N00158.AR.000290
NAS WILLOW GROVE
5090.3a

SAMPLING AND ANALYSIS PLAN
for
INTERIM GROUNDWATER MONITORING

SITE 3 - NINTH STREET LANDFILL

NAVAL AIR STATION
JOINT RESERVE BASE
WILLOW GROVE, PENNSYLVANIA

Naval Facilities Engineering Command
Mid-Atlantic
Contract No. N62467-04-D-0055
Contract Task Order 412

MARCH 2008

TETRA TECH NUS, INC.

SAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
March 2008

Site 3 Interim Groundwater Monitoring
NAS JRB Willow Grove

Prepared for:
Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
610-491-9688

Prepared under:
N62467-04-D-0055
CTO 412

Review Signatures:

Tom Johnston 3-4-08
Tom Johnston/CLEAN QAM/Date
Tetra Tech

Russell E Turner 3-5-2008
Russell Turner/Project Manager/Date
Tetra Tech

Approval Signatures:

Curtis A. Frye
Curtis Frye/RPM/Date
NAVFAC BRAC PMO, NE

Lisa Cunningham/RPM/Date
EPA Region 3

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Approval Signatures:

Curtis Frye/RPM/Date
NAVFAC BRAC PMO, NE

Lisa Cunningham/RPM/Date
EPA Region 3

EXECUTIVE SUMMARY

Tetra Tech NUS (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for Interim Groundwater Monitoring (IGWM) at the Naval Air Station Joint Reserve Base (NAS JRB) Willow Grove, Pennsylvania. The principal contaminants associated with Site 3 groundwater are volatile organic compounds (VOCs) and metals.

This SAP contains the technical scope of work and associated sampling plan to perform groundwater level measurements and groundwater and surface water sampling at Site 3. The intent of the IGWM is to update the Remedial Investigation (RI) groundwater data while the Remedial Investigation/Feasibility Study (RI/FS) process is on hold as the Navy and EPA attempt to delineate the extent of landfill cells discovered at Site 3 in 2007.

This SAP outlines the organization, project management and objectives, planned activities, measurement/data acquisition, assessment/oversight, and data review procedures associated with IGWM activities. This SAP specifies requirements for fieldwork related to field operations, the collection of groundwater and surface water samples, measurement of groundwater levels, the collection of samples from monitoring wells at Site 3, and field and laboratory analyses of groundwater and surface water. This SAP includes 37 worksheets required by the Uniform Federal Policy for QAPPs (UFP-QAPP) guidance as the main body of the document. Relevant appendices (A through C) are included behind the worksheets.

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Acronyms

°C	Degrees Centigrade
%R	Percent Recovery
ACT-POC	Activity Point of Contact
ALSI	Analytical Laboratory Services, Inc.
ARS	Air Reserve Station
AWQC	Ambient Water Quality Criteria
CCC	Calibration Check Compound
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Chain of Custody
CSM	Conceptual Site Model
DoD QSM	Department of Defense Quality Systems Manual
DON	Department of the Navy
DQI	Data Quality Indicator
DVM	Data Validation Manager
EICP	Extracted Ion Current Profile
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographical Information System
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HDPE	High Density Polyethylene
HNO_3	Nitric Acid
HSM	Health and Safety Manager
ICP-AES	Inductively Couple Plasma-Atomic Emission Spectroscopy
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IDW	Investigation-Derived Waste
IS	Internal Standard
L	Liter
LCS	Laboratory Control Sample
MDL	Method Detection Limit
mL	Milliliter

Acronyms (Continued)

MPC	Measurement performance criteria
MSD	Matrix Spike Duplicate
MS	Matrix Spike
NA	Not Applicable
NAS JRB	Naval Air Station Joint Reserve Base
NAVFAC	Naval Facilities Engineering Command
NELAP	National Environmental Laboratory Accreditation Program
NFESC	Naval Facilities Engineering Service Center
O&M	Operations and Maintenance
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU	Operating Unit
PADEP	Pennsylvania Department of Environmental Protection
PID	Photoionization Detector
PM	Project Manager
PQOs	Project Quality Objectives
PWC-DET	Public Works Center Detachment
QA	Quality Assurance
QAM	Quality Assurance Manager
QC	Quality Control
QL	Quantitation Limit
RBC	Risk Based Concentration
RI	Remedial Investigation
RF	Response Factor
ROICC	Resident Officer in Charge of Construction
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SSO	Site Safety Officer
TBD	To Be Determined
TCL	Target Compound List

Acronyms (Continued)

TOC	Total Organic Carbon
Tetra Tech	Tetra Tech NUS, Inc.
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USEPA	United States Environmental Protection Agency
U.S. Navy	United States Navy
VOC	Volatile Organic Compound
WS	Worksheet

SAP Worksheet No. 2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Site 3
Operable Unit: NA
Contractor Name: Tetra Tech NUS, Inc.
Contract Number: N62472-04-D-0055
Contract Title: CLEAN
Work Assignment Number (optional): CTO 412

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

2. Identify regulatory program: CERCLA

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Tetra Tech Project Meeting	6/12/2007
NASJRB Willow Grove Team Meeting 15	9/19/2007

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

Title	Date
NA	NA

6. List organizational partners (stakeholders) and connection with lead organization:
EPA (regulatory oversight), PADEP (regulatory oversight), BRAC PMO (property owner), Tetra Tech NUS, Inc. (Navy IRP contractor)

7. Lead organization

NAVFAC

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:

Worksheet 8 omitted. 40-Hour HAZWOPER Training and 8-Hour Refresher documented in HASP.

UFP-QAPP Worksheet No.	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	Not Applicable, Worksheet Completed
2	Table of Contents SAP Identifying Information	Not Applicable, Worksheet Completed
3	Distribution List	Not Applicable, Worksheet Completed
4	Project Personnel Sign-Off Sheet	Not Applicable, Worksheet Completed
Project Organization		
5	Project Organizational Chart	Not Applicable, Worksheet Completed
6	Communication Pathways	Not Applicable, Worksheet Completed
7	Personnel Responsibilities and Qualifications Table	Not Applicable, Worksheet Completed
8	Special Personnel Training Requirements Table	Documentation in HASP
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	Not Applicable, Worksheet Completed
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Not Applicable, Worksheet Completed
11	Site-Specific Project Quality Objectives	Not Applicable, Worksheet Completed
12	Measurement Performance Criteria Table	Not Applicable, Worksheet Completed
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not Applicable, Worksheet Completed
14	Summary of Project Tasks	Not Applicable, Worksheet Completed
15	Reference Limits and Evaluation Table	Not Applicable, Worksheet Completed
16	Project Schedule/Timeline Table	Not Applicable, Worksheet Completed
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Not Applicable, Worksheet Completed
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	Not Applicable, Worksheet Completed
19	Analytical Methods/SOP Requirements Table	Not Applicable, Worksheet Completed
20	Field Quality Control Sample Summary Table	Not Applicable, Worksheet Completed
21	Project Sampling SOP References Table Sampling SOPs	Not Applicable, Worksheet Completed
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Not Applicable, Worksheet Completed
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	Not Applicable, Worksheet Completed
24	Analytical Instrument Calibration Table	Not Applicable, Worksheet Completed
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Not Applicable, Worksheet Completed
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Not Applicable, Worksheet Completed
27	Sample Custody Requirements, Procedures/SOPs Sample Container	Not Applicable, Worksheet Completed

UFP-QAPP Worksheet No.	Required Information	Crosswalk to Related Information
	Identification Example Chain-of-Custody Form and Seal	
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Not Applicable, Worksheet Completed
Data Management Tasks		
29	Project Documents and Records Table	Not Applicable, Worksheet Completed
30	Analytical Services Table Analytical and Data Management SOPs	Not Applicable, Worksheet Completed
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Not Applicable, Worksheet Completed
32	Assessment Findings and Corrective Action Responses Table	Not Applicable, Worksheet Completed
33	QA Management Reports Table	Not Applicable, Worksheet Completed
D. Data Review		
34	Verification (Step I) Process Table	Not Applicable, Worksheet Completed
35	Validation (Steps IIa and IIb) Process Table	Not Applicable, Worksheet Completed
36	Validation (Steps IIa and IIb) Summary Table	Not Applicable, Worksheet Completed
37	Usability Assessment	Not Applicable, Worksheet Completed

SAP Worksheet No. 3 -- Distribution List
(UFP-QAPP Manual Section 2.3.1)

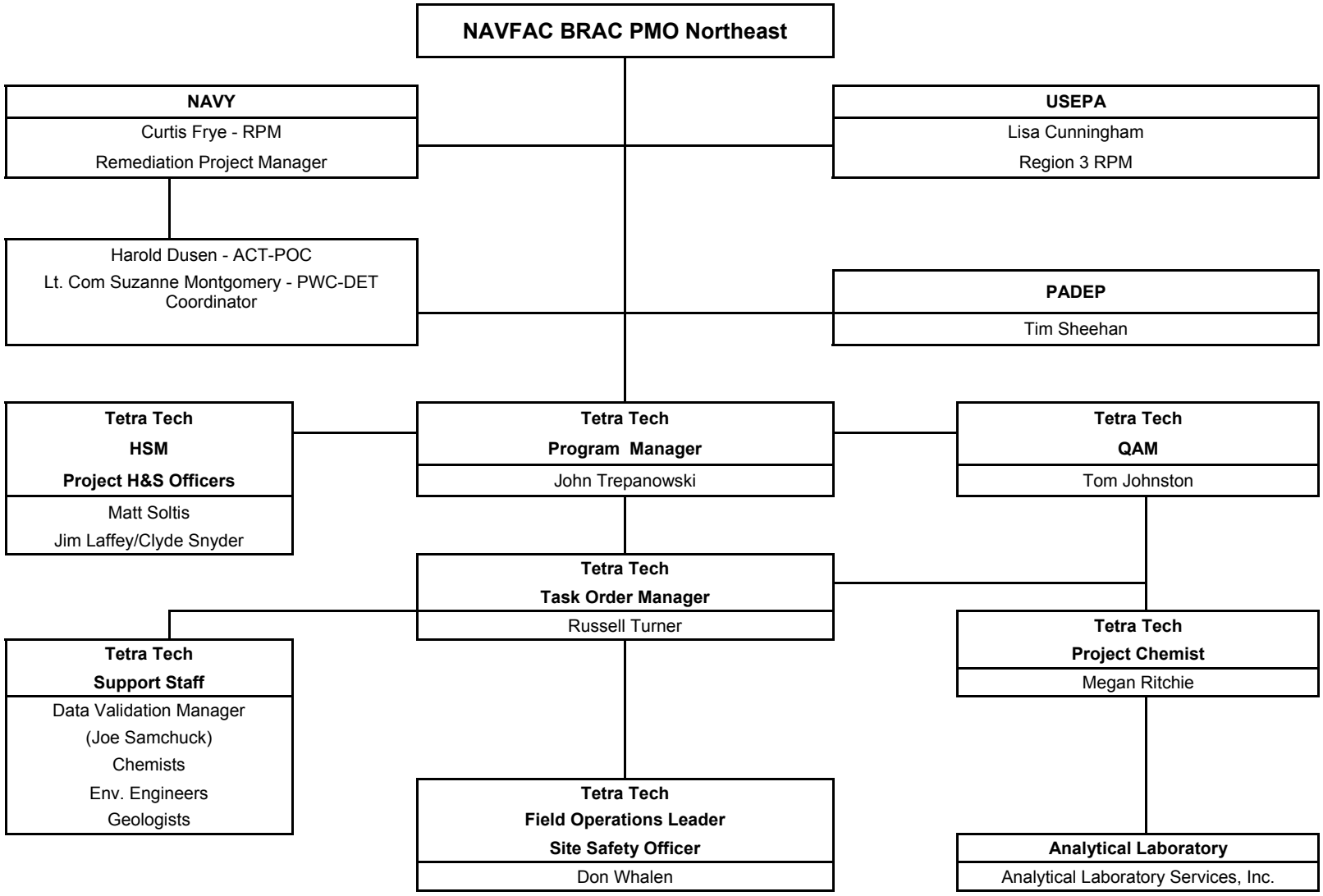
Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Curtis Frye	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	215-897-4914	curtis.frye@navy.mil	NA
Harold Dusen	Environmental Director Activity Point of Contact (ACT-POC)	NAS JRB Willow Grove	215-443-6937	harold.dusen@navy.mil	NA
Lt. Commander Suzanne Montgomery	Coordinator	Public Works Center Detachment (PWC-DET)	215-443-6221	suzanne.montgomery@navy.mil	NA
Gloria Abarca	Facility Contact	NAVFAC Mid-Atlantic	215-773-2106	gloria.abarca@navy.mil	NA
Lisa Cunningham	EPA RPM	EPA Region 3	215-814-3363	cunningham.lisa@epamail.gov	NA
Tim Sheehan	PADEP Project Manager	PADEP	484-250-5721	tsheehan@state.pa.us	NA
Russell Turner	Project Manager (PM)	Tetra Tech	610-491-9688	russ.turner@tetratech.com	NA
Tom Johnston	Quality Assurance Manager (QAM)	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Don Whalen	Field Operations Leader (FOL)/Site Safety Officer(SSO)	Tetra Tech	610-491-9688	don.whalen@tetratech.com	NA
Megan Ritchie	Project Chemist	Tetra Tech	610-491-9688	megan.ritchie@tetratech.com	NA
Scott Brunk	Laboratory Project Manager	Analytical Laboratory Services, Inc. (ALSI)	717-944-5541	sbrunk@analyticallab.com	NA
Dave Barclift`	Technical Manager	NAVFAC	215-897-4913	david.barclift@navy.mil	NA
Bob Lewandowski	Environmental Coordinator	BRAC PMO NE	215-897-4908	robert.f.lewandowski@navy.mil	NA

SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Russell Turner	Tetra Tech PM	610-491-9688			
Don Whalen	Tetra Tech FOL/SSO	610-491-9688			
Tom Johnston	Tetra Tech QAM	412-921-8615			
Joe Samchuck	Tetra Tech Data Validation Manager (DVM)	412-921-8510			
Megan Ritchie	Tetra Tech Project Chemist	610-491-9688			
Scott Brunk	ALSI Laboratory PM	717-944-5541			
Lisa Cunningham	EPA RPM	215-814-3363			
Tim Sheehan	PADEP PM	484-250-5721			

DVM - Data Validation Manager
FOL - Field Operations Leader
PM - Project Manager
QAM - Quality Assurance Manager
SSO - Site Safety Officer
RPM - Remedial Project Manager

SAP Worksheet No. 5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)



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 (timing, pathway to & from, etc.)
Regulatory Agency Review	EPA RPM PADEP PM	Lisa Cunningham Tim Sheehan	215-814-3363 484-250-5721	Review technical documents.
Field Task Modification Request (FTMR)	Tetra Tech FOL	Don Whalen	610-382-1536	Immediately gets approval from Tetra Tech PM; Document via FTMR form.
SAP Amendments	Navy RPM	Cutis Frye	215-897-4914	Immediately informs Tetra Tech PM; Document via FTMR form.
Changes in Schedule	Tetra Tech PM	Russell Turner	610-382-1534	Informs Navy via schedule impact letter as soon as impact is realized
Issues in the field that result in changes in scope of field work	Tetra Tech FOL Tetra Tech PM	Don Whalen Russell Turner	610-382-1536 610-382-1534	FOL informs PM; PM informs RPM; RPM issues scope change if warranted; Scope change to be implemented before work is executed.
Recommendations to stop work and initiate work upon corrective action	Tetra Tech FOL Tetra Tech PM Tetra Tech QAM Tetra Tech Health and Safety Manager (HSM) Navy RPM	Don Whalen Russell Turner Kelly Carper Matt Soltis Curtis Frye	610-382-1536 610-382-1534 412-921-7273 412-921-8912 215-897-4914	Responsible Party immediately informs subcontractors, the Navy, and Project Team.
Analytical data quality issues	ALSI Tetra Tech Project Chemist	Scott Brunk Megan Ritchie	717-944-5541 610-382-1527	Immediately notify Tetra Tech Project Chemist; Notify Data Validation Staff and Tetra Tech PM if necessary

ALSI - Analytical Laboratory Services, Inc.
FOL - Field Operations Leader
FTMR - Field Task Modification Request
HSM - Health and Safety Manager
PM - Project Manager
QAM - Quality Assurance Manager
RPM - Remedial Project Manager

SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Russell Turner	PM	Tetra Tech	Oversees project, financial, schedule, and technical day to day management of the project.	B.A. Natural Sciences, 30 years environmental experience
Curtis Frye	Navy RPM	NAVFAC Mid-Atlantic	Oversees IRP for Navy at NASJRB Willow Grove	
Sherri Eng	Navy QAO	NAVFAC Mid-Atlantic	Performs UFP-QAPP review for Navy Chemists	
Don Whalen	FOL, SSO	Tetra Tech	Supervises, coordinates, and performs field sampling activities	B.A. Geology, M.S. Marine Studies, 17 years environmental experience
Tom Johnston	QAM	Tetra Tech	Reviews SAP, prepares lab scope, coordinates with lab, and data quality review. Ensure Quality aspects of the CLEAN program.	PhD Chemistry, 30 years environmental experience as technical and quality specialist.
Joe Samchuck	DVM	Tetra Tech	Quality assurance of data validation deliverables.	PhD Chemistry, MBA, M.S. Finance, 23 years environmental experience
Megan Ritchie	Project Chemist	Tetra Tech	Coordinates analyses with lab chemists, ensures the scope is followed, QA data packages, communicates with Tetra Tech staff.	B.S. Biology/Environmental Studies, 10 years environmental experience
Matt Soltis	HSM	Tetra Tech	Oversees CLEAN Program Health and Safety Program	B.S. Industrial Safety Sciences, 24 years of environmental experience

CLEAN - Comprehensive Long-term Environmental Action Navy
HSM - Health and Safety Manager
QAM - Quality Assurance Manager
DVM - Data Validation Manager
PM - Project Manager
SSO - Site Safety Officer
FOL - Field Operations Leader
QA - Quality Assurance
Tetra Tech - Tetra Tech NUS, Inc.

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

(UFP-QAPP Manual Section 2.4.4)

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

No special training is required for this activity. Certification of 40-Hour HAZWOPER and 8-Hour Refresher Training is located in the site-specific Health and Safety Plan (HASP).

Analytical Laboratory Services, Inc (ALSI) has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program and described in the Department of Defense Quality Systems Manual (DoD QSM) (January 2006) and is additionally certified by the National Environmental Laboratory Accreditation Program (NELAP), which is the recognized certifying authority for the state of Pennsylvania. ALSI's last Navy audit was conducted on November 28 through 30, 2007. The Navy determined that ALSI's Corrective Action Plan (CAP) was acceptable, and ALSI is presently in the process of submitting the documentation supporting the CAP. The Navy has extended ALSI's certification through February of 2008, until the new certification is issued. The Navy certification letter is included in Appendix B.

SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 3 IGWM Projected Date(s) of Sampling: 03/10/2008 - 03/14/2008 and 09/15/08 - 09/19/08 Project Manager: Russell Turner	Site Name: Site 3 Site Location: Willow Grove, Pennsylvania

Date of Session: unknown
Scoping Session Purpose: Preliminary Scoping Meeting

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Russell Turner	PM	Tetra Tech	(610) 382-1534	russ.turner@tetratech.com	Management
Curtis Frye	Navy RPM	Navy	(215) 897-4914	curtis.frye@navy.mil	Management

Comments/Decisions: Defined general sampling approach, number of samples, sample analyses.

Action Items: Start Worksheets and text for SAP.

Consensus Decisions: 1,4-dioxane and hex chrome will be included in sample analysis. Include off-Base well in sampling plan.

Project Name: Site 3 IGWM Projected Date(s) of Sampling: 03/10/2008 - 03/14/2008 and 09/15/08 - 09/19/08 Project Manager: Russell Turner	Site Name: Site 3 Site Location: NAS JRB Willow Grove

Date of Session: 6/12/2007
Scoping Session Purpose: Preliminary Scoping Plan for Navy/EPA review

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Russell Turner	PM	Tetra Tech	610-491-9688	russ.turner@tetratech.com	Management
Don Whalen	FOL	Tetra Tech	610-491-9688	don.whalen@tetratech.com	Task Management
Megan Ritchie	Project Chemist	Tetra Tech	610-491-9688	megan.ritchie@tetratech.com	QA/QC

Comments/Decisions: Discussed project activities. Distribute assignments to team members.

Action Items: Complete Worksheets and text for SAP and implement review process.

Consensus Decisions: Worksheets will be completed by Megan Ritchie (project chemist) and then reviewed by technical personnel and QA Manager, Tom Johnston. Don Whalen will coordinate generation of site maps and well locations with the Geographical Information System (GIS) department.

Project Name: NAS JRB Willow Grove Site 3 Projected Date(s) of Sampling: 03/10/2008 - 03/14/2008 and 09/15/08 - 09/19/08 Project Manager: Russell Turner	Site Name: Site 3 Site Location: Willow Grove, Pennsylvania

Date of Session: 9/19/2007
Scoping Session Purpose: Update team on monitoring plan / encourage input

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Russell Turner	PM	Tetra Tech	(610) 382-1534	russ.turner@tetratech.com	Management

Project Name: NAS JRB Willow Grove Site 3 Projected Date(s) of Sampling: 03/10/2008 - 03/14/2008 and 09/15/08 - 09/19/08 Project Manager: Russell Turner	Site Name: Site 3 Site Location: Willow Grove, Pennsylvania

Date of Session: 9/19/2007
Scoping Session Purpose: Update team on monitoring plan / encourage input

Curtis Frye	Navy RPM	Navy	(215) 897-4914	curtis.frye@navy.mil	Management
Kevin Kilmartin	Geologist	Tetra Tech	(610) 382-1173	kevin.kilmartin@tetratech.com	Lead Geologist
Megan Ritchie	Project Chemist	Tetra Tech	(610) 382-1527	megan.ritchie@tetratech.com	Chemist
Linda Watson	Risk Assessor	EPA		watson.linda@epamail.gov	Regulatory Oversight
Lisa Cunningham	BRAC PMO	EPA	(215) 814-3363	cunningham.lisa@epamail.gov	management
Bernice Pasquini	Hydrogeologist	EPA	(215) 814-3326	pasquini.bernice@epamail.gov	Regulatory Oversight
Bob Lewandowski	Base Coordinator	Navy	(215) 897-4908	robert.lewandowski@navy.mil	Management
Dave Barclift	Technical Manager	Navy	(215) 897-4913	david.barclift@navy.mil	Management
Jessica Kasmari	Hydrogeologist	PADEP	(484) 250-5724		Regulatory Oversight

Comments/Decisions: Refined sampling plan objectives and procedures..

Action Items: Complete Worksheets and text for SAP and implement review process.

Consensus Decisions:

SAP Worksheet No. 10 -- Problem Definition
(UFP-QAPP Manual Section 2.5.2)

SITE BACKGROUND

NAS JRB Willow Grove, Pennsylvania is located in Horsham Township, Montgomery County in southeastern Pennsylvania, approximately 20 miles north of the city of Philadelphia. The Ninth Street Landfill occupies about 9 acres immediately north of Ninth Street along the western boundary of NAS JRB Willow Grove (Figure 10-1). Figure 10-2 shows the site features.

The Ninth Street Landfill was active from 1960 to 1967. The site was used for landfilling activities by the Public works Department and also as an open disposal ground by various operations at the Base. The landfill method consisted of burning the refuse and burying the residue in trenches. Reportedly, the waste disposed of in the landfill consisted mainly of general refuse, but also included paint wastes; paint stripper; TCE; sewage sludge; petroleum, oils and lubricants; and industrial pretreatment plant sludge. After closure of the landfill, a salvage yard operated in the eastern portion of the site. Between 1980 and 1983, clean fill consisting of construction debris was deposited in the area southwest of the stormwater retention basin.

PHYSICAL SETTING

Surface Features

The Air Station is comprised of flat to slightly rolling terrain and is generally bounded by State Route 611 to the east, State Route 463 to the southwest, and Keith Valley Road to the north. The Ninth Street Landfill is located in an undeveloped area between Ninth Street and Dawes Road. The ground surface is well vegetated with a mixture of grass and woody vegetation. The ground surface slopes north toward a wetland area, intermittent stream, and storm water retention basin located approximately 200 feet from what historical information indicates was the northern limit of waste disposal. Surface recreational facilities including a pavilion, playground, and baseball diamond are located in the northeastern portion of the site.

Geology

Regional Geology

NASJRB Willow Grove is located within the Triassic Basin of southeastern Pennsylvania. The bedrock underlying the NASJRB consists of the middle arkose member of the Late Triassic age Stockton Formation. The Stockton Formation locally is about 5,000 feet thick and is unconformably underlain by Ordovician to PreCambrian age basement rocks. Previous environmental investigations indicate that the top of bedrock at the air station is generally found in the range of 5 feet to 25 feet below ground surface (bgs).

The Stockton Formation is composed of fine- to coarse-grained arkosic sandstones and conglomerates that are interbedded with finer grained shales and siltstones. Bedding is very irregular throughout the Stockton Formation, although coarse-grained units commonly overlie fine-grained units. Based principally on dominant grain size and lithology, the Stockton Formation is divided into lower, middle, and upper members.

The middle member of the Stockton Formation is approximately 4,200 feet thick and consists of fine- to medium-grained arkosic sandstone interbedded with shale. Beds of shale and siltstone are common in the upper portion of the member, and coarser grained units are more common in the lower portion of the member. The rocks of the middle member are well sorted and weakly cemented, which creates a relatively high porosity compared to the lower and upper members of the formation (Rima et al., 1962). The middle member of the Stockton Formation typically weathers to a depth of 15 to 35 feet.

Geology of Site 3

Soil borings and monitoring well boreholes drilled in the vicinity of the Site 3 typically encountered the top of bedrock at a depth of 10 feet to 15 feet below the ground surface. The overburden consists of silty to sandy clay that grades downward to weathered siltstone or shale. The overburden is generally thinnest (3 feet to 5 feet) in the northwest portion of the site.

The maximum depth of the monitoring well boreholes at Site 3 is 225 feet. The bedrock to this depth typically consists of alternating sequences of sandstone, siltstone, and shale. In general, the lithologic units at this site are rather thick and fairly continuous, although it is not unusual to encounter local, finer-grained lenses within a generally coarser matrix within any particular unit.

