMP = XX°C
mV = xx
```

The calibration results will also be sent to the printer, if the printer has been turned on.

Calibration:	*hh:mm mm/dd/yyyy*
Version:	*xxxx*
Method:	*METHOD NAME*
mV = *xx.x*	Temp. = *xx.x* °C
A = *xxx.x*	B = *xx.xxx*
Offset = *x.x*	

The program function will then move to the next step in the chosen method. If the temperature is outside the acceptable range the following error message will be displayed:

```
Temperature
Error xx.x °C
```

A temperature error indicates that the ambient conditions are not suitable for measurement. Save all extracts and move to a location where the temperature is within the acceptable range.

If the electrode output is outside the acceptable range, an error will be displayed:

```
CAL ERROR
mV = xx
```

Pressing the <ENTER/ON> key will move the program back to the beginning of the **Analysis Loop**. (See "CAL ERROR" in **Error Messages** under **Trouble Shooting**)

Blank Determination

All of the pre-programed methods have the option of subtracting a reagent blank built into the method. If blank subtraction has been enabled in the chosen method, this option will be presented after each calibration. The option will be presented as:

```
USE BLANK
YES/NO
```

If NO is chosen, the program moves on to the next step in the method as described below in **Analysis**. If YES is chosen, the option will be presented:

```
USE PREVIOUS
BLANK YES/NO
```

If YES is chosen, an opportunity will be presented to change the stored blank or to enter a fixed value at:

```
USING BLANK OF
0.00
```

This number, in ppm chloride, can be edited or accepted, as is, by pressing the <ENTER/ON> key. The program then continues to the next step as described below under **Analysis**.

If NO is chosen, the screen will prompt for the blank solution:

```
PUT PROBE INTO
BLANK     <ENTER>
```

The blank solution must be prepared as a sample would be, using all of the sample preparation steps and reagents but without an actual sample.[2] After reaction the blank must be allowed to reach room temperature as described below. Insert the electrode into the solution, swirl gently for a few seconds and allow to sit. With the electrode in the solution, press the <ENTER/ON> or <YES>> key. After the measurement has been made, the blank result will be displayed as:

```
BLANK READING
01.2
```

After pressing the <ENTER/ON> key, the program will continue to the next step in the analysis as described below. A measured blank cannot be edited. Only stored blank values can be edited.

---

[2] When preparing a blank using the Standard Soil Extraction Solvent, DO NOT add any of the water from the bottom of the solvent vial.

Analysis

Prepare samples for analysis as described under **Sample Preparation**. Once the samples have been prepared, choose the appropriate analysis method and calibrate the meter. The program will display instructions as determined by the chosen method.

1. Make sure that the method has been selected, the instrument has been recently calibrated (including any blank determinations) and that the analysis solutions have been allowed to temperature equilibrate for at least 5 minutes. The display should read:

   ANALYZE SAMPLE
   method name

   Press the <ENTER/ON> key to continue to the screen:

   SAMPLE ID (save)
   sample id

   If the sample ID is correct, proceed to the next step. Otherwise change the ID as described under **Entering Data**. The indication "save" will appear in this screen if the option to save data points as they are taken has been selected (See **Data Management**)

2. Remove the electrode from the rinse solution and wipe the body carefully with a tissue. NOTE: DO NOT WIPE THE TIP OF THE ELECTRODE AS THIS MAY DAMAGE IT. Place the electrode in the vial to be analyzed and swirl it gently for several seconds.

3. Press the <ENTER/ON> key. The L2000DX will take a series of readings. Once the readings have converged on a constant value, the result will be displayed along with crucial method information. A beeper will sound to indicate that the new value is ready.

4. To analyze additional samples, press the <ENTER/ON> key. The program will check the ambient temperature and, if it is still within the allowable range, the program will cycle back to begin the next analysis. Proceed as in step 1 above to make the next measurement.

5. Samples may be saved for analysis at a later time, but the vials should be tightly capped. Once they have been read, samples should be discarded. Do **not** re-analyze samples.

6. Pipettes and oil should be disposed of as PCB/Organo-chlorine waste. Analysis solution can be disposed of as ordinary aqueous waste containing nickel.

Entering Data

All user input to the L2000DX Analyzer is through the 16 key keypad on the front panel. There are 12 alpha-numeric keys and 4 function keys. Audible feedback is provided, on all keys, to indicate that a key press has been registered by the processor. Data is entered using the alpha-numeric keys. These keys are active only when an input is required and will be acted on only when they are appropriate for the active screen.

When numeric input is required, only the numbers indicated on the keys will be registered. To change an entered number, use the arrow keys to move the cursor to the desired location and press the correct number key. To input letters in a text string, such as in a method name or sample ID, both an alpha-numeric key and the ALPHA key must be pressed. When an alpha-numeric key is pressed, the number associated with that key will be displayed first. The ALPHA key is then pressed once, twice, or three times to change the character to the desired letter. Continued ALPHA key presses will "scroll" the characters through the letters and the number shown on the face of the key. For example: Pressing the <7> key will first cause the "7" to be displayed. The cursor will move to the next space and, if the ALPHA key is pressed next, the "7" changes first to an "S", then a "T", followed by a "U". (Pressing the< ALPHA> key again will cycle the character back to the "7".) Once the desired character is displayed, pressing an alpha-numeric key will enter the next character, above the cursor.

Having a sample ID that includes a number automatically incrementing with each successive sample, will make assigning a unique sample ID much easier. This is accomplished by using "wildcards" in the sample ID. There are two "wildcard" characters that can be used. These are the "*" and the "?". They function much the same as their DOS equivalents. Entering an "*" in a sample ID will add an automatically sequencing number to the sample ID. The number begins at 00001 and increments up to 99999. (If less than 5 spaces are left in the sample ID then the number is limited to the number of digits left.) The "?" has the same function as the "*" but the appended number will only have the number of digits as question marks entered. NOTE: Editing a sample ID with a "wildcard" will freeze the number at the current value and eliminate the wildcard function.

Special Keys

The <NO and YES> keys function as either a yes/no response or as arrow keys to move the cursor through a text entry.

The four special function keys, ALPHA, OPTION, BACK/OFF and ENTER/ON are used to direct the operation of the program. The ALPHA key is used to input letters in a text string. The OPTION key, from the first or second screen, will transfer program operation to the **OPTIONS Menu**. From the **RESULT Screen** in the **Analysis Loop**, the OPTION key is used to view the chosen parameters of the currently active "Method". The BACK/OFF key is used to exit from any section of the program, back through successive screens to the **First Screen** and, ultimately, to turn the meter off. The ENTER/ON key will turn the instrument on and is used to enter any options chosen or to step forward in the program.

Electrode Check

The electrode is the most important component in the system. It is, therefore, important that the electrode be properly maintained and the initial setup will determine how well the electrode is functioning. Before turning the L2000DX analyzer on, insure that the electrode is properly connected to the back of the unit. Check the fill level in the electrode and, if necessary, refill the electrode with the Orion filling solution supplied with each lot of reagents. To fill the electrode, insert the tip of the filling spout of the filling solution bottle into the hole on the side of the electrode. Slowly squeeze the solution into the electrode body until it reaches the level of the hole.

Once the electrode has been filled, check the electrode output by going to "**Diagnostics**" from the **OPTIONS Menu**. Rinse and refill the RINSE vial with fresh rinse solution and swirl the electrode in the solution gently for a few seconds. Allowing the electrode to sit in the rinse solution, verify that the mV reading is greater than 140 mV. If the output does not reach at least 140 mV within a minute, refill the RINSE vial with fresh solution and recheck the output. If this does not improve the output, refill the electrode with fresh fill solution. (See **Electrode Care and Maintenance**).

Sample Preparation

Oil Samples

Before a transformer oil sample can be analyzed with the L2000DX, the PCB/Chlorinated Organics must be chemically converted to chloride.

1. Remove the cap from a black-capped tube and add oil up to the 5 mL line using a polyethylene pipettor (See Figure 2). Replace the cap tightly on the tube.

2. Break the bottom (colorless) ampule in the tube. Shake the tube well for 10 seconds.

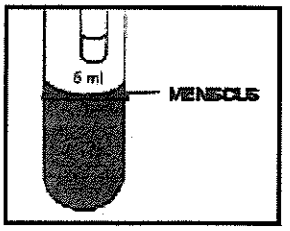


Figure 2

3. Break the top (gray) ampule in the tube. Shake the tube vigorously for 10 seconds Allow the reaction to proceed for additional 50 seconds (total of one minute), while shaking intermittently several times.

4. Using the 5 mL pipette, add five milliliters of extract solution to the back-capped tube. Tighten the cap securely and shake vigorously until the foam and dark color disappear. Vent the tube by partially unscrewing the black cap while holding the tube upright. Squeeze the test tube slightly while re-tightening the cap, and shake the tube vigorously for 20 seconds more. Vent again, tighten cap and stand the tube upside-down on the flat top of the cap and allow to settle for two minutes.

5. Place a polyethylene filter funnel in one of the 20 mL glass vials marked with the sample number. Position the black-capped test tube directly over the top of the funnel and open the dispenser nozzle (See Figure 3). Dispense the solution by carefully squeezing the sides of the tube. Stop as soon as the first drop of oil appears. Allow the solution to pass through the funnel, but remove the funnel before any oil can get through. Allow the solution to cool for five minutes. The sample is now ready for analysis.

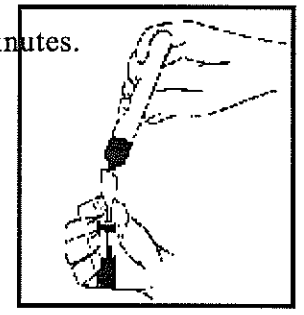


Figure 3

Soil Samples

To analyze a soil sample for PCB/Chlorinated Organics, the analyte must first be extracted from the sample. There are two solvent extraction systems available. Depending on the specific application, either the standard extraction solvent or the two-step procedure will be most appropriate. Both systems provide good, reproducible recoveries on dry sandy loam type soils. The standard procedure is somewhat faster and has fewer steps, but should not be used on wet or heavy clay soils. Regardless of the solvent system used, it is important that split samples be periodically analyzed by a reputable laboratory, not only to identify/confirm the Aroclor present, but also to confirm the extraction efficiency of the L2000DX solvent system. Contact Dexsil before trying to analyze other types of materials. NOTE: The quality of the final result is directly determined by sampling technique. It is, therefore, important to use proper protocols for sample collection and homogenization.

Standard Procedure for Soil Analysis

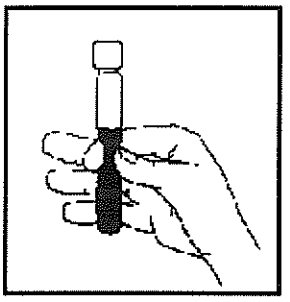


Figure 4

1. Using the metal spatula and the portable electronic balance supplied with the kit, weigh out ten grams of the soil into the empty, white-capped test tube. NOTE: Be careful not to get any foreign material into the weighed sample, i.e., absorbent material, large rocks, etc. The empty tube can be tared by placing it on the balance and pressing the <ON/OFF/ZERO> key.

2. Remove the black cap from the glass vial containing the extraction solvent and pour the entire contents of the vial into the tube containing the soil. Replace the white cap on the plastic test tube tightly and shake the tube vigorously for one minute. Break up any lumps of soil by squeezing the sides of the test tube during the shaking process. (See Figure 4) Allow the tube to settle upright for two minutes.

3. Remove the drying column from its foil pouch by poking the pointed end of the column through the foil and remove the red end-cap. Pull the plunger completely out of the 10cc syringe. Attach the end of the blue drying column to the tip of the syringe by sliding it over the collar on the tip of the syringe. This is a tight fit and the drying column may have to be carefully worked onto the syringe. Make sure that it is seated tightly.

4. Remove the black dispensing cap from the plastic test tube that contains two glass ampules. Slide the syringe-drying column assembly part of the way into the test tube which contains the two ampules. Stand the whole assembly upright. Using the polyethylene pipettor, remove the extraction solvent from on top of the soil and dispense it into the top of the open syringe barrel. You will need to recover enough extraction solvent to fill the syringe barrel to the 7 mL level. Try not to remove any soil with the solvent as this may clog up the drying column. After 7 mL of solvent has been dispensed into the syringe, replace the plunger into the back of the syringe and apply pressure so that the solvent is forced through the drying column at the rate of 2 or 3 drops per second. (NOTE: Do not force the solvent through the drying column too fast.) When the dried solvent fills the test tube to the 5 mL line, pull back on the plunger to stop the flow of solvent. Remove the syringe-drying column assembly from the test tube, and screw the black dispensing cap tightly onto the test tube.

5. Break the bottom (colorless) ampule in the test tube by squeezing the sides of the tube and shake the mixture well for 10 seconds. Break the top (gray) ampule in the test tube and shake the tube vigorously for 10 seconds. Allow the reaction to proceed for additional 50 seconds (total of one minute), while shaking intermittently several times.

6. Using the 5 mL pipettor, add five milliliters of extract solution to the back-capped tube. Tighten the cap securely and shake vigorously until the foam and dark color disappear. Vent the tube by partially unscrewing the black cap while holding the tube upright. Squeeze the test tube slightly while re-tightening the cap, and shake the tube vigorously for 20 seconds more. Vent again, tighten cap and stand the tube upside-down on the flat top of the cap and allow to settle for two minutes.

7. Place a polyethylene filter funnel in one of the 20 mL glass vials marked with the sample number. Position the black-capped test tube directly over the top of the funnel and open the dispenser nozzle. (See Figure 3) Dispense the solution by carefully squeezing the sides of the tube. Stop as soon as the first drop of the organic solvent appears. Allow the solution to pass through the funnel, but remove the funnel before any oil can get through. Allow the solution to cool for five minutes. The sample is now ready for analysis.

Two-Step Procedure for Soil Analysis:

1. Using the metal spatula and the portable electronic balance supplied with the kit, weigh out ten grams of the soil into the empty, white-capped test tube. NOTE: Be careful not to get any foreign material into the weighed sample, i.e. absorbent material, large rocks, etc. The empty tube can be tared by placing it on the balance and pressing the <ON/OFF/ZERO> key.

2. Add the contents of one break-top glass solvent vial to the test tube of soil sample. Replace the cap tightly on the tube and shake contents for 3 minutes making sure the entire soil sample is thoroughly wetted.

3. Add the colored water component contained in the 6 ml black-capped vial to the test tube. Recap tightly and shake for an additional 2 minutes.

4. Allow the mixture to separate for 2 minutes.

5. Remove the plunger from a syringe/filter unit in the foil package and remove the black-dispensing cap from the reaction test tube and stand it in the rack.

6. Remove the top layer from the soil test tube (solvent layer) using the polypropylene pipette provided and while holding the syringe/filter over the reaction tube, add 7 ml to the syringe/filter.

7. Add solvent from the syringe/filter up to the 5 ml line of the black-capped test tube. Replace the cap tightly on the tube.

8. Break the bottom (colorless) ampule in the black-capped test tube by squeezing the sides of the tube and shake the mixture for 10 seconds.

9. Break the top (gray) ampule in the test tube and shake the tube vigorously for 10 seconds. Allow the reaction to proceed for an additional 40 seconds (total of one minute), while shaking intermittently several times.

10. Using the 5 milliliter pipettor from the basic L2000 system, add 5 ml of the L2000 Extraction solution to the black-capped tube. Tighten the cap securely and shake the tube vigorously until the foam and dark color disappears. Vent the tube by partially unscrewing the cap while holding the tube upright. Squeeze the test tube slightly while retightening the cap and shake a second time. Vent again, retighten cap and stand the tube upside-down on the flat top of the cap and allow to settle for two minutes.

11. Place a filter funnel in one of the 20 ml glass vials marked with a sample number. Position the black-capped test tube directly over the top of the funnel and open the dispenser nozzle. Dispense the solution by carefully squeezing the sides of the tube. Allow the solution to pass thorough the funnel, but remove the funnel before any oil can get through. Allow the filtered solution to cool for five minutes. The sample is now ready for analysis with the L2000 Analyzer.

Water Samples

High Range (5-2000 ppm)

1. Fill sample tube with 10 grams of water sample.

2. Add 10 mL of isooctane extraction solvent and shake vigorously for 30 seconds.

3. Allow to separate into two phases for a minimum of 2 minutes. (If a emulsion forms, add sodium sulfate*, shake and allow to separate again). Using the disposable plastic pipette remove 5 mL of the top solvent layer and add to the black capped reaction tube.
(*available from major chemical suppliers).

4. Proceed with test as for an oil sample and quantify using the appropriate LX2000DX method.

Low Range (20 ppb - 5 ppm)

1. Collect sample in 1 quart narrow mouth glass bottle with zero head space. Cap tightly and store on ice until analysis.

2. When ready to analyze the sample, invert sample container gently once or twice and, using a plastic pipette, remove 35 mL of water from the sample jar by weighing 35 grams of water into a tared waste container and discard. Then add 10 mL of isooctane to sample container and shake vigorously for 2 minutes.

3. Add sufficient chlorine free distilled water to bring the water level up into the neck of the sample bottle (the solvent level should be just shy of the top of the neck) and allow to set for 3 minutes

4. Withdraw 5 mL of the upper solvent layer (**Do Not Remove Any Water With the Solvent**), add to the black capped reaction tube and cap tube tightly.

5. Proceed with the analysis as for an oil sample and quantify using the appropriate L2000DX method.

Wipe Samples

1. Locate the sealed glass vials containing chromatographic grade hexane and carefully break off the tip. Pour the entire contents into the vial containing the gauze pad. Grasp the soaked gauze pad with the disposable forceps and using an approved technique, wipe an area of 1000 cm^2. 1000

cm^2 is equivalent to a square measuring 31.6 cm or 12.5 inches on each side. It is equal to 1.08 ft^2. Allow the hexane to evaporate from the wiping material (approximately 1 minute).

2. Being careful not to contaminate the wipe, place it as loosely as possible into the tube with the white dispenser cap. Pour the extraction fluid, (10 mL of isooctane), into the white-capped tube. Tighten the cap and solvate the gauze thoroughly for 30 seconds. Squeeze the tube to make sure that the isooctane completely washes the gauze. This solvent now contains all the PCBs that were removed during the wiping procedure.

3. Remove the black dispenser cap from one of the reaction tubes. Open the spout on the white dispenser-capped test tube and dispense the isooctane extract into the black-capped tube up to the 5 mL line. Replace the cap tightly on the tube.

4. Break the bottom (colorless) ampule in the tube. Shake the tube well for 10 seconds.

5. Break the top (gray) ampule in the tube. Shake the tube vigorously for 10 seconds Allow the reaction to proceed for additional 50 seconds (total of one minute), while shaking intermittently several times.

6. Using the 5 mL pipette, add five milliliters of extract solution to the back-capped tube. Tighten the cap securely and shake vigorously until the foam and dark color disappear. Vent the tube by partially unscrewing the black cap while holding the tube upright. Squeeze the test tube slightly while re-tightening the cap, and shake the tube vigorously for 20 seconds more. Vent again, tighten cap and stand the tube upside-down on the flat top of the cap and allow to settle for two minutes.

7. Place a polyethylene filter funnel in one of the 20 mL glass vials marked with the sample number. Position the black-capped test tube directly over the top of the funnel and open the dispenser nozzle (See Figure 3). Dispense the solution by carefully squeezing the sides of the tube. Stop as soon as the first drop of oil appears. Allow the solution to pass through the funnel, but remove the funnel before any oil can get through. Allow the solution to cool for five minutes. The sample is now ready for analysis.

The OPTIONS Menu

Except for the **Analysis Loop**, all program functions of the L2000DX are accessed through the **OPTIONS Menu**. The options available are:

```
1) METHOD - CREATE/EDIT
2) HEADER INFORMATION
3) DATA MANAGEMENT
4) PRINTER OPTIONS
5) TIME AND DATE
6) DIAGNOSTICS
7) PC UPLOAD
8) SET TEMP
```

The **OPTIONS Menu** is entered by pressing the <OPTION> key at the **First Screen** or the **SELECT METHOD Screen**. Once in the **OPTIONS Menu**, each option will be displayed for 3 seconds and they will continually loop for 3 minutes or until an option is chosen. An option is chosen by pressing the appropriate number for the desired option.

I) Creating and Editing a Method

The key to the utility of the L2000DX analyzer is the versatility of the methods that can be developed for the instrument. A "method" is the complete set of parameters that are necessary to convert a chloride reading into an accurate quantification of the targeted analyte. Preprogramed in the meter are a number of methods for the analysis of many common environmental contaminants. These methods can be modified and custom methods can be built by setting any of the conversion parameters used to convert the chloride reading into an equivalent analyte concentration.

Each method requires 8 parameters to be set. These parameters are determined by the composition of the sample and the chemistry of the sample preparation and/or extraction procedures used to introduce the sample into the system. They can be either calculated from known parameters or determined empirically from preliminary experiments. The parameters common to all methods and their allowable ranges are tabulated below.

Parameter	Range of Values
Blank Subtraction	yes/no
Chlorine Content	10 - 99.9
Sample Size Correction	0.01 - 99.9
Extraction Efficiency Correction	0.01 - 99.9
Matrix	Oil, Soil, Water, Wipe
Units to be Displayed	ppm, mg/kg/ ppb, ug/sdm
Analyte Label	16 character alpha-numeric
Method Name	16 character alpha-numeric

To create a new method or edit an existing method choose 1) METHOD CREATE/EDIT" from the **OPTIONS Menu**. Then choose either 1) EDIT METHOD@ to edit an existing method or 2) CREATE NEW@ to create an entirely new method. Choosing 2) CREATE NEW@ will begin the parameter selection for the new method directly. If 1) EDIT METHOD@ is chosen, the next screen will be:

```
CHOOSE METHOD:x
method names
```

to choose the method to edit. The existing method names can be viewed by pressing the <<NO> key to scroll backward or <YES>> key to scroll forward. Once the desired method is displayed pressing the <ENTER/ON> key will select the method and the choice will be given to either:

```
1) MODIFY METHOD
2) DELETE METHOD
```

Choosing 2) DELETE METHOD@ will delete the chosen method. This is the only way to delete unwanted methods. (NOTE: Preprogramed methods cannot be deleted. Only user created methods can be deleted.) Choosing 1) MODIFY METHOD@ will begin the method parameter editing. After this screen, whether editing or creating a method, the steps are the same.

Blank Subtraction

The first parameter that will be displayed will be:

```
BLANK SUBTRACT
YES OR NO
```

Enabling blank subtraction allows for the subtraction of a reagent blank, if any, from the raw chloride measurement before the final result is displayed. When blank subtract is on, the analyst will be prompted to either measure a blank, use a previous blank or input a blank value to be used. During the analysis of unknowns, if a blank is subtracted, the results will be displayed with a "B" in the upper right corner of the display.

Chlorine Content

The next parameter that can be input is the chlorine content of the target analyte.

```
% CHLORINE XX.X
99.9%
```

Input as a percentage, this parameter is used to convert the total chloride reading into an equivalent analyte concentration prior to the display of the result.

Size Correction

The size correction is used to adjust the results for the sample size used for a particular analysis. The assumed sample size is 5 grams of a liquid sample, 10 grams for a soil sample and 1000 cm^2 for wipe samples. The correction would be calculated from the ratio of the expected sample size to the actual sample size. For example, if a 5 gram sample is used for soil analysis, the correction would be 10/5 or 2. NOTE: In the analysis of transformer oil, the sample is typically taken volumetrically, therefore, a correction for the density of the oil is incorporated into all pre-programed Oil Methods. The density of a typical dielectric fluid used in transformers, Diala-A, is 0.8833 g/cc. The size multiplier, built into each Oil Method, is then 1/0.8833 = 1.13. This allows the results to be displayed as ppm or mg/kg. The size correction screen is:

```
SIZE MULTIPLIER
1.13
```

Extraction Efficiency Correction

Because there are one or more extractions involved in the preparation of samples for measurement, a correction for extraction efficiency has been provided for in the method parameters in the screen:

```
EXTRACTION MULT
1.11
```

The correction is entered as a multiplier applied to the final result. For example the recovery of chloride ions from the organic phase in the final extraction of oil samples is 90%. For oil samples, this is the only extraction step, therefore, the correction multiplier would then be 1/0.90 or 1.11. The typical extraction efficiency for the recovery of organics from soil samples is 80%. Combined with a chloride recovery of 90% the overall extraction correction is (1/(0.80 x 0.90)) or 1.39.

Matrix

The matrix label for sample type appears in the header information and indicates what type of sample was analyzed. The available labels are:

```
1)OIL 2)SOIL
3)WATER 4)WIPE
```

To choose the matrix label enter the number of the matrix option and press the <ENTER/ON key. NOTE: This is a label only. Choosing a matrix does not set any of the numeric conversion factors. They must be set separately.

Units

The units variable in a particular method development is simply the text that appears associated with the displayed, printed or stored result. To choose a label, press the number for the desired units. The available labels are:

```
1)PPM 2)MG/KG
3)PPB 4)ug/sdm
```

Any of the units can be combined with any of the matrices chosen, however, ug/sdm is only meaningful for wipe samples. The units "ug/sdm" is shorthand notation for micrograms per square decimeter which is equivalent to micrograms per 100 square centimeters, i.e., ug/sdm = ug/100scm.

Analyte Label

The analyte label is a 16 character label that will appear on the display indicating the analyte equivalent concentration, assuming that all organic chlorine detected in the sample were that analyte. The entry screen appears as:

```
ANALYTE LABEL
analyte label
```

This label can be left blank, however, something should be entered as this is the only way to identify the expected analyte when the final analytical result is displayed.

Method Name

After all of the method variables have been selected, the user will be prompted for the name under which the method will be stored in memory:

```
METHOD NAME
method name
```

There must be a unique name associated with each method. A method name consisting of all blanks is not allowed for this option. If an existing method name is entered, the prompt:

```
EXISTING METHOD
OVERWRITE IT?
```

will appear. Choosing YES will cause the new method to over write the existing one. (NOTE: Pre-programed methods cannot be over-written. If they are modified, they must be stored as a new method.) Choosing NO will loop the program back to the **METHOD NAME** screen for a new file name. Once a valid method name has been selected, program operation will return to the **OPTIONS Menu**.

2) Header Information

Associated with each block of data are descriptors that will identify all of the important information needed to use the collected data at a later date. Along with the method information, date and time, etc., are header fields available for user selected information. Each of these fields is 16 characters long and the information is entered from the key pad as described under **Entering Data**. The available fields are:

Operator ID
Customer ID
Job ID
Location ID
Comments

If any of these parameters are changed a new header will be stored in memory, to be linked with the data, as well as printed on the printout, if the printer is enabled. The individual fields are edited through each of the individual screens. Text is entered as described under **Data Entry**.

When printed, the header information will appear as follows:

```
Time (hh:mm) Date (mm/dd/yyyy)          Operator ID
Customer ID                             Job ID
Location                                Comments
Method Name                             Matrix

BLANK SUBTRACT: (yes/no, If yes: value)
                                        Analyte Label
                CHLORIDE                ANALYTE
SAMPLE ID       READING                 CONCENTRATION
```

Pressing the <2> key, while in the **OPTION Menu**, will begin the editing of header information at the screen:

```
HEADER SETUP
<BACK> TO RETURN
```

Pressing the <ENTER/ON> key will continue to the next screen:

```
OPERATOR ID
XXXXXXXXXXXXXXXX
```

After editing the text, pressing the <ENTER/ON> key will allow editing of the next header field:

```
CUSTOMER ID
XXXXXXXXXXXXXXXX
```

After editing the text, pressing the <ENTER/ON> key will allow editing of the next header field:

```
JOB ID
XXXXXXXXXXXXXXXX
```

After editing the text, pressing the <ENTER/ON> key will allow editing of the next header field:

```
LOCATION ID
XXXXXXXXXXXXXXXX
```

After editing the text, pressing the <ENTER/ON key will allow editing of the next header field:

```
COMMENTS
XXXXXXXXXXXXXXXX
```

After editing COMMENTS, pressing the <ENTER/ON> key will loop the program back to the first **HEADER SETUP** screen. Pressing the <BACK/OFF> key will exit to the **OPTION Menu** or pressing the <ENTER/ON> key will begin the editing process again.

3) DATA MANAGEMENT

A number of data management options are available to the user. They can be selected from the **DATA MANAGEMENT Menu**. Data management options are selected independent of the method chosen and remain in effect until changed by selecting a new option. Enter the **DATA MANAGEMENT Menu** by pressing the <3> key from the **OPTION Menu**. The first option is:

```
STORE EACH
POINT (Y/N)?
```

Activating this option by pressing the <YES>> key will cause each data point to be stored in memory as it is taken. Data points are stored along with all of the method and header information associated with them. The default for data storage is to store each data point. All data points will be stored automatically unless this option is changed. Pressing the <<NO> key will deactivate this option and no further data will be stored unless this option is activated again.

Once the data storage option has been chosen the next screen is:

```
CLEAR DATA
MEMORY (Y/N)?
```

This option should be used periodically to clear out old or unwanted data. Choosing this option erases all stored data and header information and reformats the data storage space. The current header information and the current method are not altered. After choosing this option, confirmation will be required before the data will actually be erased:

```
ALL DATA WILL BE
LOST. PROCEED?
```

Pressing the <YES>> or <<NO> key will complete this option as desired and exit back to the **OPTION Menu**.

4) PRINTER OPTIONS

The default option for the printer is to print all data points as they are collected. Anytime the header information is edited, a new header is printed. Anytime an external printer is connected to the printer port on the back of the unit, all printouts will be directed to the external printer.

The first option under **4) PRINTER OPTIONS** is:

```
PRINT POINTS
WHEN TAKEN(Y/N)?
```

Pressing the <YES>> key will cause each data point to be printed as it is taken. (This option refers only to the printing of data in the **Analysis Loop**. Printing stored data points is the next option.) This option can be turned off to save battery power in the field. It will also be turned off automatically if a low power condition exists. Turning off the print option by pressing the <<NO> key will result in no data printout when the data is taken in the **Analysis Loop**. To obtain a hard copy of the data, the data can be printed at a later time.

To print data after it has been stored, press the <YES>> key from the screen:

```
PRINT LAST XXXX
   POINTS
```

Choosing this option will cause the last xxxx data points to be printed on either the internal or an external printer. The default is all of the stored data. This number can be edited using the numeric key pad.

To print the current header information, press the <YES>> key from the screen:

```
PRINT HEADER NOW
(Y/N)?
```

This option will print all of the header information on the current printer.

The last print option is to print out a hard copy of the method parameters. At the screen:

```
PRINT METHOD NOW
(Y/N)
```

Press the <YES>> key to print the method.

5) DATE AND TIME

To change the date and/or the time, choose this option and enter the correct information in the indicated fields. The first option to change is:

```
TIME XX:XX
hh:mm <ENTER>
```

Enter the time using the 24 hour clock: hours then minutes. The last entry will cause the current time to be briefly displayed and then the screen:

```
DATE mm/dd/yyyy
mm/dd/yyyy <ENTER>
```

Enter the date as a 2-digit month and day followed by 4-digit year.

6) DIAGNOSTICS

This option should be used to periodically check the output of the electrode, such as at the beginning of each day or if the electrode has been stored for an extended period of time. Or, if an electrode error occurs, this option will allow the user to verify the fault and take corrective action. Once this option has been chosen, the L2000DX will begin measuring the mV output of the electrode and display the results along with the ambient temperature.

```
DIAGNOSTICS x.xx
xx.x mV xx °C
```

7) UP LOAD

Chose this option to upload stored data to a PC. The L2000DX Data Manager must be loaded an running on the target PC. (See **L2000DX Data Manager**.)

To upload data, first connect the L2000DX Analyzer to the PC using the upload cable provided. Once connected, select the **7) PC UPLOAD** option by pressing the <7> key from the **OPTION Menu**. The display will change to:

```
BEGIN UPLOAD
<BACK> TO STOP
```

Then, from the PC, choose BEGIN UPLOAD to begin the upload.

8) SET TEMP

This option allows for the field adjustment of the internal thermistor. When adjusting the L2000DX thermistor, the resulting temperature measurements are only as accurate as the thermometer used for adjustment.

To adjust the internal thermistor, choose **8) SET TEMP** by pressing the <8> key from the **OPTIONS Menu**. The L2000DX will check the current temperature and display the raw result on the screen:

```
INPUT TEMP IN °C
XX.X °C (RAW)
```

enter the current temperature followed by pressing the <ENTER/ON> key. The adjusted temperature will be displayed with the message:

```
NEW TEMP TO USE
XX.X °C OK(Y/N)?
```

NOTE: An adjustment of ±5°C max. is allowed. If the input temperature differs by more than this, the temperature will be fixed at the max/min allowed.

L2000DX Data Manager

Introduction

The L2000DX Data Manager is a PC based data base program specifically designed for data from the L2000DX Analyzer. It provides four basic capabilities: uploading stored data, report generation, data export functions and basic data base functions for tracking data from one or more L2000DX Analyzers.

Installation

To install the Data Manager: Insert program CD into CD drive and run setup.exe. The install program will provide guidance for all installation steps.

Uploading data

Each time you upload data from a particular L2000DX unit, the software reads from the unit the date it was last cleared and stores this date for future reference. It also stores the number of the last record uploaded. The next time you upload data from the same unit, the software compares the date it reads from the unit with the stored date. If they are the same, it continues uploading data from the last uploaded record, eliminating any duplication in your data set. If the dates are different, the software assumes you have cleared the unit since your last upload and begins uploading data with the first record in the unit.

Note that the software keeps track of multiple L2000DX units by reading from each unit its unique, factory-supplied serial number.

To upload data from an L2000DX analyzer, connect the analyzer to the PC using the supplied cable then select 7) PC UPLOAD on the L2000DX unit. Once the unit is connected and ready to upload choose **Upload** from the **Communicate** menu on the PC. Choosing it opens the **L2000DX Upload** box. Once this box is open click **OK**. The program will contact the L2000DX analyzer and begin uploading stored data.

If you want to cancel the process, click on the **X** in the upper right corner of the box, then click Abort Transfer in the resulting Communication Status message box.

Selecting Data

Choosing **Selection** at the menu bar causes the **Define Sample Selections** dialog box to appear. Here you can choose to display only some of the available data records by using combinations of various filters.

Note: These filters will affect the display, printing and export of data.

Selection Filter - This heading (left side of the dialog box) contains four selections: **Header, Customer, Job** and **Location**. Check the checkbox for the filter(s) you want to use. You cannot

combine **Header** with any of the other three filters, however the other three may be used in any combination. When you select one of the filters, a drop-down menu appears displaying the values available in the active data set.

> **Header Range Filter**- This area lets you choose a range of consecutive Header numbers. The Starting and Ending must be different, and the Ending number must be greater than the Starting number.
>
> **Sample Range Filter**- This area lets you choose a range of consecutive Ref #s. The Starting and Ending may be the same, but the Ending number must be greater than the Starting number.

As you make various filter selections, the data in the L2000DX window will change to reflect your choices, even while the Define Sample Selections dialog box is open.

The L2000DX Data Manager combines filters so that the data displayed, printed or exported satisfies all the filters. For example, if you select a **Customer** and a **Job** filter, you will see only data records with the required Customer number and the required Job number, not data with either the Customer number or the Job number.

As soon as you select any filter, a **Clear All** button appears in the dialog box to allow you to quickly clear all filter selections. A **Clear All Filters** button will also appear on the L2000DX Data Manager window so you can clear filters later without returning to the **Define Sample Selections** dialog box.

Generating a Report

Choosing **Print Report** at the **File** menu causes the **Report Page Format** dialog box to appear. This box allows you to specify a title for your report.

In the edit field, type a title of up to 70 characters to be centered in the header of the report form.

Click the **Print Preview** button to see what the report will look like. You may choose to print the report from the preview screen.

Click the **Cancel** button to dismiss the **Report Page Format** box without previewing the report.

Exporting Data

To export data to a Microsoft Excel spread sheet, choose **Export** from the **File** menu. This will open the **Save As** dialog box to allow you to choose the location for saving the currently active data set in Microsoft Excel spreadsheet format.

Care and Maintenance

Your L2000DX is designed for ease of use and reliability and requires very little maintenance. By following the instructions outlined below, you should be able to obtain maximum life from the electrode and the instrument itself.

Changing Paper

1. Turn the L2000DX Analyzer off and remove the external power plug, if attached. Turn the analyzer over, lay flat on smooth surface and remove paper access door on bottom.

2. Remove the empty paper roll by grasping the roll in the middle with one hand. With the other hand, gently push one side of the paper bracket out away from the roll, while lifting the roll in the middle. This will free one end of the spindle. Continue lifting the roll out of the bracket until it is free.

3. Remove the paper spindle from the spent core and insert into the new roll of paper.

4. With the L2000DX Analyzer still upside down and the paper access on the right side, slide the analyzer to the edge of the table until it hangs over the edge far enough for the paper slot on the keypad to be accessible. DO NOT ALLOW THE UNIT TO HANG TOO FAR OVER AS IT MAY FALL.

5. Install the new paper roll into the holder with the loose end of the paper coming off the top of the roll. (Treated side must be on the top of the paper as it exits the roll.) Grasping the roll in the middle, slide the roll between the uprights of the paper holder until the ends of the spindle snap into the holes in the paper bracket. While pushing in the paper roll, it may be necessary to slide the roll of paper to one side to deflect the paper holder enough to clear the other side. Once in place, the roll should spin freely and remain on the holder.

6. The paper should now be installed in the paper holder with the free end on top of the roll. With a clean square end, fold one corner of the paper back flush with the opposite side to form a 45• point on the end. If there is not a clean square end on the paper, pull off a length of paper and tear a square edge off, along the bottom of the case, before folding the corner.

7. Place the paper flat on the circuit board with the pointed end of the paper extending over the edge of the paper slot. Position the point approximately 1 inch from the side of the slot and, while keeping the paper flat on the board, slide the paper forward so that the point goes under the front edge of the green circuit board and into the printer. Continue pushing the paper forward, while gently moving the paper from side to side, until the paper exits the front of the analyzer.

8. Gently pull the paper through from the front of the analyzer until a section of the full width of paper is exposed.

9. If the paper does not exit the front of the analyzer, remove the paper and retry from step 7 above.

10. Once the paper has been installed, replace the paper access door and turn the analyzer back over.

General Care