Hydrogeology

Regional Hydrogeology

The major source of groundwater in the vicinity of NASJRB Willow Grove is the fractured bedrock of the Stockton Formation. These rocks form a multi-aquifer system of relatively discrete water-bearing zones separated by less permeable zones. Transmissivity and groundwater movement within water-bearing zones are greater parallel to bedding than across bedding. Groundwater can generally be found between 5 feet and 25 feet bgs.

Groundwater within the Stockton Formation locally occurs under both unconfined and confined conditions. The unconfined conditions generally extend to a subsurface depth of about 75 to 100 feet, depending on the local lithologies. Confined conditions are generally encountered below a depth of about 150 feet. A semi-confined or transitional aquifer lies between the unconfined and confined aquifers. Vertical or nearly vertical fractures that cut across bedding and the weathering of various beds are expected to permit varying degrees of leakage between individual water-bearing zones, particularly at shallower depths.

Although significant amounts of groundwater may be held in storage within the primary porosity of the fine- to medium-grained sandstones, groundwater migration is chiefly through the secondary porosity created by fractures and joints and along bedding-plane partings. The finer grained shale and siltstone beds typically have very low permeabilities. In addition, fractures and joints are typically not as well developed in these finer grained beds. Consequently, the shale and siltstone units often act as confining layers to groundwater flow.

Hydrogeology of Site 3

The occurrence and distribution of groundwater within the bedrock of Site 3 is similar to that typically reported for aquifers within the Triassic Basin. During the RI drilling, the bulk of the groundwater entered the boreholes through discrete water-entry zones. Subsequent analyses of the drilling logs, borehole video tape (when present), and borehole geophysical logs revealed that the discrete water-entry zones were either bedding-plane partings at lithologic contacts or fractures within a relatively homogeneous lithologic unit. Typically, both types of water-entry zones were present within any particular borehole.

The hydraulic head data collected during the RI indicate that two hydrogeologic units, an upper unconfined zone and a deeper semi-confined to confined zone, exist beneath Site 3. The monitoring wells at the site are completed in both of these groundwater zones, based on the vertical distribution of hydraulic head. Semi-confined to confined conditions are present in monitoring wells 03MW01I, 03MW02I, 03MW03SI, 03MW03I, and 03MW04I; monitoring wells 03MW03I and 03MW04I are typically flowing. The confining conditions at any monitoring well cluster location are not created by the presence of any single confining layer or geologic unit and cannot be predicted solely by well depth, but are related to the surface elevation of that cluster location, the elevation of the recharge area for the hydrogeologic unit being monitored, and the subsurface depth (or elevation) of the well screen.

Groundwater at the water table and within the unconfined groundwater zone beneath Site 3 flows in an overall northwestward direction. The configuration of the water table appears to be a subdued reflection

of the surface topography, as is typical for this area. Groundwater in the confined zone flows in a generally northward direction.

PREVIOUS GROUNDWATER INVESTIGATIONS

Site Inspection

The Site Inspection (SI) was completed in 1990 (EA Engineering, May 1990). Seven monitoring wells were installed and sampled for full TCL/TAL parameters, total organic carbon, and total petroleum hydrocarbons. Four of the wells, 03MW01S, 03MW02S, 03MW03S, and 03MW04S, were shallow, 4-inch wells designed to intercept the water table, and three of the wells, 03MW01SI, 03MW03SI, and 03MW04SI, were deeper, 2-inch diameter wells. The construction details for the wells are listed in Table 10-1, and Figure 10-2 shows the monitoring well locations. The monitoring wells were sampled in three rounds between June and December 1989. The sample results revealed PCE in all but one of the wells at concentrations up to 84 ug/l. PCE was detected in the upgradient wells 03MW01S and 03MW01I at maximum concentrations of 43 ug/l and 54 ug/l, respectively. Trichloroethene (TCE) and 1,1,2,2-trichloroethane were also detected in 03MW3S at concentrations of 6 ug/l and 73 ug/l, respectively.

Remedial Investigation

During the Phase I remedial investigation, a total of four new bedrock monitoring wells were installed: 03MW01I, 03MW03I, 03MW04I, and 03MW05S. 03MW05S was installed upgradient of the 03MW01 well cluster to investigate the upgradient PCE contamination. 03MW01I, 03MW03I, and 03MW04I were installed in order to investigate groundwater quality in the deeper portion of the aquifer at these well cluster locations. The construction details for the wells are listed in Table 10-1. The four new and seven existing monitoring wells were sampled for full TCL/TAL parameters in September 1991. PCE was detected in all but one of the shallow wells at concentrations up to 61 ug/l, and in all of the deeper wells at concentrations up to 56 ug/l. The new upgradient well, 03MW05S, contained 7 mg/l PCE. There were no semivolatiles detected at levels exceeding reference criteria during Phase I.

During the Phase II Remedial Investigation, a total of seven new monitoring wells were installed: 03MW02SI, 03MW02I, 03MW05I, 03MW06S, 03MW06SI, 03MW06I, and 03MW07S. The construction details for the wells are listed in Table 10-1. The 03MW06 cluster was installed upgradient of the 03MW05 cluster in order to investigate the PCE contamination further upgradient of the landfill. Samples were collected from all of the new and existing wells in June 1997 and analyzed for full TCL/TAL parameters. PCE was detected in eleven wells at concentrations ranging from 5 ug/l to 39 ug/l. The upgradient wells 03MW06S and 03MW06I contained PCE at 29 ug/l and 39/ug/l, respectively, which indicated that there was a source of PCE further upgradient of these wells. Only one semivolatile compound, bis(2-ethylhexyl) phthalate, was detected during the Phase II sampling round, in 03MW05I (6J ug/l) and 03MW06SI (11 ug/l). These concentrations exceeded reference criteria.

During the Phase II follow-on activities, two new wells, 03MW08S and 03MW08D, were installed upgradient of the 03MW06 cluster. All of the existing wells (except 03MW03I, which was obstructed) were sampled for TCL VOCs in February 2006. The two new wells were also sampled for SVOCs. PCE was detected in sixteen wells at concentrations ranging from 0.23 ug/l to 14 ug/l (Table 10-2). The new wells, 03MW08S and 03MW08D contained PCE at ND and 0.78 ug/l, respectively. 03MW08D also contained 1,1-TCA (3.0 ug/l); TCE (1.5 ug/L); 1,1DCA (5.9 ug/l); 1,1-DCE (2.5 ug/l); and cis-1,2-DCE (0.90J ug/l). The non-detection of PCE in the shallow well indicates that this well defines the upgradient limit of the Site 3 PCE plume. The contaminants in the deeper well are interpreted to represent the downgradient edge of the Site 5 groundwater contaminant plume. Site 5 is located approximately 1000 feet southwest (upgradient) of the 03MW08 well cluster. There were no semivolatiles detected in 03MW08S or 03MW08D, the only wells sampled for semivolatiles.

Two off-base wells located immediately downgradient from the site, on the adjacent Commonwealth Corporate Center property, were geophysically logged and sampled for VOCs in 1998. The wells were a 73-foot deep former golf course irrigation well (located on Lot 1 of the property) and a 150-foot deep monitoring well (located on Lot 8 of the property). The sample results showed PCE at 15 ug/l in the former irrigation well and no detections in the monitoring well.

TABLE 10-1
MONITORING WELL CONSTRUCTION SUMMARY
SITE 3 - NINTH STREET LANDFILL
NASJRB, WILLOW GROVE, PENNSYLVANIA

Monitoring Well	Well Diameter (inches)	Screen Interval (ft bgs)	Sand-Pack Interval (ft bgs)	Elevation TOC (ft amsl)
Site Inspection				
03MW01S	4	12 - 32	10 - 33	347.49
03MS01SI	2	60 - 80	57 - 81	346.98
03MW02S	4	4.5 - 22.5	4 - 23	326.15
03MW03S	4	5 - 20	4 - 22	312.96
03MW03SI	2	60 - 80	58 - 81	313.75
03MW04S	4	5 - 35	4 - 37	324.49
03MW04SI	2	60 - 80	53 - 81	323.87
Phase I RI				
03MW01I	4	159 - 179	156 - 179.5	347.50
03MW03I	4	148.5 - 168.5	145 - 171	315.00
03MW04I	4	148 - 168	145 - 169	324.85
03MW05S	4	17 - 37	15 - 37.5	362.90
Initial Phase II RI				
03MW02SI	2	55 - 65	52 - 70	326.33
03MW02I	2	134 - 144	130 - 144	326.38
03MW05I	2	82 - 92	78 - 96	390.93
03MW06S	2	26 - 36	25 - 38	367.00
03MW06SI	2	75 - 85	72 - 85	366.86
03MW06I	2	140 - 150	136 - 150	366.82
03MW07S	2	34 - 44	30.5 - 50	324.90
Phase II RI Follow-on Activities				
03MW08S	2	38 - 68	33 -- 73	365.59
03MW08D	2	163 - 173	158 - 178	365.63

The available analytical data suggest that the concentrations of PCE in the Site 3 groundwater are decreasing with time. Table 10-2 displays the PCE concentrations detected in the Site 3 monitoring wells since the Phase I RI. Of the eleven wells installed during SI and the Phase I field activities, six wells, including the five most highly contaminated wells, have shown consistent decreasing trends in PCE concentrations: 03MW01S (from 15 ug/l to 0.9 ug/l), 03MW04S (from 61 ug/l to 8.8 ug/l), 03MW05S (from 7 ug/l to 0.67 ug/l), 03MW01SI (from 29 ug/l to 4.9 ug/l), 03MW03SI (from 35 ug/l to 12 ug/l), and 03MW04SI (from 56 ug/l to 10 ug/l). Of the remaining five wells, one (03MW04S) has shown an increase in PCE concentration (8 ug/l to 14 ug/l); one (03MW03I) has shown little change; two (03MW01I and 03MW04I) were nondetect until the most recent sampling round, in which they have shown trace quantities; and one (03MW02S) has remained non-detect for all three sampling rounds. Of the seven wells installed during the initial Phase II field activities, four (03MW06S, 03MW02SI, 03MW02I, and 03MW06I) have shown decreases in PCE concentrations, two (03MW07S and 03MW05I) have shown slight increases, and one (03MW06SI) has remained non-detect. The consistency and the magnitude of the decreases in PCE concentration among the most highly contaminated monitoring wells make it unlikely that they are attributable to fluctuations in the data, and suggests that the original source (or sources) of the contamination has been depleted to the extent that it is no longer significantly contributing PCE to the contaminant plume.

TABLE10-2
HISTORICAL PCE CONCENTRATIONS IN GROUNDWATER (UG/L)
NINTH STREET LANDFILL
NASJRB WILLOW GROVE, PENNSYLVANIA

WELL	SAMPLING DATE		
	SEPTEMBER 1991	JUNE 1997	FEBRUARY 2006
SHALLOW WELLS			
03MW01S	15	6J	0.9
03MW02S	ND	ND	ND
03MW03S	8	5J	14
03MW04S	61	21	9.1
03MW05S	7	2J	0.67J
03MW06S	NA	29	1.3
03MW07S	NA	ND	1.4
SHALLOW-INTERMEDIATE DEPTH WELLS			
03MW01SI	29	---	4.9
03MW02SI	NA	6J	1.9
03MW03SI	35	18	12
03MW04SI	56	20	10
03MW05I	NA	ND	0.70J
03MW06SI	NA	ND	ND
03MW08S	NA	NA	ND
INTERMEDIATE DEPTH WELLS			
03MW01I	ND	ND	0.23J
03MW03I	5	6J	---
03MW04I	ND	ND	0.52J
03MW02I	NA	8J	2.4
03MW06I	NA	39	0.79J
03MW08D	NA	NA	0.78J

J = Value is estimated.
ND = Non detect.
NA = Not Available. Well had not been installed at time of sampling round.

The problem to be addressed by the project:

The RI/FS process is currently on hold as the Navy and EPA attempt to delineate the extent of buried waste that was discovered at Site 3 in 2007. This Interim Groundwater Monitoring (IGWM) will serve as supplemental data to assist in finalizing the RI report and preparing the FS.

The off-Base well (Lot 1 Irrigation well) is a flowing well that discharges to surface water. Historical sample results indicate that this well is contaminated with PCE, however, there have been no samples collected from the stream into which the well discharges.

Chromium was detected in unfiltered groundwater samples from six monitoring wells during the RI (see Table 10-3). These chromium detections contributed to the human health risk from site groundwater based on the conservative assumption that the chromium was present in the hexavalent form. If present in the trivalent form, the chromium would not significantly contribute to the risk. Chromium speciation data is not available to determine the actual valence of the chromium.

TABLE10-3
HISTORICAL TOTAL CHROMIUM CONCENTRATIONS IN GROUNDWATER (UG/L)
NINTH STREET LANDFILL
NASJRB WILLOW GROVE, PENNSYLVANIA

WELL	SAMPLING DATE		
	SEPTEMBER 1991	JUNE 1997	FEBRUARY 2006
SHALLOW WELLS			
03MW01S	13B	23.3	---
03MW02S	ND	ND	---
03MW03S	8B	ND	---
03MW04S	ND	ND	---
03MW05S	17B	7.7	---
03MW06S	---	10.7K	---
03MW07S	---	ND	---
SHALLOW-INTERMEDIATE DEPTH WELLS			
03MW01SI	13B	ND	---
03MW02SI	NA	ND	---
03MW03SI	ND	ND	---
03MW04SI	12B	ND	---
03MW05I	---	53.3	---
03MW06SI	---	36.9	---
03MW08S	---	ND	---
INTERMEDIATE DEPTH WELLS			
03MW01I	ND	ND	---
03MW03I	14B	ND	---
03MW04I	ND	ND	---
03MW02I	---	ND	---
03MW06I	---	90.9	---
03MW08D	---	ND	---

B = Result is considered an artifact of blank contamination.
K = Result is considered biased high.
ND = Non detect

The environmental questions being asked:

- What is the current extent of groundwater contamination at Site 3? Have contaminant concentrations changed since the completion of the RI?
- Is the site groundwater affecting off-Base, downgradient groundwater?
- Is the site groundwater affecting off-Base, downgradient surface water?
- What is the speciation of the chromium that was detected in unfiltered groundwater samples during the RI?

Observations from any site reconnaissance reports: N/A

The possible classes of contaminants and the affected matrices:
VOCs and Inorganics (including mercury and hexavalent chromium) in groundwater and surface water.

The rationale for inclusion of chemical and non-chemical analyses:
The contaminants of concern (COCs) identified by the RI risk assessment consisted of VOCs and metals. Hexavalent chromium was included because the total chromium data in the RI report exceeded the hexavalent chromium screening criteria for wells during the RI. Chromium was detected in unfiltered samples from six monitoring wells during the RI: MW-1S, MW-5S, MW-5I, MW-6S, MW-6SI, and MW-6I. The hexavalent chromium data from this event will be used to determine the chromium speciation at the site. Samples of site groundwater have never been analyzed for 1,4-dioxane. This compound has been detected in groundwater underlying Site 5, which is upgradient of Site 3. The downgradient edge of the Site 5 plume has been detected in 03MW08D, the furthest upgradient Site 3 well.

Information concerning various environmental indicators:
Region 3 RBCs and Region 3 BTAG Freshwater Screening Levels will be used to determine required detection limits for the laboratory analyses. The laboratory detection limits must be sufficiently low so that the data collected during the Site 3 IGWM is comparable to the RI data. These levels will not be used to screen the groundwater or surface water data for this activity. However, the data must have adequate detection limits in the event that this data will be used to finalize the RI and to prepare the FS.

Project decision conditions (If..., then...@ statements):
If any compound in a groundwater sample is found at a concentration greater than its PAL, then the project team will meet to discuss possible future actions.

If hexavalent chromium is not detected in the groundwater samples, then the chromium risks determined during the RI will be reevaluated.

If surface water concentrations indicate that the surface water has been impacted by the groundwater from Site 3, then surface water impacts will be considered in the remedial action alternatives in the FS. If surface water concentrations indicate that the surface water has not been impacted by the groundwater from Site 3, then no action is required.

SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

Who will use the data?
Navy and Tetra Tech NUS, specifically the engineer who will prepare the FS.

What will the data be used for?
The data will be used to update the RI groundwater data while the RI/FS process is on hold as the Navy and EPA attempt to delineate the extent of the landfill cells discovered at Site 3 in 2007. It is anticipated that the data will be used in remedial action alternative development and selection in the FS.

What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?

Fixed-based laboratory data will be used to characterize groundwater and surface water contamination. Groundwater and surface water will be analyzed for TCL VOCs, TAL total and dissolved metals, 1,4-dioxane, and hexavalent chromium.

Matrix: Groundwater and Surface Water

How "good" do the data need to be in order to support the environmental decision?
The analytical data will get the fullest level of QC and documentation. Data will undergo 100% validation. The laboratory must hold a current NELAP accreditation in Pennsylvania and comply with the requirements of NFESC in analytical results reporting and QA/QC.

How much data are needed (number of samples for each analytical group, matrix, and concentration)?

Two semi-annual rounds of groundwater and surface water data will be collected. Based on the result of Remedial Investigation (RI), 100% of RI groundwater monitoring wells will be sampled to represent the entire plume. For each round, one groundwater sample will be collected from each of twenty (20) monitoring wells, one (1) off-Base well (golf course) for TCL VOCs, total metals, dissolved metals, mercury, and 1,4-dioxane. Eight (8) of the 21 monitoring well locations will be analyzed for hexavalent chromium. These eight wells will consist of the six wells in which chromium was detected during the RI and two "control" wells. Two (2) surface water samples will be collected for TCL VOCs, metals, mercury, and 1,4-dioxane. Concentrations are expected to be low to medium.

Where, when, and how should the data be collected/generated?
All data will be generated at a fixed-base laboratory upon receipt of the groundwater and surface water samples generated in the field at Site 3. Groundwater samples will be collected using the low-flow sampling procedure where possible. The flowing off-Base irrigation well and surface water samples will be collected by directly filling the sample bottles. See Worksheet 18.

Site maps and figures detailing the site location, boundaries, and sampling locations are included in Appendix C of this SAP.

SAP Worksheet No. 12 -- Measurement Performance Criteria Table note matrix in table entry
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table - Field QC Samples

QC Sample	Analytical Group[1]	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Trip Blank	VOC	One per cooler of VOC samples shipped to laboratory	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	S & A
Field Duplicate	VOC, Metals	One per 10 samples	Precision	Values > 5X QL: ±30% Values < 5X QL: absolute difference of values must be within 2X QL	S & A
Field/Rinsate Blanks	VOC, Metals	One per 20 samples per sampling equipment	Bias / Contamination	No target analytes ≥QL; with the exception of common field/laboratory contaminants	S
Temperature Blank	VOC, Metals	One per cooler	Accuracy / Representativeness	Between 2 and 6 °C	S

[1]If information varies within an analytical group, separate by individual analyte.

% - Percent
°C - Degrees Celsius
QC - Quality Control
QL - Quantitation Limit
VOC - Volatile Organic Compound

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
RI Phase I Analytical Data	RI Report for Sites 1, 2, 3, and 5, Naval Air Station Willow Grove, Pennsylvania	Halliburton NUS Environmental Corporation, February 1993	Secondary data will be compared to current concentrations to determine if the RI risk assessment accurately reflects current conditions.	1,4-dioxane and MTBE data not available. Validation criteria for the secondary data were the same as will be used for the current data. Secondary data meet the required measurement performance criteria.
RI Phase II Analytical Data including February 2006 Data	Preliminary Draft RI Report for Site 3 - Ninth Street Landfill	Tetra Tech NUS, January 2007 (data collected in 1997)	Secondary data will be compared to current concentrations to determine if the RI risk assessment accurately reflects current conditions.	1,4-dioxane not available. Validation criteria for the secondary data were the same as will be used for the current data. Secondary data meet the required measurement performance criteria.

RI - Remedial Investigation

SAP Worksheet No. 14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

Groundwater Level Measurements

Prior to the collection of groundwater samples, one round of water level measurements will be collected from all of the wells to be sampled. All measurements will be collected within an 8-hour period of consistent weather conditions to minimize atmospheric or precipitation effects on groundwater levels. The water levels will also be obtained a minimum of 12 to 24 hours after a significant rainfall event in order to negate the effects of short-term fluctuations in hydraulic head. Water level measurements are discussed in TtNUS SOP GH-1.2.

Monitoring Well Sampling and Analysis

All monitoring wells will be purged prior to sampling. All screened monitoring wells with a screen length of 10 feet or less will be purged and sampled using the low-flow sampling techniques in accordance with the EPA Region 3 Recommended Procedure for Low-Flow Purging and Sampling of Groundwater Monitoring Wells, Bulletin No. QAD023, June 16, 1999 (Appendix D). The monitoring wells will be purged and sampled using a submersible pump with an adjustable flow rate. In each well, the pump will be positioned within the well screen interval at the depth of the water-producing fracture. This depth will be identified using the available boring logs and geophysical logs. The pumping rate will be set at between 0.1 to 0.4 liters/minute. The purge water discharge will be monitored for pH, specific conductivity, turbidity, redox potential, and dissolved oxygen. When the levels of these parameters stabilize, and a minimum of two saturated screen volumes have been removed from the well, the purging is complete and sampling can begin. Parameter stabilization is defined as three successive readings (taken at least 5 minutes apart) within 0.1 unit for pH, 3% for conductivity, 10% for turbidity and DO, and 10 mv for redox potential. If possible, the stabilized turbidity reading should be less than 10 Nephelometric Turbidity Units (NTUs). The pumping rate will be controlled and the well's water level will be constantly monitored to assure that the static water level is not drawn down into or below the screen. The vertical location of the pump, the required minimum purge volume, and the calculations for parameter stabilization will be recorded on the groundwater sample log sheet.

Monitoring wells that have a screen length greater than 10 feet will be sampled by purging three to five well volumes prior to sample collection. The pump will be positioned at the top of the well screen interval. Water quality indicator parameters will be recorded for every half well volume. Monitoring well sampling procedures are discussed in SOP SA-1.1, which is included in Appendix A.

The off-Base, Lot 1 irrigation well is a continuously flowing well that discharges via a pipe to surface water. This well will be sampled by directly filling the sample bottles from the well discharge.

The locations of the existing monitoring wells to be sampled are shown in Figure 14-1. The sampling and analysis program is outlined in Worksheet 18, and the sampling requirements for each type of analyses (i.e., bottleware, preservation, holding time) are listed in Worksheet 19.

Surface Water Sampling and Analysis

Surface water samples will be collected by directly filling the bottles from the stream. Water quality parameters will be recorded for each sampling location. The surface water sampling procedures discussed in SOP SA-1.2 (included in Appendix A) will be followed. The surface water sampling locations are shown in Figure 14-1. The sampling and analysis program is outlined in Worksheet 18, and the sampling requirements for each type of analyses (i.e., bottleware, preservation, holding time) are listed in Worksheet 19.

DATA MANAGEMENT TASKS

This section describes how project information will be managed, organized, and maintained for efficient use by project personnel. The information management process is outlined from data generation to ultimate storage.

Project Documentation and Records

A summary of project documentation and records to be generated and stored in the project files is provided in Worksheet 29.

Data Package Deliverables

Certain field measurements (i.e., photo-ionization detection, etc.) are made primarily for health and safety monitoring. Additional field measurements may include readings such as pH, temperature, and specific conductance to monitor ambient conditions prior to sample collection. These data will be recorded on the appropriate log sheets.

For the Site 3 IGWM sampling events, a fixed-base laboratory will provide Contract Laboratory Program (CLP) equivalent data packages for all analyses. Additionally electronic deliverables, formatted according to the requirements stated in the laboratory subcontracts, will be provided by the laboratories for all analytical data. Worksheet No. 30 summarizes the analytical requirements.

Data Reporting Formats

Field data will be recorded on log sheets or in the project logbook. The laboratory will provide CLP equivalent data reporting forms 1 through 15 for the required metals and organic analyses presented in the previous paragraph.

Data Handling and Management

The data-handling procedures to be followed by the laboratories will meet the requirements in the laboratory subcontracts. All analytical and field data will be maintained in the project files. The project files will contain hard copies of the COC forms, sample log forms, sample location maps, and documentation of QA of data manipulation.

Data Tracking and Control

A “cradle-to-grave” sample tracking system will be used from the beginning to the end of the sampling event. Before field mobilization, the FOL will coordinate/initiate the sample tracking process. Sample jar labels will be hand-written in the field.

The labels will be reviewed for adherence to SAP requirements as well as for accuracy. The PM will coordinate with the analytical laboratories to ensure that they are aware of the number and type of samples and analyses.

When field sampling is underway, the FOL forwards the chain of custody (COC) forms to the PM or designee and the laboratories for each day that samples are collected. The PM or designee will confirm that the COC forms provide the information required by the SAP.

This will allow for early detection of errors made in the field so that adjustments can be made while the field team is mobilized. After successful completion of all requested analyses, the laboratories will submit an electronic deliverable for every sample delivery group (SDG). When all electronic deliverables have been received from the laboratories, the PM or designee will ensure that the laboratories performed all the requested analyses. Ideally, discrepancies can be noted early enough so that all samples can be analyzed within the prescribed holding times.

Sample Information

Data from field measurements will be recorded using the appropriate log sheets.

Reduction of field data entails the summarization and presentation of these data in tabular form. The reduction of laboratory data entails the manipulation of raw data instrument output into reportable results. Field data (e.g., photo-ionization detection) will be verified on a daily basis by the FOL. Laboratory data will be verified by the group supervisor and then by the laboratory's QC/Documentation Department.

For field data, the FOL will coordinate with the geographical information system (GIS) lead to ensure that all survey technical specifications are consistent with the underlying coordinate system in the GIS.

Electronic data arriving from the laboratories will pass through to the data validation manager (DVM) for database compilation and validation. The DVM will compile all the formatted laboratory electronic deliverables into a working project database. Data that are to be validated will be printed as data packages, which include the samples as part of each SDG and the appropriate analytical fraction. The data packages will be distributed to the appropriate data validators. The data validators will enter all data qualifiers and qualifier codes into the database and print out a hard copy and return it to the DVM. The DVM will check the data qualifiers and qualifier codes in the project database and print the final validated data for incorporation into the data validation letter. When all samples and analyses have been accounted for and validated, the PM will ensure that the analytical data are incorporated into the project database.