Do not allow the instrument to get wet. As with any electronic instrument, water is the quickest way to destroy the L2000DX's components. Store the instrument in its case when it is not in use and do not expose it to extremely humid environments.

Be careful not to spill any of the solutions on the instrument. They are all acidic and will quickly destroy the circuitry if they come into contact with it. If solutions are spilled onto the instrument, wipe it off quickly with a damp cloth. Do not store solutions in the same case with the instrument, as any leakage may cause serious damage. This is particularly important when the instrument is to be shipped by air.

When using the L2000DX where AC power is available, operate the instrument with the AC adapter. This will prolong the life of the battery and insure that the battery is always charged.

Always store the L2000DX with fully charged battery. (A completely dead battery could take approximately 6-8 hours to charge fully.)

If the L2000DX is to be stored for prolonged periods of time, it is important to periodically charge the battery. This will prolong the battery life and insure that the analyzer is always ready for use.

Protect the analyzer from static electric charges. The keypad and case are isolated from the internal circuitry and are not susceptible to static discharge. However, when connecting the electrode to the meter, under certain environmental conditions, the static discharge may reset the power management circuit. Before connecting the electrode, first touch the outer ring of the BNC connector with one hand while holding the metal connector on the electrode in the other. Then connect the electrode to the Analyzer.

The L2000DX analyzes for PCBs/Chlorinated Organics by finding the total amount of chlorine in a given sample. It is important, therefore, to keep all extraneous sources of chlorine away from the instrument and the various solutions. Take extra precaution when working near salt water or under very warm conditions when contamination by perspiration is possible. Never pour used CALIBRATION, RINSE, or EXTRACT solution back into a storage bottle as the entire bottle may become contaminated.

Electrode Care and Maintenance

The electrode is the most sensitive component of the L2000DX system. Care must be taken not to damage the sensing membrane on the tip of the electrode. If it does become damaged or contaminated, it may be possible to restore the electrode response by polishing. To polish the electrode: Remove one of the membrane polishing strips from the instrument case and place it flat on a solid surface, abrasive side up. Place one or two drops of RINSE solution or distilled water on the abrasive strip. With the electrode perpendicular to the abrasive strip, polish the electrode tip by gently moving it in a circular motion for about 30 seconds. Apply constant pressure, but do not push too hard on the electrode. When finished, soak the electrode for five minutes in rinse solution. Polishing strips can be reused several times. NOTE: DO NOT OVER POLISH. ELECTRODES SHOULD BE POLISHED ONLY WHEN NECESSARY.

When an electrode is not to be used for a period of more than one week or for an indefinite period, drain the filling solution from the electrode, flush the inside one or more times with distilled water, and store it dry with the protective black cap to protect the sensing membrane. Make sure to follow the procedures for restoring electrodes before using the electrode again (see **Restoring the Electrode After Extended Storage**).

Otherwise, the electrode filling solution should not be allowed to evaporate causing crystallization. For short periods of time between sample measurements including up to one week storage, maintain filling solution in the electrode and store the electrode in rinse solution. (NOTE: If a period of 12 hours [overnight] or longer elapses between measurements, drain a portion of the filling solution from the electrode, add additional filling solution to the filling hole, and start with fresh rinse solution.)

Trouble Shooting

The instrument does not turn on.

1. The battery is dead. Plug in the AC adapter and retry.

2. The power management circuit has been overloaded and is locked due to a severe static electric discharge during the connection of the electrode. Open the back of the instrument by removing the four Phillips head screws. Disconnect the battery and reconnect it. The analyzer should turn on as normal except that the time and date will have been lost and must be re-entered. Follow the procedure outline under **Electrode Care** for connecting the electrode.

The instrument turns on but the time and date have been lost.

1. The power management circuit has been overloaded and has reset. This is most likely due to a static electric discharge during the connection of the electrode. Re-enter the time and date and proceed with the operation of the instrument. Follow the procedure outline under **Electrode Care** for connecting the electrode. Check electrode output in `DIAGNOSTIC` Mode.

2. The battery has been disconnected and reconnected, resetting the power management circuit. Proceed as above.

The instrument turns off when plugging in the electrode.

1. A large static electric discharge has overloaded the power management circuit. Turn Analyzer on in the normal manner. Power-up will be to the time and date screen. Follow the procedure outline under **Electrode Care** for connecting the electrode.

The instrument does not provide a millivolt reading, in "DIAGNOSTICS", after the electrode has been plugged in.

Check the electrode to verify that it contains the filling solution. If it is empty or low, refill the electrode as described in: **Restoring the Electrode After Extended Storage.**

1. Make sure that the bottom half inch (centimeter) of the electrode is completely immersed in the rinse solution.

2. Check the cable for loose connections.

The millivolt reading will not exceed 140 mV.

1. Replace the RINSE solution, and swirl the electrode for several seconds before letting the reading stabilize.

2. Replace the filling solution in the electrode as described in: **Restoring the Electrode After Extended Storage.**

3. Polish the end of the electrode. Remove one of the membrane polishing strips from the instrument case and place it flat on a solid surface, abrasive side up. Place one or two drops of RINSE solution or distilled water on the abrasive strip. With the electrode perpendicular to the abrasive strip, polish the electrode tip by moving it in a circular motion for about 30 seconds. Apply constant pressure, but do not push too hard on the electrode.

4. DO NOT OVER POLISH!! ELECTRODES SHOULD BE POLISHED ONLY WHEN NECESSARY.

The electrode will not calibrate.

1. During calibration the output from the electrode is checked. If it is outside the acceptable range, the electrode will not calibrate and the program will jump to "DIAGNOSTICS". In this mode check the output of the electrode. It should be between 50 mV and 75 mV. To obtain the proper millivolt output, the tip of the electrode must be immersed in calibration solution. If the electrode contains adequate filling solution, and is immersed in **fresh** calibratio solution and allowed to equilibrate, the output should reach the correct level.

2. The electrode will not calibrate, even after it has equilibrated:

   a. Change the calibration solution and refill the electrode as described in the **Restoring the Electrode After Extended Storage.**
   b. If this does not solve the problem, polish the electrode as outlined in **Care and**

Maintenance.