Project Data Compilation

The analytical laboratories will generate a PDF file of the analytical data packages, as well as electronic database deliverables. The electronic database will be checked against the PDF file provided by the laboratories and updated as required, based on data qualifier flags applied during the data validation process. The data generated during the implementation of the QAPP will be incorporated into the NAS JRB Willow Grove database and GIS. All data, such as units of measure and chemical nomenclature, will be manipulated to maintain consistency with the project database.

Geographical Information System

Data management systems consist of a relational database and GIS that are being used to manage environmental information pertaining to NAS JRB Willow Grove. The relational database stores chemical, geological, hydrogeologic, and other environmental data collected during environmental investigations. The GIS is built from the relational database and contains subsets of the larger data pool. Using the GIS, environmental data can be posted on base mapping to provide a graphical representation of the information.

Upon compilation of sample, chemical, biological, and positional data, the data will be compiled and incorporated into the NAS JRB Willow Grove GIS. The GIS system can be used to generate various maps for NAS JRB Willow Grove data including site location maps, sample location maps, and contaminant tag maps, as needed. The GIS software that is used will be documented in performance monitoring reports.

DATA REVIEW

The internal data verification requirements for this project include the maintenance and periodic review of field documentation (i.e., site logbooks, instrument calibration logs, chain of custody forms (COC), field summary reports, and field modification records) and laboratory analytical data packages. After receipt of analytical laboratory results, TtNUS will perform data validation according to the requirements outlined in the Region 3 Modifications to the National Functional Guidelines for Organic Review (September 1994) and method-specific requirements to ensure that the analytical results meet the project quality objectives.

After the data are validated, a list of nonconformities will be generated. Nonconformities require data qualifiers, which are used to alert the data user to inaccurate or imprecise data. For situations in which several quality control criteria are out of specification with regard to the limits specified in the DoD QSM (January, 2006), the data validator may make professional judgments and/or comments on the validity of the overall data package. In situations where the validity of an entire data package is in question, it may be necessary for the sample(s) to be reanalyzed. The reviewer will then prepare a technical memorandum presenting changes in the data, if necessary, and the rationale for making such changes.

The net result is a data package that has been carefully reviewed for its adherence to prescribed requirements and is suitable for its intended use. Data validation therefore plays a major role in determining the confidence with which key technical evaluations may be made.

Data validation reports for all parameters will be generated according to the procedures described in Standard Operating Procedures (SOP) DV-02 and DV-04. The final data validation report will include a technical memorandum, qualified analytical results, results reported by the laboratory, and documentation to support data qualification. All data will be flagged by an appropriate qualifying symbol.

The data and field records will also be reviewed by project personnel to ensure that the samples represent the intended sampling conditions and populations. Data qualified during validation will be reviewed to assess the impact of the qualifiers on the attainment of project objectives.

Analysis Tasks:
TCL VOCs, 1,4-Dioxane, TAL total and dissolved metals, and hexavalent chromium analysis of groundwater and surface water.

Quality Control Tasks:
QA/QC field samples including duplicates, field blanks, rinsate blanks, and trip blanks. Chain of custody procedures implemented.

Secondary Data:
Historical analytical data collected for the RI Phase I and II.

Assessment / Audit Tasks:
Routine audits of field personnel, field procedures, and 100% data validation for laboratory analytical data.

SAP Worksheet No. 15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater

Analyte	CAS Number	Project Action Limit Region 3 RBC Tap Water [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
VOC - SW-846 8260B				
Dichlorodifluoromethane	75-71-8	3.5E+02	0.2	1
Chloromethane	74-87-3	1.1E+01	0.3	1
Vinyl chloride	75-01-4	1.5E-01*	0.6	2
Bromomethane	74-83-9	8.5E+00	0.6	2
Chloroethane	75-00-3	3.6E+00	0.3	1
Trichlorofluoromethane	75-69-4	1.3E+03	0.3	1
1,1-Dichloroethene	75-35-4	3.5E+02	0.3	1
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5.9E+04	0.4	2
Acetone	67-64-1	5.5E+03	3	10
Carbon disulfide	75-15-0	1.0E+03	0.3	1
Methyl acetate	79-20-9	6.1E+03	0.8	3
Methylene chloride	75-09-2	4.1E+01*	0.3	1
trans-1,2-Dichloroethene	156-60-5	1.1E+02	0.2	1
Methyl tert-butyl ether	1634-04-4	2.6E+00	0.2	1
1,1-Dichloroethane	75-34-3	9.0E+02	0.3	1
cis-1,2-Dichloroethene	156-59-2	6.1E+01	0.3	1
2-Butanone	78-93-3	7.0E+03	3	10
Bromochloromethane	74-97-5		0.4	2
Chloroform	67-66-3	1.5E00*	0.2	1
1,1,1-Trichloroethane	71-55-6	1.7E+03	0.2	1
Cyclohexane	108-94-1	1.2E+04	0.3	1

Analyte	CAS Number	Project Action Limit Region 3 RBC Tap Water [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
Carbon tetrachloride	56-23-5	1.6E-00*	0.4	1
Benzene	71-43-2	3.4E00*	0.2	1
1,2-Dichloroethane	107-06-2	1.2E-01	0.2	1
Trichloroethene	79-01-6	2.6E-01*	0.3	1
Methylcyclohexane	108-87-2	6.3E+03	0.3	1
1,2-Dichloropropane	78-87-5	1.6E00*	0.3	1
Bromodichloromethane	75-27-4	1.7E-01	0.3	1
cis-1,3-Dichloropropene	10061-01-5	4.4E-01	0.3	1
4-Methyl-2-pentanone	108-10-1		2	10
Toluene	108-88-3	2.3E+03	0.2	1
trans-1,3-Dichloropropene	10061-02-6	4.4E00*	0.2	1
1,1,2-Trichloroethane	79-00-5	1.9E00*	0.4	1
Tetrachloroethene	127-18-4	1.0E00*	0.4	1
2-Hexanone	591-78-6		2	10
Dibromochloromethane	124-48-1	1.3E00*	0.3	1
1,2-Dibromoethane	106-93-4	5.3E-02*	0.3	1
Chlorobenzene	108-90-7	9.0E+01	0.3	1
Ethylbenzene	100-41-4	1.3E+03	0.3	1
o-Xylene	95-47-6	2.1E+02	0.3	1
m,p-Xylene	179601-23-1	2.1E+02	0.4	2
Styrene	100-42-5	1.6E+03	0.2	1
Bromoform	75-25-2	8.5E+01*	0.4	2
Isopropylbenzene	98-82-8	6.6E+02	0.3	1
1,1,2,2-Tetrachloroethane	79-34-5	5.3E-01*	0.3	1
1,3-Dichlorobenzene	541-73-1	1.8E+01	0.2	1
1,4-Dichlorobenzene	106-46-7	4.7E00*	0.3	1
1,2-Dichlorobenzene	95-50-1	2.7E+02	0.2	1

Analyte	CAS Number	Project Action Limit Region 3 RBC Tap Water [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
1,2-Dibromo-3-chloropropane	96-12-8	2.0E-04	2	7
1,2,4-Trichlorobenzene	120-82-1	6.1E+01	0.3	2
1,2,3-Trichlorobenzene	87-61-6		0.6	2
SVOC - SW-846 8270D				
1,4-Dioxane	123-91-1	6.1E+00	0.6	3
Inorganics - SW-846 6010B/7470A				
Aluminum	7429-90-5	3.7E+04	12.2	110
Antimony	7440-36-0	1.5E+01	6.6	28
Arsenic	7440-38-2	4.5E-01*	0.2	6
Barium	7440-39-3	7.3E+03	0.5	11
Beryllium	7440-41-7	7.3E+01	0.1	4
Cadmium	7440-43-9	1.8E+01	0.4	2
Calcium	7440-70-2		3.3	110
Chromium	7440-47-3	1.1E+02	1.6	6
Cobalt	7440-48-4		1.2	6
Copper	7440-50-8	1.5E+03	1.4	11
Iron	7439-89-6	2.6E+04	17	70
Lead	7439-92-1	1.5E+01	2.2	6.7
Magnesium	7439-95-4		11.3	110
Manganese	7439-96-5	7.3E+02	0.3	6
Mercury	7439-97-6	3.7E+00	0.04	0.2
Nickel	7440-02-0	7.3E+02	3.2	22
Potassium	7440-9-7		70	560
Selenium	7782-49-2	1.8E+02	3.5	10
Silver	7440-22-4	1.8E+02	1.2	4
Sodium	7440-23-5		30	560

Analyte	CAS Number	Project Action Limit Region 3 RBC Tap Water [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
Thallium	7440-28-0	2.6E+00	0.3	1
Vanadium	7440-62-2	3.7E+01	1.3	6
Zinc	7440-66-6	1.1E+04	1.5	20
Miscellaneous - SW-846 7196A				
Hexavalent Chromium	18540-29-9	1.1E+02	2	10

[1] EPA Region III Risk Based Concentration Screening Values (10/2007), Office of Solid Waste and Emergency Response (OSWER) for lead.

* The RBC has been increased by a factor of ten for carcinogens to correspond to a single-chemical risk of 10^{-5}. It is recognized that method detection limits are greater than certain 'project action limits'. However, it is anticipated that this will not be a problem for planned data use.

MDL - Method Detection Limit QL - Quantitation Limit RBC - Risk Based Concentration
PAL - Project Action Limit VOC - Volatile Organic Compound

SAP Worksheet No. 15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water

Analyte	CAS Number	Project Action Limit Region 3 Benchmark Freshwater [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
VOC - SW-846 8260B				
Dichlorodifluoromethane	75-71-8		0.2	1
Chloromethane	74-87-3		0.3	1
Vinyl chloride	75-01-4	9.3E+02	0.6	2
Bromomethane	74-83-9		0.6	2
Chloroethane	75-00-3		0.3	1
Trichlorofluoromethane	75-69-4		0.3	1
1,1-Dichloroethene	75-35-4	2.5E+01	0.3	1
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1		0.4	2
Acetone	67-64-1	1.5E+03	3	10
Carbon disulfide	75-15-0	9.2E-01	0.3	1
Methyl acetate	79-20-9		0.8	3
Methylene chloride	75-09-2	9.8E+01	0.3	1
trans-1,2-Dichloroethene	156-60-5	9.7E+02	0.2	1
Methyl tert-butyl ether	1634-04-4	1.1E+04	0.2	1
1,1-Dichloroethane	75-34-3	4.7E+01	0.3	1
cis-1,2-Dichloroethene	156-59-2	5.9E+02	0.3	1
2-Butanone	78-93-3	1.4E+04	3	10
Bromochloromethane	74-97-5		0.4	2
Chloroform	67-66-3	1.8E+00	0.2	1
1,1,1-Trichloroethane	71-55-6	1.1E+01	0.2	1
Cyclohexane	108-94-1		0.3	1

Analyte	CAS Number	Project Action Limit Region 3 Benchmark Freshwater [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
Carbon tetrachloride	56-23-5	1.3E+01	0.4	1
Benzene	71-43-2	3.7E+02	0.2	1
1,2-Dichloroethane	107-06-2	1.0E+02	0.2	1
Trichloroethene	79-01-6	2.1E+01	0.3	1
Methylcyclohexane	108-87-2		0.3	1
1,2-Dichloropropane	78-87-5		0.3	1
Bromodichloromethane	75-27-4		0.3	1
cis-1,3-Dichloropropene	10061-01-5	5.5E-02	0.3	1
4-Methyl-2-pentanone	108-10-1	1.7E+02	2	10
Toluene	108-88-3	2.0E+00	0.2	1
trans-1,3-Dichloropropene	10061-02-6	5.5E-02	0.2	1
1,1,2-Trichloroethane	79-00-5	1.2E+03	0.4	1
Tetrachloroethene	127-18-4	1.1E+02	0.4	1
2-Hexanone	591-78-6	9.9E+01	2	10
Dibromochloromethane	124-48-1		0.3	1
1,2-Dibromoethane	106-93-4		0.3	1
Chlorobenzene	108-90-7	1.3E+00	0.3	1
Ethylbenzene	100-41-4	9.0E+01	0.3	1
o-Xylene	95-47-6	1.8E+00	0.3	1
m,p-Xylene	179601-23-1	1.8E+00	0.4	2
Styrene	100-42-5	7.2E+01	0.2	1
Bromoform	75-25-2	3.2E+02	0.4	2
Isopropylbenzene	98-82-8	2.6E+00	0.3	1
1,1,2,2-Tetrachloroethane	79-34-5	6.1E+02	0.3	1
1,3-Dichlorobenzene	541-73-1	5.0E+00	0.2	1
1,4-Dichlorobenzene	106-46-7	5.0E+00	0.3	1
1,2-Dichlorobenzene	95-50-1	5.0E+00	0.2	1

Analyte	CAS Number	Project Action Limit Region 3 Benchmark Freshwater [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
1,2-Dibromo-3-chloropropane	96-12-8		2	7
1,2,4-Trichlorobenzene	120-82-1	5.0E+00	0.3	2
1,2,3-Trichlorobenzene	87-61-6	5.0E+00	0.6	2
SVOC - SW-846 8270D				
1,4-Dioxane	123-91-1	6.1E+00	0.6	3
Inorganics - SW-846 6010B/7470A				
Aluminum	7429-90-5	8.70E+01	12.2	110
Antimony	7440-36-0	3.00E+01	6.6	28
Arsenic	7440-38-2	5.00E+00	0.2	6
Barium	7440-39-3	4.00E+00	0.5	11
Beryllium	7440-41-7	6.60E-01	0.1	4
Cadmium	7440-43-9	2.50E-01	0.4	2
Calcium	7440-70-2	1.16E+05	3.3	110
Chromium	7440-47-3	8.50E+01	1.6	6
Cobalt	7440-48-4	2.30E+01	1.2	6
Copper	7440-50-8	9.00E+00	1.4	11
Iron	7439-89-6	3.00E+02	17	70
Lead	7439-92-1	2.50E+00	2.2	6.7
Magnesium	7439-95-4	8.20E+04	11.3	110
Manganese	7439-96-5	1.20E+02	0.3	6
Mercury	7439-97-6	2.60E-02	0.04	0.2
Nickel	7440-02-0	5.20E+01	3.2	22
Potassium	7440-9-7	5.30E+04	70	560
Selenium	7782-49-2	1.00E+00	3.5	10
Silver	7440-22-4	3.20E+00	1.2	4
Sodium	7440-23-5	6.80E+05	30	560

Analyte	CAS Number	Project Action Limit Region 3 Benchmark Freshwater [1] (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
Thallium	7440-28-0	8.00E-01	0.3	1
Vanadium	7440-62-2	2.00E+01	1.3	6
Zinc	7440-66-6	1.20E+02	1.5	20

[1] Freshwater Benchmark Criteria available at http://www.epa.gov/reg3hwmd/risk/eco/btag/sbv/fw/screenbench.htm

Note: It is recognized that method detection limits are greater than certain 'project action limits'. However, it is anticipated that this will not be a problem for planned data use.

MDL - Method Detection Limit QL - Quantitation Limit RBC - Risk Based Concentration
PAL - Project Action Limit VOC - Volatile Organic Compound

SAP Worksheet No. 16 -- Project Schedule / Timeline Table (optional format)
(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Semi-annual Event 1 Groundwater Sampling and Groundwater Level Measurements	Tetra Tech	03/10/2008	03/14/2008	Site 3 IGWM Letter Report - Ninth Street Landfill	06/06/2008 (draft) 09/18/2008 (final)
Semi-annual Event 2 Groundwater Sampling and Groundwater Level Measurements	Tetra Tech	09/15/2008	09/19/2008	Site 3 IGWM Letter Report - Ninth Street Landfill	12/07/2008 (draft) 03/07/2009 (final)

IGWM - Interim Groundwater Monitoring

SAP Worksheet No. 17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):
Existing site-related monitoring wells appropriately bound the area of concern; all of the site monitoring wells will be sampled. In addition, one off-Base well (Lot 1 Irrigation well) will be sampled and two surface water samples, upgradient (03SW10) and downgradient (03SW11) of the off-Base well, will be sampled because the off-Base well discharges to the surface water. Site maps and figures detailing the site location, boundaries, and sampling locations are included in Appendix C of this SAP.

One groundwater sample will be collected at each of 20 monitoring well locations, one off-Base well and two surface water locations for TCL VOCs, TAL total metals, and 1,4-dioxane analyses. TAL dissolved metals will be collected at 20 monitoring wells and the off-Base well. Hexavalent chromium will be analyzed at 8 well locations: 03MW01S, 03MW01I, 03MW03I, 03MW05S, 03MW05I, 03MW06S, 03MW06SI, and 03MW06I.

Hexavalent chromium will be collected at well locations which previously exhibited total chromium detections. The RI assumed chromium risks at these locations based on hexavalent chromium criteria. Therefore, hexavalent chromium will be analyzed to determine the type of chromium in these well locations.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations):

See Worksheet 18.

SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
03MW01I-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1
03MW01S-YYYYMMDD and DUP-01	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	2	SA-1.1
03MW01SI-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW02I-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW02S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW02SI-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW03I-YYYYMMDD and DUP-02	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI) (No field duplicate for Cr(VI))	2	SA-1.1

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
03MW03S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW03SI-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW04I-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW04S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW04SI-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW05I-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1
03MW05S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1
03MW06I-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1
03MW06S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
03MW06SI-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals, Cr(VI)	1	SA-1.1
03MW07S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW08D-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03MW08S-YYYYMMDD	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	1	SA-1.1
03IW01-YYYYMMDD and DUP-03	Groundwater	NA	VOC, 1,4-Dioxane, Total and Dissolved Metals	2	SA-1.1
03SW10-YYYYMMDD	Surface Water	NA	VOC, 1,4-Dioxane, Total Metals	1	SA-1.2
03SW11-YYYYMMDD	Surface Water	NA	VOC, 1,4-Dioxane, Total Metals	1	SA-1.2

[1]Standard operating procedure (SOP) or worksheet that describes the sample collection procedures.

NA - Not Applicable
VOC - Volatile Organic Compound

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[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Groundwater and Surface Water	Volatiles	SW-846 8260B ALSI SOP-02-8260B	3 - 40 mL glass vials with Teflon septum	120 mL	HCl, cool to 4°C; no headspace	14 days to analysis
Groundwater and Surface Water	1,4-Dioxane	SW-846 8270D ALSI SOP-02-8270	2 - 1L Amber glass bottles	2 L	Cool to 4°C	7 days to extraction; 40 days to analysis
Groundwater and Surface Water	Metals	SW-846 6010 ALSI SOP-03-6010B	1 - 500 mL Polypropylene bottle	150 mL minimum	Cool to 4°C with HNO_3	180 days to analysis
Groundwater	Mercury	SW-846 7470 ALSI SOP-03-Hg	Any size plastic or glass	100 mL minimum	Cool to 4°C with HNO_3	28 days to analysis
Groundwater	Hexavalent Chromium	SM 3500-Cr B ALSI SOP-04-Cr+6	3 - 40 mL glass vials with Teflon septum	125 mL	Cool to 4°C	24 hours to analysis

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

[2] Provide the minimum sample volume or mass requirement if it differs from the container volume.

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

°C - Degrees Celsius
ALSI - Analytical Laboratory Services, Inc.
HCl - Hydrochloric Acid
HNO_3 - Nitric Acid
L - Liter
mL - Milliliter
NaOH - Sodium Hydroxide
SOP - Standard Operating Procedure

SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations[2]	No. of Field Duplicates	No. of MS/MSDs[1]	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples[3]	Total No. of Samples to Lab
Groundwater	Volatiles	21	2	2/2	1	2	4	NA	32
Groundwater	1,4-Dioxane	21	2	2/2	1	2	NA	NA	28
Groundwater	Total Metals	21	2	2/2	1	2	NA	NA	28
Groundwater	Dissolved Metals	21	2	2/2	1	2	NA	NA	28
Groundwater	Mercury	21	2	2/2	1	2	NA	NA	28
Groundwater	Hexavalent Chromium	8	1	1/1	1	1		NA	12
Surface Water	Volatiles	2	1	1/1	1	0	1	NA	6
Surface Water	1,4-Dioxane	2	1	1/1	1	0	1	NA	6
Surface Water	Total Metals	2	1	1/1	1	0	NA	NA	5
Surface Water	Mercury	2	1	1/1	1	0	NA	NA	5

[1] Although the MS/MSD is not typically considered a field QC it is included here because location determination is often established in the field.

[2] If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

[3] The number of Batch or Project-specific proficiency testing (PT) samples is optional but highly recommended.

MS/MSD - Matrix Spike/Matrix Spike Duplicate
NA - Not Applicable
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
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing	Tetra Tech	Bladder Pump, Peristaltic Pump, Submersible Pump	N	
SA-1.2	Surface Water and Sediment Sampling	Tetra Tech	Bottleware	N	
CT-04	Sample Nomenclature	Tetra Tech	NA	Y	Samples will be named according to Worksheet 18
SA-6.1	Non-radiological Sample Handling	Tetra Tech	Sample Bottleware, Packaging Material, Shipping Materials	N	
SA-6.3	Field Documentation	Tetra Tech	NA	N	
SA-7.1	Decontamination of Field Equipment	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	
ME-12	Photo-Ionization Detector	Tetra Tech	PID	N	

NA - Not Applicable
PID - Photoionization Detector
SOP - Standard Operating Procedure

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	Resp. Person	SOP Reference[2]	Comments
Photo-Ionization Detector	Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	ME-12	
Multi-parameter Water Quality Meter	Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	SA-1.1	
Bladder Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Replace	FOL	SA-1.1	
Redi Flo™ Submersible Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Replace	FOL	SA-1.1	
Photo-Ionization Detector	Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	ME-12	

[1] Activities may include calibration, verification, testing, and maintenance.

[2] Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet No. 21).

FOL - Field Operations Leader
SOP - Standard Operating Procedure

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?[1] (Y/N)
ALSI SOP-02-8260B	Volatile Organics by GC/MS: Capillary Column Technique Rev. 9, 8/17/06	Definitive	Organic/GC/MS	GC/MS	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-02-8270	Semi-volatile Organic Compounds by GC/MS rev 6 3/22/06	Definitive	Organic/GC/MS	GC/MS	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-09-3015	Microwave Assisted Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP or GFAA Spectroscopy rev 7 2/20/06	Definitive	Inorganic/Metals	NA/ Digestion	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-03-6010B	Analysis of Total Metals by Inductively Coupled Plasma Using the TJA Trace ICP rev 12, 2/13/07	Definitive	Inorganic/Metals	ICP-AES	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-03-Hg	Mercury by Cold Vapor Atomic Absorption Using an Automated Continuous-Flow Vapor Generator rev 12 3/23/06	Definitive	Inorganic/Metals	Cold Vapor, either PSA or CETAC	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-04-Cr+6	Hexavalent Chromium in Water	Definitive	Inorganic/Wet Chemistry	Spectrophotometer	Analytical Laboratory Services, Inc. of Middletown, PA	N

[1] If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy QAO.

GC/MS - Gas Chromatograph/Mass Spectrometer
GFAA - Graphite Furnace Atomic Absorption
ICP-AES - Inductively Coupled Plasma-Atomic Emission Spectroscopy
NA - Not Applicable

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 (CA)	Person Responsible for CA[2]	SOP Reference[1]
Gas Chromatograph /Mass Spectrometer (GC/MS)	Minimum five point calibration for all analytes.	Instrument receipt, instrument change (new trap, column, etc.), when Continuing Calibration Verification (CCV) does not meet criteria.	Relative Standard Deviation (RSD) for each Calibration Check Compound (CCC) < 30%, minimum mean RF for each System Performance Check Compound (SPCC) as noted in 7.3.5.4 of method 8260B or 8270C. If RSD for an analyte is > 15% apply linear (r2 > 0.99) or quadratic method for quantitation.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	ALSI SOP-02-8260B
GC/MS	Minimum five point calibration for all analytes	Instrument receipt, instrument change (new trap, column, etc.), when CCV does not meet criteria, as needed	RSD for each CCC < 30%, minimum mean RF for each SPCC. If RSD for an analyte is > 15% apply linear (r2 > 0.99) or quadratic method for quantitation	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	ALSI SOP-02-8270
Cold Vapor, either PSA or CETAC	Minimum five calibration standards and a calibration blank.	Every 24-hours or if QC is out of criteria.	4-5 point calibration plus blank per manufacturer's guidelines; analytes run at their calibration levels must fall within 90-110% of True Values.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	ALSI SOP-03-Hg

SAP Worksheet No. 24 -- Analytical Instrument Calibration Table (Continued)
(UFP-QAPP Manual Section 3.2.2

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA2	SOP Reference1
Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES)	4-5 point calibration plus blank per manufacturer's guidelines.	At the beginning of each day or if QC is out of criteria.	4-5 point calibration plus blank per manufacturer's guidelines; analytes run at their calibration levels must fall within 90-110% of True Values.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	ALSI SOP-03-6010B

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet No. 23).
[2] Name or title of responsible person may be used.