c. If these procedures do not work, the electrode may need to be replaced. Contact Dexsil for return information.

Insufficient quantity of solvent recovered from soil extraction.

Some soils are extremely dry or may contain a large percentage of organic material which can absorb enough extraction solvent so that there is less than the required amount of solvent available to complete the test. When soils of this type are being tested, five grams of sample instead of ten may be used. The final reading is then doubled or the Method is modified so that the **Sample Size Correction** is changed to **twice** its normal value. For instance, if, after weighing in five grams of soil, the final reading on theL2000DX is 35 ppm, the actual result for that sample is 70 ppm. Make sure to use the full quantity of extraction solvent and follow the instructions as you would fo any sample. NOTE: This technique causes a loss of precision and should not be used unless absolutely necessary.

Error Messages

CAL ERROR: This message is generated during calibration if the electrode output is not between 50 mV and 75 mV. If this message occurs:

1. Check solutions and change if not correct or not fresh.

2. Check electrode output and refill with fresh fill solution if necessary.

CHECK DRIFT: This message indicates that there is excessive drift in the electrode output either during a measurement or calibration. If this message occurs during the measurement of an unknown, the best estimate of the analytical result will be displayed below the warning. This number is for reference only, it may not be reliable. The sample must be re-analyzed to obtain an acceptable result. If this message occurs during a calibration, the electrode mV output will appear below the warning. If the warning appears, check the following:

1. Check the electrode performance as described under **Restoring the Electrode After Extended Storage**. Follow the steps for electrode draining and refilling, then recheck the electrode output. If this message occurs during calibration, the electrode is most likely damaged. See above under **The electrode will not calibrate.**

2. Check electrode leakage - The electrode is designed to leak a small amount of filling solution through the gap between the sensing pellet and the epoxy body. If the leak rate is too slow or too fast the performance of the electrode may be adversely affected. The electrode leak rate should be between 0.2 cm and 3 cm in 24 hours (as measured from the filling hole to the top of the fill solution).

3. Check for sample contamination. Some samples contain high levels of sulfur, heavy metals, waste oil, or other organics that may carry over to the final extract. If these compounds come into contact with the electrode, they can poison the sensing element. The output from the electrode will deteriorate with repeated exposure to these contaminants. If repeated samples cause a drift error, but the electrode checks out with the standard solutions, check for sample contamination.

CHECK ELECTRODE: This message indicates that the electrode output has exceeded 180 mV. If this message appears, the electrode has been damaged or the wrong electrode has been attached to the L2000DX analyzer. Check for the correct electrode then follow steps for restoring an electrode.

HIGH: This message indicates that an unknown reading is above 5000 ppm in analyte concentration. This message only appears during an unknown measurement. If this message occurs, re-analyze the sample using a smaller sample size.

LOW: This message indicates that an unknown reading has resulted in a negative analyte concentration. This can be caused by either an electrode malfunction or an improper blank value used, i.e., a large blank value not representative of the true blank. If this message occurs, check the blank value used or check the electrode function as described above. If the electrode checks out, investigate possible co-contaminants in the sample that may have adversely affected the electrode.

Appendix A: Additional Information Available from Dexsil

DTR-10-01 "Alternative Methods of PCB Analysis", Stephen Finch, Dexsil Corporation; Generators Journal, Winter 1990.

DTR-10-02 "One Example Where Chromatography May Not Necessarily Be the Best Analytical Method", S.R. Finch, D.A. Lavigne-Dexsil Corporation, R.P.W. Scott, Ph.D.-Georgetown University; Journal of Chromatographic Science-July 1990.

DTR-11-01 "A Comparison of Current PCB Analytical Techniques", Stephen Finch-Dexsil Corporation; PCB Forum, 3rd International Conference for the Remediation of PCB Contamination, 1991.

DTR-11-02 "Case Study of a New Field Screening Tool for Delineating Soil PCB Contamination", Mark B. Williams, PE and John S. Flickinger-Dames & Moore (Madison, WI), Joseph E. Shefchek, CHMM-Wisconsin Power & Light (Madison, WI), E. Jonathan Jackson, CHMM-Haliburton NUS Environmental Corp. (Aiken, SC); Proceedings: 1991 EPRI PCB Seminar.

DTR-11-03 "PCB Determination, Simple/Low Cost or Complex/Expensive, Which Method is the Most Reliable in the Field?", S. Finch, pp. 45-1 to 45-4; Proceedings: 1991 EPRI PCB Seminar

DTR-12-01 "Application of a New PCB Field Analysis Technique for Site Assessment", Roger D. Griffin-Griffin Environmental; Proceedings of Hazmacon '92 March-1992.

DTR-12-02 "Available Options for the Analysis of PCBs", Stephen Finch; Environmental Science and Engineering p. 15., Feb.-March.

DTR-13-01 "Electrochemical Method for Surface Testing of PCB Contaminated Electrical Equipment", Stephen Finch; 7th Annual Industry and PCB Forum, June 1993, Canadian Electricity Forum.

DTR-13-02 "A Comparison of Popular Field Screening Methods for PCB Contamination in Soil", Alvia Gaskill-Environmental Reference Materials, 1993 EPRI PCB Seminar.

DTR-14-02 "Comparison of the Response of PCB Test Methods to Different PCB Aroclors", Stephen Finch-Dexsil Corporation; Proceedings of "The Tenth Annual Waste Testing and Quality Assurance Symposium", July 11-15, 1994 Arlington, VA.

DTR-14-03 "Effect of Transformer Oil, Petroleum Hydrocarbons and Inorganic Salt as Interferences in Field Screening for PCB Contamination of Soil", Alvia Gaskill; Proceedings of "The Tenth Annual Waste Testing and Quality Assurance Symposium", July 11-15, 1994 Arlington, VA.

DTR-17-01 "Determination of Chlorinated Hydrocarbon Concentrations in Soil Using a Total Organic Halogen Method", T.B. Lynn, J.C. Kneece, B.J. Meyer, A.C. Lynn; Presented at the 13th Annual Waste Testing & Quality Assurance Symposium, July 6-9, 1997, Arlington, VA.

DTR-17-02 "Improved Extraction Efficiency of Polychlorinated Biphenyls From Contaminated Soil Using a Total Halogen Screening Method", W.S. Schutt-Young, Ph.D., A.C. Lynn, T.B. Lynn, Ph.D., B.J. Meyer, M.J. Krumenacher; Presented at "EnvirACS '97" held at the 13th Annual Waste Testing & Quality Assurance Symposium, July 7-9, 1997, Arlington, VA.

DTR-18-03 "Electrochemical Technique/Ion Specific Electrode Dexsil Corporation L2000 PCB/Chloride Analyzer", A.B. Dindal, S. Billetts, Environmental Technology Verification Report, US-EPA, Office of Research and Development, Washington, D.C., EPA/600/R-98/109, August1998

DTR-20-01 PCB Detection Technology Dexsil Corporation L2000DX Analyzer, A.B. Dindal, C.K. Bayne, E.N. Koglin, Draft Environmental Technology Verification Report, US-EPA Office of Research and Development, Washington, D.C. Report, December 2000

DTR-21-01 "Analysis of Water for PPB Range Chlorinated Organics Using a Total Organic Chlorine Analyzer," Theodore B. Lynn, Ph.D., Mary Kate Boggiano, Larry M. Sacramone, David L. Balog, Andrew C. Lynn, Dexsil Corporation, Presented at the Waste Testing and Quality Assurance Symposium 2001, August 13-16, 2001, Arlington, VA