CCC - Calibration Check Compound
CCV - Continuing Calibration Verification
GC/MS - Gas Chromatograph/Mass Spectrometer
ICP-AES - Inductively Coupled Plasma-Atomic Emission Spectroscopy
RSD - Relative Standard Deviation
SPCC - System Performance Check Compound
r2 - Correlation Coefficient

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[2]	SOP Reference[1]
Gas Chromatograph /Mass Spectrometer	Check pressure and gas supply daily. Bake out trap and column, manual tune if bromofluorobenzene (BFB) not in criteria, change septa as needed, cut column as needed, change trap as needed.	VOC Analysis	Initial and Continuing Calibration	Initial Calibration: Instrument receipt, instrument change (new trap, column, etc.), when CCC does not meet criteria; Continuing Calibration: At beginning of each 12 hour shift immediately after BFB tune.	Initial Calibration: RSD for each CCC < 30%, min. mean RF for each SPCC as noted in 7.3.5.4 of method 8260B. If RSD for an analyte is > 15% apply linear or quadratic method for quantitation; Continuing Calibration: % difference for each CCC < 20%, min. RF for each SPCC as noted in 7.3.5.4 of method 8260B.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. Record maintenance activities in MS05-02 Maintenance Log 02-0160.	Analyst/ Supervisor	ALSI SOP-02-8260B
GC/MS	Check pressure and gas supply daily. Bake out column, manual tune if DFTPP not in criteria, change septa as needed, cut column as needed, change trap as needed.	SV Analysis	Initial Calibration, Continuing Calibration	Initial Cal: Instrument receipt, instrument change (new column, etc.), when CCC does not meet criteria Cont. Cal: At beginning of each 12 hour shift immediately before sample analysis.	Initial Cal: RSD for each CCC < 30%, min. mean RF for each SPCC. If RSD for an analyte is > 15% apply linear or quadratic method for quantitation Cont. Cal: %D for each CCC < 20%, min. RF for each SPCC.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. Record maintenance activities in Maintenance Log.	Analyst/ Supervisor	ALSI-SOP 02-8270

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
Inductively Coupled Plasma-Atomic Emission Spectroscopy	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed.	Metals Analysis	Initial Calibration, Initial Calibration Verification (ICV), Continuing Calibration Verification	Initial Calibration: At the beginning of each day or if QC is out of criteria; Initial Calibration Verification: Immediately after instrument calibration; Continuing Calibration Verification: After every 10 samples and at end of analytical sequence.	Initial Calibration: 4-5 point calibration plus blank with .995 or better correction coefficient; Initial Calibration Verification and Continuing Calibration Verification: 90-110% of true value for Inductively Coupled Plasma.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected metals. Record maintenance activities in Thermo Trace Maintenance Logbook 03-0102.	Analyst/ Supervisor	ALSI SOP-03-6010B
Cold Vapor, either PSA or CETAC	Clean optical cell when needed. Check lamp, gas pressure, replace peristaltic pump tubing as needed.	Mercury Analysis	Initial Calibration, Initial Calibration Verification, Continuing Calibration Verification	Initial Calibration: Every 24-hours; Initial Calibration Verification: Immediately after instrument calibration; Continuing Calibration Verification: After every 10 samples and at end of analytical sequence.	Initial Calibration: 4-5 point calibration plus blank with .995 or better correction coefficient; Initial Calibration Verification: 90-110% of true value for Inductively Coupled Plasma; Continuing Calibration Verification: 80-120% of true value for Inductively Coupled Plasma.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected samples. Record maintenance activities in CETAC M-6100 Hg Analyzer Maintenance Logbook 03-0381.	Analyst/ Supervisor	ALSI SOP-03-Hg
Spectrophotometer	5point curve	Hexavalent Chromium	Continuing Calibration Verification	After every 10 samples And at the end of Analytical sequence.	90-110% of true value.	Recalibrate and/or perform necessary equipment maintenance Check Cal. Stds. Reanalyze affected data.	Analyst/ Supervisor	ALSI- SOP04-Cr+6

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

[2] Name or title of responsible person may be used.

CCC - Calibration Check Compound
RSD - Relative Standard Devia
SPCC - System Performance Check Compound
RF- Response Factor
QC - Quality Control
VOC - Volatile Organic Compound

SAP Worksheet No. 26 -- Sample Handling System
(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Don Whalen/Tetra Tech, Xuejun Chang/Tetra Tech
Sample Packaging (Personnel/Organization): Don Whalen/Tetra Tech, Xuejun Chang/Tetra Tech
Coordination of Shipment (Personnel/Organization): Don Whalen/Tetra Tech, Xuejun Chang/Tetra Tech
Type of Shipment/Carrier: Overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample custodians/ALSI (ALSI SOP 19-Rec/Han)
Sample Custody and Storage (Personnel/Organization): Sample custodians/ALSI
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff/ALSI
Sample Determinative Analysis (Personnel/Organization): GC/MS, ICP-AES, CVAA, Spectrophotometer/ALSI
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 30 days from submittal of final report
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 30 days from submittal of final report
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Sample custodians/ALSI (ALSI SOP 19-Disp)
Number of Days from Analysis: 30 days from submittal of final report

ALSI - Analytical Laboratory Services, Inc.
ICP - Inductively Couple Plasma
CVAA - Cold Vapor Atomic Absorption
Tetra Tech - Tetra Tech NUS, Inc.
GC/MS - Gas Chromatograph/Mass Spectrometer

SAP Worksheet No. 27 - Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Following sample collection into 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 duct or clear packaging tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. Laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4 degrees centigrade (°C) until delivery to the laboratories. Proper custody procedures will be followed throughout all phases of sample collection and handling. Chain of custody (COC) 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 TtNUS SOP SA-6.1.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): ALSI SOP 19-COC/19-Rec/Han

Sample Identification Procedures: Sample nomenclature will be conducted in general accordance with the procedures outlined in TtNUS SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature for each tracking number includes the site being investigated, sample media identifier, and sample location number. The standard sample matrix and type codes used for this field event are as follows: Duplicate samples will be submitted to the laboratory as blind duplicates. Therefore duplicate codes will be reflective of the standard sample matrix code followed by a “DUP” tag and sequentially listed. Due to the blind nature of the duplicate samples, no sample depth or date will be listed with the duplicated sample. An example of a duplicate sample would be “03DUP001”. The QA/QC type codes used for this field event are as follows: TB for Trip Blanks, FB for field blanks, and RB for rinsate blanks. Field QC blanks will be labeled sequentially followed by the date (i.e., TB20070430, FB20070501). Samples to be used for matrix spikes and matrix spike duplicates will be labeled MS/MSD on the bottle label and noted on the chain-of-custody, as required in the laboratory QA Plan; however, “MS/MSD” will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in TtNUS SOP SA-6.3.

Chain-of-custody Procedures: After recovery, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples recovered, custody records will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the custody record. Attached SOP SA-6.3 (Field Documentation) provides further details on the COC procedure. COC requirements are also documented with instructions contained in each shipment from the laboratory. (ALSI SOP 19-COC)

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater/Surface Water
Analytical Group	VOC
Analytical Method / SOP Reference	SW-846 8260B/ ALSI SOP-02-8260B

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One every 12 hours prior to sample analysis	No target compounds ≥ ½ QL (quantitation limit) except common lab contaminants which should be < RL (reporting limit)	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds ≥ ½ quantitation limit except common lab contaminants which should be < reporting limit).
Surrogates	3 per sample	Statistically derived limits.	(1) Re-prep and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Statistically derived limits
Laboratory Control Spike (LCS)	One per batch of 20 or less	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.	(1) Evaluate and reanalyze if possible (2) If a matrix spike / matrix spike duplicate was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are <quantitation limit) narrate otherwise re-prep and reanalyze.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.
Internal Standards (IS)	3 per sample	Retention time ± 30 seconds; extracted ion current profile area within -50% to +100% of last calibration verification (12 hours) for each IS.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention time ± 30 seconds; extracted ion current profile area within -50% to +100% of last calibration verification (12 hours) for each IS.

Matrix	Groundwater/Surface Water
Analytical Group	VOC
Analytical Method / SOP Reference	SW-846 8260B/ ALSI SOP-02-8260B

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix spike / Matrix spike duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the laboratory control sample and matrix spike / matrix spike duplicate are unacceptable re-prep the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits.

Matrix	Groundwater/Surface Water
Analytical Group	Semivolatile Organic Compounds (SVOCs)
Analytical Method / SOP Reference	SW-846 8270/ ALSI SOP-02-8270

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per 20 samples in analytical batch	No target analytes ≥1/2 QL; with the exception of common field/laboratory contaminants	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst / Laboratory Supervisor	Bias / Contamination	No target analytes ≥1/2 QL; with the exception of common field/laboratory contaminants
Laboratory Control Spike	1 per 20 samples in analytical batch	Statistically derived limits.	(1) Evaluate and reanalyze if possible (2) If an MS/MSD was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are <QL narrate otherwise re-prep and reanalyze.	Analyst / Laboratory Supervisor	Accuracy / Bias	Statistically derived limits.
Surrogate Standards	Each sample	Statistically derived limits.	(1) Re-prep and reanalyze for confirmation of matrix interference when appropriate.	Analyst / Laboratory Supervisor	Accuracy / Bias	Statistically derived limits.
Internal Standards	Each sample	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst / Laboratory Supervisor	Accuracy / Bias / Precision	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS
Matrix spike/matrix spike duplicate	1 per 20 samples in analytical batch	Statistically derived limits, 30% RPD	(1) CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable re-prep the samples and QC.	Analyst / Laboratory Supervisor	Accuracy / Bias / Precision	Statistically derived limits, 30% RPD
Field Duplicates	1 per 10 environmental samples	Values > 5X QL: ±30% Values < 5X QL: difference between results must be <±QL	No corrective action by laboratory.	Data Validator	Precision	Values > 5X QL: ±30% Values < 5X QL: difference between results must be <± QL

Matrix	Groundwater/Surface Water
Analytical Group	Metals
Analytical Method / SOP Reference	SW-846 6010B/ ALSI SOP-03-6010B

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples	Less than 1/2 quantitation limit)	1) Investigate source of contamination. Re-digest and reanalyze all associated samples if sample concentration ≥ quantitation limit) and <10x the blank concentration.	Laboratory Supervisor	Bias / Contamination	Less than ½ quantitation limit
Laboratory Control Sample	One per digestion batch of 20 or fewer samples	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. Re-digest and reanalyze all associated samples.	Laboratory Supervisor	Accuracy / Bias / Contamination	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.
Duplicate Sample	One per digestion batch of 20 or fewer samples	Relative percent difference (RPD) ≤20% for duplicate spikes.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Precision	Relative percent difference ≤20% for duplicate spikes
Matrix Spike	One per digestion batch of 20 or fewer samples	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Recovery ± 25% of true value, if sample < 4x spike added.
ICP Serial Dilution	One per digestion batch	If original sample result is at least 50x Instrument Detection Limit (IL), 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyzed sample to eliminate interference.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.

Matrix	Groundwater/Surface Water
Analytical Group	Mercury
Analytical Method / SOP Reference	SW-846 7470A/ ALSI SOP-03-Hg

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch	No analyte detected > ½ quantitation limit)	(1) Investigate source of contamination. (2) Report all sample results < quantitation limit). (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.	Laboratory Supervisor	Bias Contamination	No analyte detected >½ quantitation limit)
Laboratory Control Sample	One per prep batch	80-120 % recovery	(1) If the laboratory control sample (LCS) fails high, report samples that are <quantitation limit). (2) Recalibrate and/or reanalyze other samples.	Laboratory Supervisor	Accuracy / Bias	80-120 % recovery
Duplicate Sample	One sample duplicate per 20 samples	Relative percent difference ≤20 for samples >3X the Quantitation Limit	(1) Investigate problem and reanalyze sample in duplicate (2) If Relative Percent Difference still >20, report original result with notation or narration.	Analyst, Laboratory Supervisor and Data Validator	Precision	Relative percent difference ≤20 for samples <3X the quantitation limit and <100% for samples >3X the quantitation limit
Matrix Spike	One for every set of 10 samples	85-115% recovery if sample is < 4x the spike added	(1) If both the laboratory control sample and MS are unacceptable re-prep and reanalyze the samples and QC. (2) For 7196, dilute a new pH adjusted aliquot, re-spike and reanalyze to confirm matrix interference; narrate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	85-115% recovery

Matrix	Groundwater
Analytical Group	Cr(VI)
Analytical Method / SOP Reference	SM 3500-Cr B/ ALSI SOP-04-Cr+6

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch	No analyte detected > ½ quantitation limit	(1) Investigate source of contamination. (2) Report all sample results < quantitation limit. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.	Laboratory Supervisor	Bias Contamination	No analyte detected >½ quantitation limit
Laboratory Control Sample	One per prep batch	80-120 % recovery	(1) If the laboratory control sample fails high, report samples that are <quantitation limit. (2) Recalibrate and/or reanalyze other samples.	Laboratory Supervisor	Accuracy / Bias	80-120 % recovery
Duplicate Sample	One sample duplicate per 20 samples	Relative percent difference ≤20 for samples >3X the quantitation limit	(1) Investigate problem and reanalyze sample in duplicate (2) If Relative percent difference still >20, report original result with notation or narration.	Analyst, Laboratory Supervisor and Data Validator	Precision	RPD ≤20 for samples <3X the quantitation limit and <100% for samples >3X the quantitation limit
Matrix Spike	One for every set of 10 samples	85-115% recovery if sample is < 4x the spike added	(1) If both the LCS and MS are unacceptable re-prep and reanalyze the samples and QC. (2) For 7196, dilute a new pH adjusted aliquot, re-spike and reanalyze to confirm matrix interference; narrate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	85-115% recovery

GC - Gas Chromatograph IS - Internal Standard LCS - Laboratory Control Sample QC - Quality Control QL - Quantitation Limit IS - Internal Standard
Mg/L - Milligrams per Liter PQL - Practical Quantitation Limit RL - Reporting Limit RPD - Relative Percent Difference

SAP Worksheet No. 29 -- Project Documents and Records Table
(UFP-QAPP Manual Section 3.5.1)

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
-Field Logbook -Field Sample Forms - Chain of custody (COC) Records -Air bills -Sampling Instrument Calibration Logs - Sampling Notes and Drilling Logs -Photographs - Field task modification request (FTMR) Forms -This Sampling and analysis plan (SAP) -Health and Safety Plan	-Field Sample Data -Field Logbook -Field Sample Forms -Sampling Instrument Calibration Logs -Field Sample Data - Chain of custody record/Form - Field task modification request Forms	-Sample receipt, custody, and tracking record -Standards traceability logs -Equipment calibration logs -Sample preparation logs -Run logs -Equipment maintenance, testing, and inspection logs -Corrective action forms -Reported field sample results -Reported results for standards, quality control checks, and quality control samples -Sample storage and disposal records -Telephone logs -Extraction/clean-up records -Raw data (stored electronically)	-Field Sampling Audit Checklist -Analytical Audit Checklist -Data Review Reports -Laboratory QA Plan -Tabulated Data Summary Forms -Data Validation Memoranda -Performance Monitoring Report	NA