DTR-21-02 "Low Level Detection of PCE in Monitoring Well Samples Using a Total Organic Chlorine Based Field Method," Theodore B. Lynn, Ph.D., Dexsil Corporation, Keith A. Wright, Camino, California, Presented at the Waste Testing and Quality Assurance Symposium 2001, August 13-16, 2001, Arlington, VA

DTP-09-01 "Accurate On-Site Analysis of PCBs in Soil, A Low Cost Approach", Deborah Lavigne-Dexsil Corporation.

DTP-09-02 "Field Test Kit for Quantifying Organic Halogens in Water and Soil", Deborah Lavigne-Dexsil Corporation.

DTP-11-01 "PCB Analysis by Gas Chromatography-What do the Numbers Mean? 1991", Stephen Finch-Dexsil Corporation.

DMR-16-01 EPA Method 9078 "Screening Test Method for Polychlorinated Biphenyls in Soil", 3rd edition SW 846- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, June 1997; Federal Register Vol. 62, No. 114, 6/13/97, Rules & Regulations.

Appendix B: L2000DX System Methods Table

Compound	Blank Subtract	%Chlorine	Size Multiplier	Extraction Multiplier	Matrix	Units	Analyte Label	Method Name
Aroclor 1242	yes	42	1.13	1.11	Oil	ppm	AROCLOR 1242	1242 OIL
Aroclor 1242	yes	42	1	1.33	Soil	ppm	AROCLOR 1242	1242 SOIL
Aroclor 1242	yes	42	1	1.1	Soil	ppm	AROCLOR 1242	2 STEP 1242
Aroclor 1242	yes	42	1	1	Wipe	• g/100scm	AROCLOR 1242	1242 WIPE
Aroclor 1242	yes	42	1	1.25	Water	ppm	AROCLOR 1242	1242 WATER HIGH
Aroclor 1254	yes	54	1.13	1.11	Oil	ppm	AROCLOR 1254	1254 OIL
Aroclor 1254	yes	54	1	1.33	Soil	ppm	AROCLOR 1254	1254 SOIL
Aroclor 1254	yes	54	1	1.1	Soil	ppm	AROCLOR 1254	2 STEP 1254
Aroclor 1254	yes	54	1	1	Wipe	• g/100scm	AROCLOR 1254	1254 WIPE
Aroclor 1254	yes	54	1	1.25	Water	ppm	AROCLOR 1254	1254 WATER HIGH
Aroclor 1260	yes	60	1.13	1.11	Oil	ppm	AROCLOR 1260	1260 OIL
Aroclor 1260	yes	60	1	1.33	Soil	ppm	AROCLOR 1260	1260 SOIL
Aroclor 1260	yes	60	1	1.1	Soil	ppm	AROCLOR 1260	2 STEP 1260
Aroclor 1260	yes	60	1	1	Wipe	• g/100scm	AROCLOR 1260	1260 WIPE
Aroclor 1260	yes	60	1	1.25	Water	ppm	AROCLOR 1260	1260 WATER HIGH
Askarel A	yes	99	1.13	1.11	Oil	ppm	ASKAREL A	ASKAREL A OIL
Askarel A	yes	99	1	1.33	Soil	ppm	ASKAREL A	ASKAREL A SOIL
Askarel A	yes	99	1	1.1	Soil	ppm	ASKAREL A	2 STEP ASKAREL A
Askarel A	yes	99	1	1	Wipe	• g/100scm	ASKAREL A	ASKAREL A WIPE
Askarel A	yes	99	1	1.25	Water	ppm	ASKAREL A	ASK A WATER HIGH

Compound	Blank Subtract	%Chlorine	Size Multiplier	Extraction Multiplier	Matrix	Units	Analyte Label	Method Name
DDT	yes	50	1	1.91	Soil	ppm	DDT	DDT SOIL
Toxaphene	yes	68	1	1.54	Soil	ppm	TOXAPHENE	TOXAPHENE SOIL
Chlordane	yes	69	1	1.42	Soil	ppm	CHLORDANE	CHLORDANE SOIL
PCP*	yes	67	1	2.54	Soil	ppm	PCP	PCP SOIL
Trichloro ethylene	yes	81	1	1.00	Soil	ppm	TRICHLOR	TRICHLOR SOIL
Trichloro ethylene	yes	81	10	1.86	Water	ppb	TRICHLOR	TRICHLOR WATER
1,1,1 Tri-chloro ethane	yes	80	10	2.03	Water	ppb	111 TRICHLETHANE	111 TRICHL WATER
Tetrachloro ethylene	yes	86	1	1.00	Soil	ppm	TETRACHLOR	TETRACHLOR SOIL
Tetrachloro ethylene	yes	86	10	1.89	Water	ppb	TETRACHLOR	TETRACHLOR WATER
Methylene Chloride	yes	83	10	13.0	Water	ppm	METHCHLORIDE	METHCHLOR WATER
Vinyl Chloride	yes	57	10	5.36	Water	ppb	VINYLCHLORIDE	VINYLCHLOR WATER
Dichloro ethylene	yes	73	10	3.46	Water	ppb	DICHLORETHYLENE	DICHLOR WATER
Chloride	yes	100	1	1	None	ppm	CHLORIDE	CHLORIDE

* Requires two-step solvent system with filter omitted.

Appendix C: Technical Data

Physical

Size:	w = 9" (230mm) d = 9.5" (240mm) h = 4.25" (110mm)
Weight:	5 lbs. 12 oz. (2.6 kg)
Shipping Weight:	17 lbs. (7.75 kg)
Case:	Painted Aluminum
Keypad:	16 Position UV-Resistant Polyester Membrane Keypad

Electrical

Battery Power:	Internal 8V Gel Cell (3 Days of Operation on Full Charge)
Line Power:	12 W, 115V or 220V Wall Transformer
Charging:	Internal IC Controlled Charging Circuit
Power Management:	Automatic Shut-Off After 15 Minutes of Non-Use With Low-Power Warning and Shut-Off

Digital

Processor:	AM188EM
A/D Converter:	24 Bit Auto-Ranging
Memory:	
Method Storage:	28 Pre-programed Methods + Up to 22 User Defined Methods
Data Storage:	1800 pts

Input

Range:	± 300 mV
Electrode:	BNC Connector (Back Panel)Electrode Type:ORION 96-17B Combination Chloride Electrode

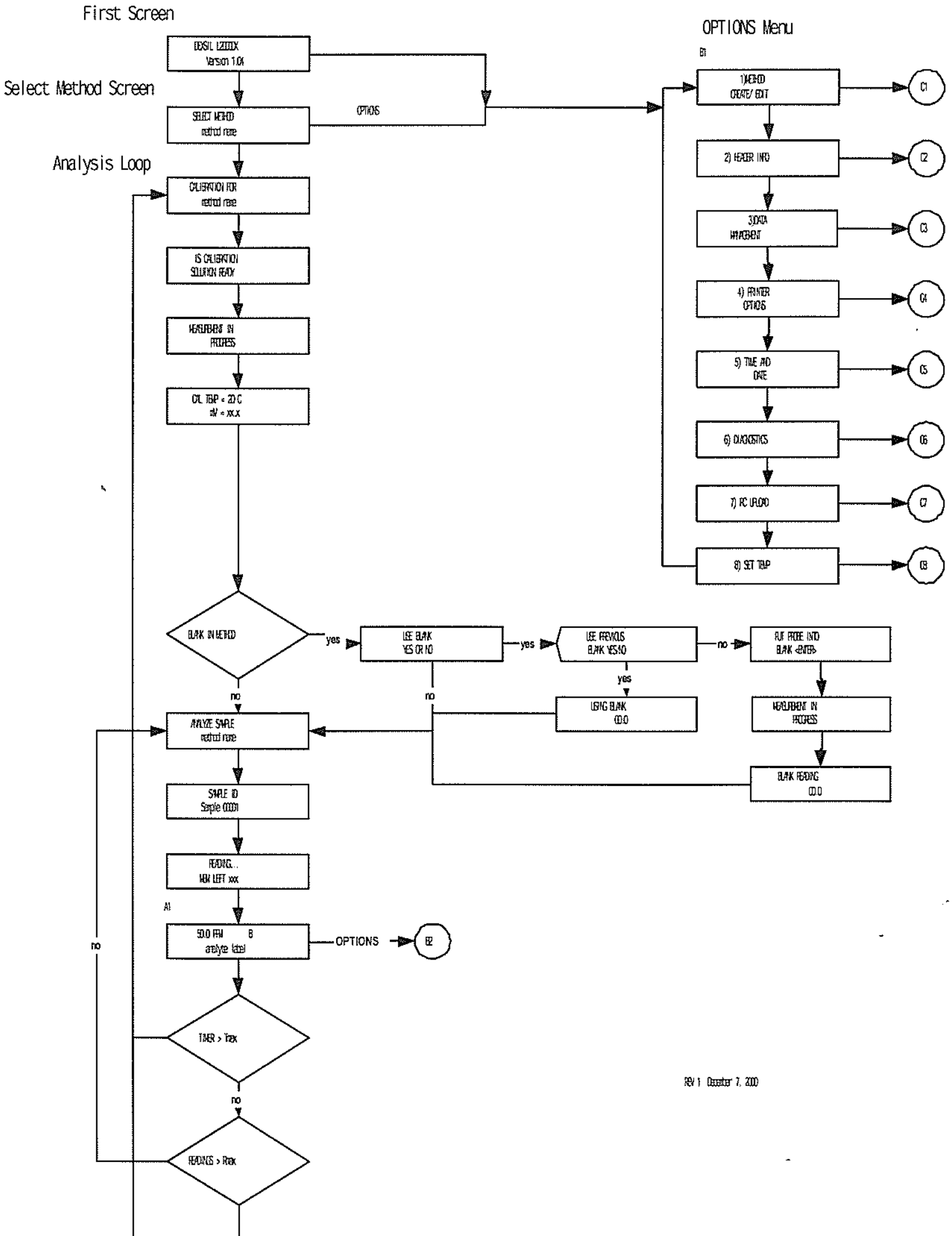
Output

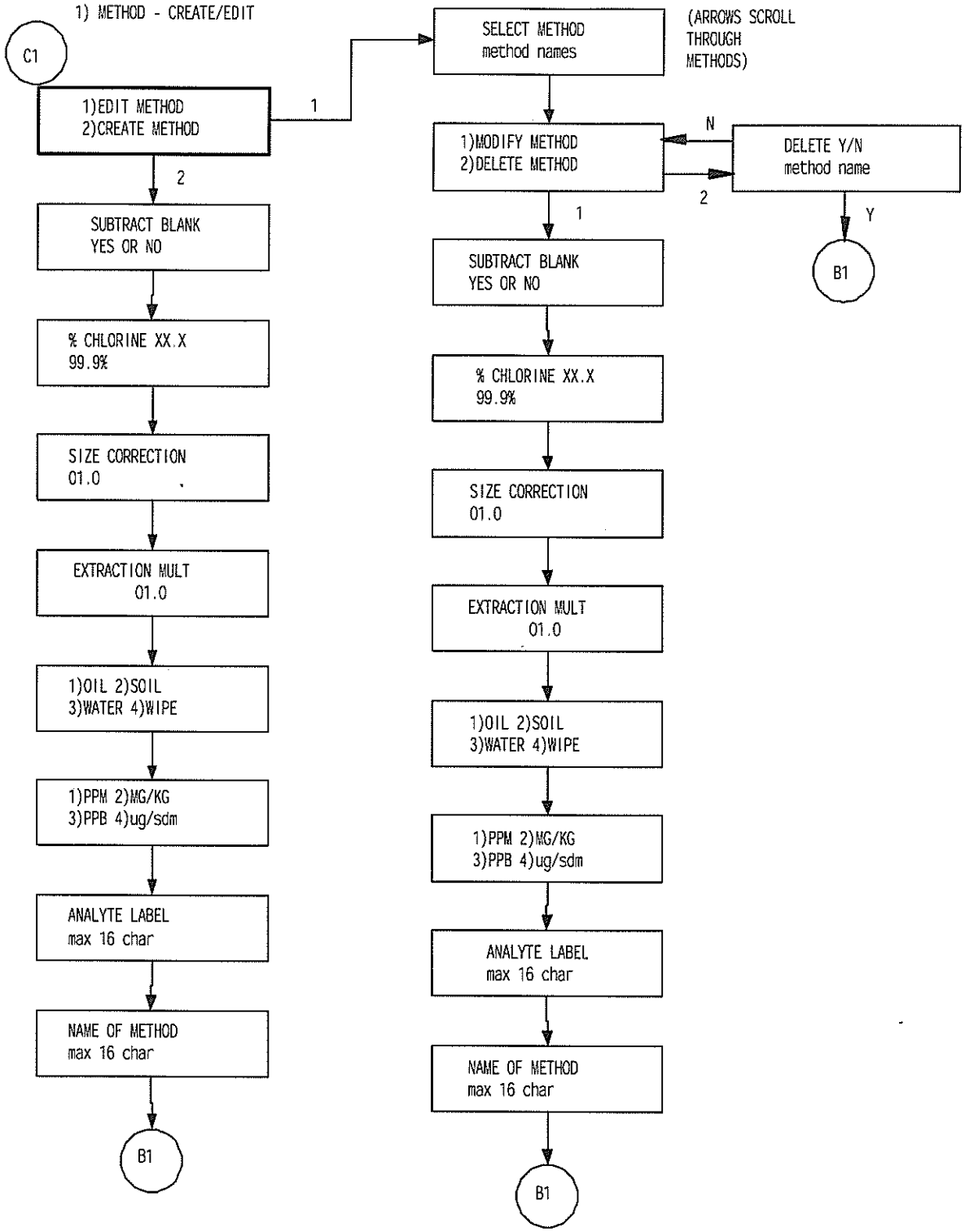
Display:	Backlit, 2 x 16 LCD, 0.32" Character Height
Printer:	40 Character Onboard Thermal Printer (Paper Re-Order PN: LP-THP-01)
External Printers:	Canon BJC-4300, BJC 250, and BJ-200ex; Epson ActionLaser 1400, and Stylus 740, HP DeskJet 870Cse, and LaserJet 4000T
Parallel Printer Port:	25-pin Female Connector (bidirectional)
RS-232:	Data Upload to PC (L2000DX Data Manager required)

Operating Environment

Temperature:	55 °F - 100 °F (13 °C - 38 °C)
Humidity:	85% (non-condensing)
Sunlight:	Do Not Operate With Electrode Exposed to Direct or Indirect Sunlight

Appendix D: Program Flow Chart





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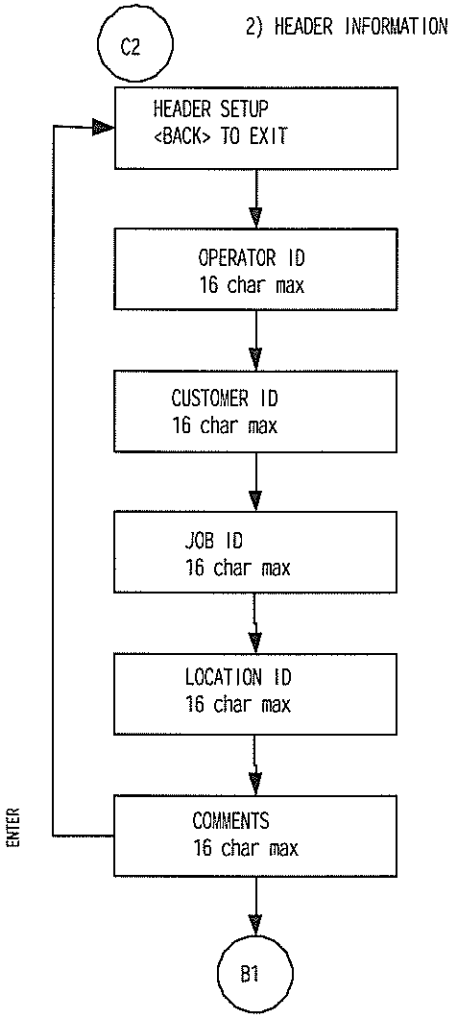
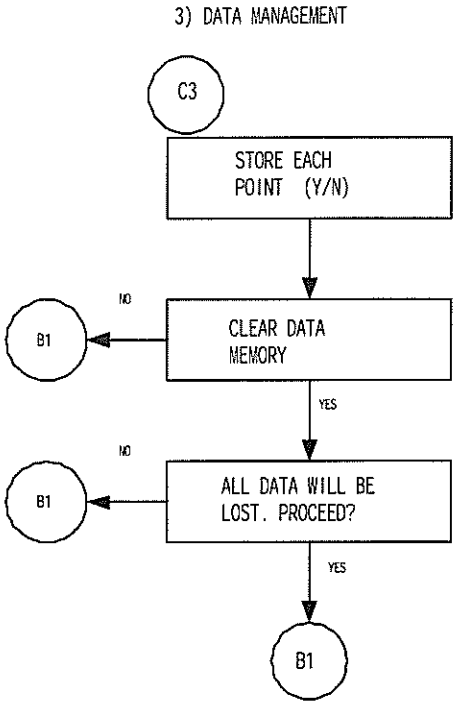
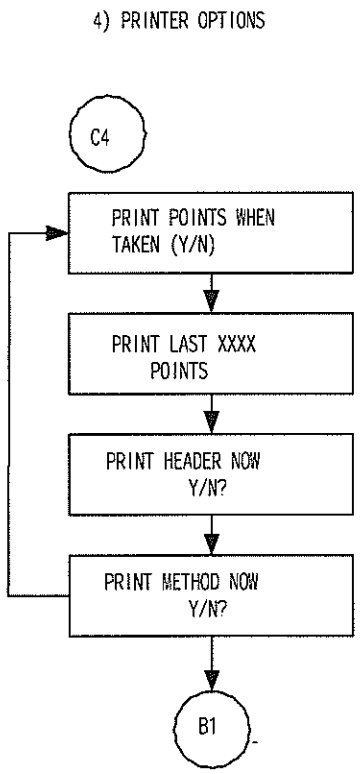
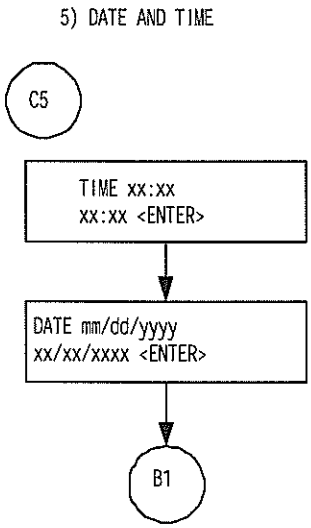


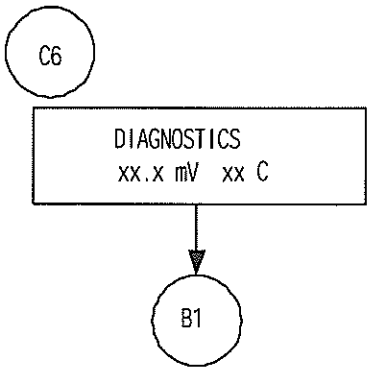




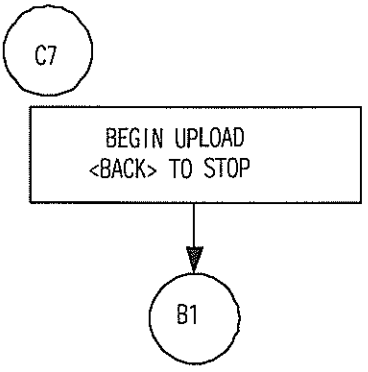


REV 2. December 7, 2000

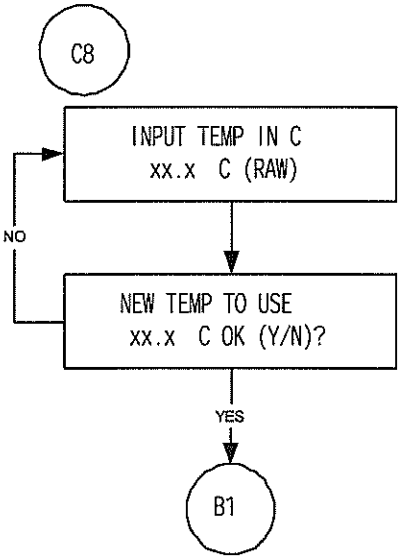
6) DIAGNOSTICS



7) PC UPLOAD



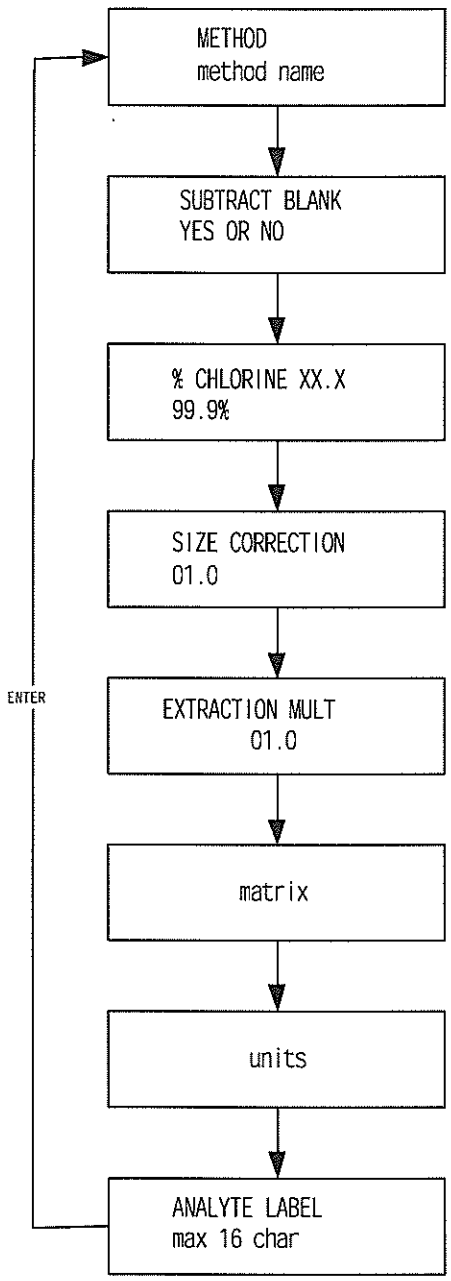
8) SET TEMP



REV 0, December 7, 2000

METHOD INFORMATION

B2



ENTER SCROLLS THROUGH METHOD. BACK RETURNS TO A2.

REV 1 December 7, 2000

DEXSIL CORPORATION
L2000DX ANALYZER
ONE YEAR LIMITED WARRANTY

DEXSIL CORPORATION warrants the L2000DX Analyzer against defects in material or workmanship for a period of one year from the date of purchase. During the warranty period, any product which is determined by DEXSIL to be defective in material or workmanship and returned DEXSIL as specified below, will be, as the exclusive remedy, repaired or replaced at DEXSIL's option.

The cost of repair or replacement is included, shipping costs are not and are to be paid by the customer.

In the event that an L2000DX Analyzer is suspected to be defective contact a DEXSIL representative at the address below to obtain a return authorization:

DEXSIL CORPORATION
ONE HAMDEN PARK DRIVE
HAMDEN, CT 06517
TEL:(203) 288-3509
FAX:(203) 248-6523

Upon return of the unit it will be inspected and a determination will be made as to whether the pro is defective. If defective, arrangements will be made for repair or replacement without charge.

THE WARRANTY SET FORTH ABOVE IS EXCLUSIVE AND NO OTHER WARRANTY, WHETHER WRITTEN OR ORAL, IS EXPRESSED OR IMPLIED. DEXSIL SPECIFICALLY DISCLAIMS THE IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

DEXSIL CORPORATION IS NOT LIABLE FOR INDIRECT OF CONSEQUENTIAL DAMAGES IN CONNECTION WITH THE USE OF THE PRODUCT.

Some states do not allow the exclusion or limitation of incidental or consequential damages, so t above limitation or exclusion may not apply to you.

This Warranty applies only to parts or components which are defective and does not cover repairs necessary due to normal wear, misuse, or accidents.

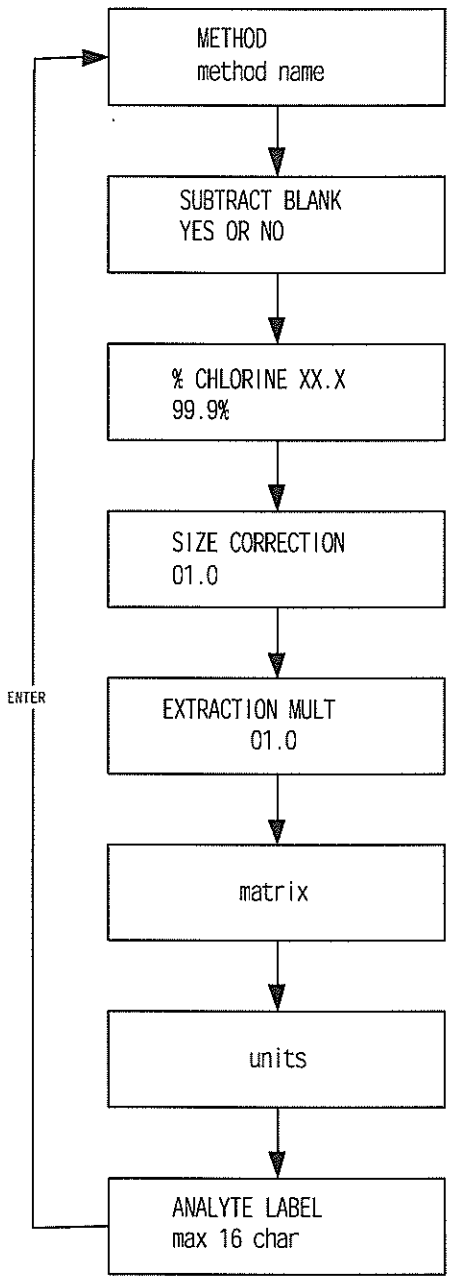
All warranty repairs reimbursable under this warranty must be performed by DEXSIL CORPORATION or its representative using approved replacement parts.

Repairs or attempted repairs by anyone other than a DEXSIL representative are not reimbursable under this warranty. In addition, these unauthorized repair attempts may result in additional malfunctions, the correction of which is not covered by warranty.

This warranty gives you specific legal rights, and you may also have other rights which vary from to state.

METHOD INFORMATION

B2



ENTER SCROLLS THROUGH METHOD. BACK RETURNS TO A2.

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