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[1] (name and address, contact person and telephone number)	Backup Laboratory / Organization[1] (name and address, contact person and telephone number)
Groundwater and Surface Water	VOCs	See Worksheet 18	SW-846 8260B ALSI SOP-02-8260B	21 days	Analytical Laboratory Services, Inc. 34 Dogwood Lane Middletown, PA 17057 Phone: 800-794-7709 Fax: 717-944-1430 Scott Brunk 717-944-5541	NA
Groundwater and Surface Water	1,4-Dioxane	See Worksheet 18	SW-846 8260B ALSI SOP-02-8270	21 days	Same as above	NA
Groundwater and Surface Water	Metals	See Worksheet 18	SW-846 6010 ALSI SOP-03-6010B	21 days	Same as above	NA
Groundwater and Surface Water	Mercury	See Worksheet 18	SW-846 7470 ALSI SOP-03-Hg	21 days	Same as above	NA
Groundwater	Hexavalent Chromium	See Worksheet 18	SM 3500-Cr B ALSI SOP-04-Cr+6	21 days	Same as above	NA

ALSI - Analytical Laboratory Services, Inc.
NA - Not Applicable
SOP - Standard Operating Procedure
VOC - Volatile Organic Compound

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 (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Health and Safety	1 per contract year	Internal	Tetra Tech	TBD	PM	Auditor and HSM	HSM, Matt Soltis
Laboratory Systems Audit	Every 18 months	External	NFESC	TBD	Laboratory QA Manager	Laboratory QA Manager	Laboratory QA Manager
Field Quality Assurance	1 per contract year	Internal	Tetra Tech	TBD	PM	Auditor and QAM	QAM, Kelly Carper

HSM - Health and Safety Manager
NFESC - Naval Facilities Engineering Service Center
PM - Project Manager
QA - Quality Assurance
QAM - Quality Assurance Manager
TBD - To Be Determined
Tetra Tech - Tetra Tech NUS, Inc.

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
Health and Safety Audit	Audit checklist and written audit finding summary	Tetra Tech PM, FOL, and Program Manager	Dependant on findings, if major, a stop work may be issued immediately, however, if minor, within 1 week of audit.	Written Memo	Tetra Tech HSM, Auditor, and Program Manager	Within 48 hours of notification
Field Sampling System Audit	Audit checklist and written audit finding summary	Tetra Tech PM, FOL, and Program Manager	Dependant on findings, if major, a stop work may be issued immediately, however, if minor, within 1 week of audit.	Written Memo	Tetra Tech QAM, Auditor, and Program Manager	Within 48 hours of notification
Laboratory Systems Audit	Written audit report	Laboratory QA Manager	Not specified by NFESC	Letter	NFESC	Specified by NFESC

FOL - Field Operations Leader
HSM - Health and Safety Manager
NFESC - Naval Facilities Engineering Service Center
PM - Project Manager
QA - Quality Assurance
QAM - Quality Assurance Manager
Tetra Tech - Tetra Tech NUS, Inc.

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	DVM or designee	DVM or designee	Tetra Tech PM, project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately	Tetra Tech QAM	Tetra Tech PM, project file
Project monthly progress report	Monthly for duration of project	Monthly	Tetra Tech PM	Tetra Tech PM, QAM, Program Manager, project file
Field progress report	Daily, oral, during the course of sampling	Everyday that field sampling is occurring	Tetra Tech FOL	Tetra Tech PM
Laboratory QA report	When significant plan deviations results from unanticipated circumstances	Immediately	Subcontracted laboratories	Tetra Tech, project file

DVM - Data Validation Manager
FOL - Field Operations Leader
PM - Project Manager
QA - Quality Assurance
QAM - Quality Assurance Manager
SDG - Sample Delivery Group
Tetra Tech - Tetra Tech NUS, Inc.

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)
Sample Tables	Proposed samples verified to have been collected.	Internal	Tetra Tech FOL or designee
Chain of custody	Chain of custody records will be reviewed internally by the Project Manager or designee and compared against sample tables listing the proposed samples to verify that all planned samples have been collected.	Internal	Tetra Tech PM or designee
Sample Coordinates	Sample locations have been verified to be correct and in accordance with the SAP (overlay map of proposed locations against map of actual locations)	Internal	Tetra Tech PM, FOL, or designee
Data Package	Verify that the data package contains all the elements required by the functional guidelines and scope of work, this occurs as part of the data validation process.	Internal	Tetra Tech Data Validator
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	Tetra Tech PM or designee

FOL - Field Operations Leader
PM - Project Manager
QAM - Quality Assurance Manager
Tetra Tech - Tetra Tech NUS, Inc.

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[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Data Package	Validator will verify that elements of the data package that are required for validation is present and if not the lab will be contacted and the missing information will be requested. Validation will be performed as per Worksheet 36.	Tetra Tech Data Validator
IIa	Field Logs / Sample Coordinates	Verify that the sampling plan was implemented and carried out as written and any deviations are documented.	Tetra Tech PM
IIa	Electronic Data	Verify all data have been transferred correctly and completely to the final SQL database.	Tetra Tech PM or designee
IIa	SAP, SOPs, Field Logs, COCs	Validator will verify that elements of the data package that are required for validation is present and if not the lab will be contacted and the missing info will be requested. Validation will be performed as per Worksheet 36.	Tetra Tech PM, FOL, or designee

[1] IIa=compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP manual, V.1, March 2005.]
IIb=comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP manual, V.1, March 2005]

COC - Chain of Custody
FOL - Field Operations Leader
PM - Project Manager
SOP - Standard Operating Procedure
SQL - Structured Query Language
Tetra Tech - Tetra Tech NUS, Inc.

SAP Worksheet No. 36 -Analytical Validation (Steps IIa and IIb) Summary Table
(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Groundwater and Surface Water	Volatiles	SW-846 8260B, Tetra Tech SOP DV-02 and SAP Worksheets 12, 15, and 28.	Megan Ritchie
IIa and IIb	Groundwater and Surface Water	1,4-Dioxane	SW-846 8270, Tetra Tech SOP DV-02 and SAP Worksheets 12, 15, and 28.	Megan Ritchie
IIa and IIb	Groundwater and Surface Water	Total and Dissolved Metals/Mercury	SW-846 6010B/7470A, Tetra Tech SOP DV-04 and SAP Worksheets 12, 15, and 28.	Megan Ritchie
IIa and IIb	Groundwater	Hexavalent Chromium	SM 3500-Cr, Tetra Tech SOP DV-04 and SAP Worksheets 12, 15, and 28.	Megan Ritchie

SOP - Standard Operating Procedure
Tetra Tech - Tetra Tech NUS, Inc.

SAP Worksheet No. 37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

Data validation will determine which data are usable. Qualifiers will be applied to each value based on the results of the data validation. Rejected values (qualified with "R") and blank qualified values ("B") will be eliminated from further consideration. Estimated and biased values (J [estimated], K [biased high], and L [biased low]) will be used as the reported value.

Statistical tests are not expected to be performed for this data.

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

After all data evaluations are completed, any limitations on the use of data will be known and the limitations will be considered during decision making. If necessary, investigation objectives may be revised in anticipation of additional data collection in order to meet project quality objectives for the site.

After data validation and an overall review of data quality indicators, the data will be reconciled with MPCs to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and statistical analyses may be performed to estimate several of the data set characteristics. Simple summary statistics for target analytes, such as the maximum concentration, minimum concentration, number of samples exhibiting no detectable analyte, the number of samples exhibiting detectable analytes, and the proportion of samples with detectable and undetectable analytes.

Identify the personnel responsible for performing the usability assessment:

One hundred percent of the laboratory data from chemical analyses will be validated. Validation of analytical data will be conducted by Tetra Tech. Final review and approval of validation deliverables will be completed by the DVM or designee. VOC, metals, mercury, and hexavalent chromium, results will be validated according to the requirements of the methods, Tetra Tech data validation SOPs for non-CLP data, and USEPA Region III Modifications (USEPA, 1993 and 1994) to the extent practicable.

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APPENDIX A

TETRA TECH SOPs

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1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

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

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

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

001 - Monitoring Well 1
N32E92 - Grid location 32 North and 92 East
D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

BC - Blue Crab
GB - Blue Gill
CO - Corn
SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

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

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 DEVIATIONS

Any deviation from this SOP must be addressed in detail in the site specific planning documents.

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1.0 SW-846 ORGANICS BY GC/MS

1.1 Volatiles (Method 8260B)

1.1.1 Applicability

Method 8260B is used to determine volatile organic compounds in most waste matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 8260B analyte list includes of the volatile CLP 3/90 Target Compound List (TCL) (Section 1.1.1) plus the following compounds*:

Acetonitrile
Acrolein
Acrylonitrile
Allyl chloride
Chloropropene
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
Dibromomethane
trans-1,4-Dichloro-2-butene
Dichlorodifluoromethane
trans-1,2-Dichloroethene
Ethyl methacrylate
Iodomethane
Methacrylonitrile
Methyl methacrylate
2-Picoline
Pyridine
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl acetate

* Appendix IX target compounds

Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared by Method 5030.

1.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

1.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.1.4 Sample Preparation

Method 5030 is a purge-and-trap procedure performed to prepare and extract volatile compounds from samples and introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

1.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

1.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J): nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

1.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >25%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds whose RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J), when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 25% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply, and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

1.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance, nondetects are not qualified based on high surrogate recovery.

1.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for appropriately evaluating MS/MSD analyses.

1.1.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate data validation protocol.

1.1.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

1.1.6.8 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than

the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.1.6.9 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

1.2 Semivolatiles (Method SW8250A, 8270C)

1.2.1 Applicability

Methods are applicable to most types of samples, regardless of water content, including groundwater, 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.

These methods can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of elution without derivatization as sharp peaks from a gas chromatographic column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

The above methods specifically analyze for the semivolatile Target Compound List (TCL) (Section 1.1.2) plus the following compounds*:

Acetophenone	Hexachlorophene	N-nitrosodimethylethylamine
Aniline	Hexachloropropene	N-nitroso-di-n-butylamine
Benzyl alcohol	Isodrin	N-nitrosomorpholine
Bis(2-chloroisopropyl)ether	Isosafrole	N-nitrosopiperidine
Chlorobenzilate	Kepone	Pentachlorobenzene
Diallate	Methapyrilene	Pentachloronitrobenzene
2,6-Dichlorophenol	3-Methylcholanthrene	Phenacetin
Dimethoate	Methyl methanesulfonate	p-Phenylenediamine
p-Dimethylaminoazobenzene	3-Methylphenol	Phorate
7,12-Dimethylbenz(a)anthracene	1,4-Naphthoquinone	2-Picoline
3,3'-Dimethylbenzidine	4-Nitroquinoline-1-oxide	Pronamide
a,a-Dimethylphenylamine	1-Naphthylamine	Safrole
1,3-Dinitrobenzene	2-Naphthylamine	1,2,4,5-Tetrachlorobenzene
Diphenylamine	5-Nitro-o-toluidine	Thionazin
Ethyl methanesulfonate	N-nitrosodiethylamine	o,o,o-Triethylphosphorothioate
Famphur	N-nitrosodimethylamine	1,3,5-Trinitrobenzene

* Appendix IX target compounds

The preceding methods are based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures, Method 8270C uses GC/MS capillary column technique.

1.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source, depending upon the diversity of the industrial complex or waste being sampled.

1.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or sonication (Method 3550) procedures.

1.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

1.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Affected positive results are generally qualified as estimated (J), nondetects (UJ). Alternately, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance. If the sample was extracted beyond 14 days from collection (28 days for solid samples), the holding time exceedance is considered to be gross and positive results are qualified as estimated (J) or (L); nondetects are rejected (R). Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.

1.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs, and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >30%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 30%, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.2.6.3 Blank Contamination

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The action levels for qualification are 10X the maximum amount of phthalates found in the blanks (phthalates are common contaminants) and 5X the maximum amount of other contaminants found in the blanks. The actual action level applied is sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and moisture content. The type and manner in which the qualifiers are applied vary with protocol [i.e., use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate data validation protocol for specific guidance.

1.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Semivolatile compounds are divided into two classes, base-neutral compounds and acid-extractable compounds. Each class of compounds has its own associated surrogates. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and nondetects are rejected, (R). These results are biased low.

No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.

If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); nondetects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; nondetects are not qualified based on high surrogate recoveries.

1.2.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

1.2.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

1.2.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I BNA-TIC reports and the laboratory raw data. The guidance given in the 3/90 National Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

1.2.6.8 Other Considerations

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results. Consider nondetects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be less than 35%; for soil matrix results, less than 50%. Qualification of sample data is limited to that specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); and nondetects (UJ). Bias for these results cannot be determined.

In some USEPA regions a "Percent Solids" rule applies. For example, if a sediment contains less than 50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.2.6.9 Quantitation

Verify and record quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.0 SW846 NON-CLP ORGANICS BY GAS CHROMATOGRAPHY

2.1 Volatiles (SW 5030/SW 8011/8015B/8021A/8031)

2.1.1 Applicability

Method 8011 is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

1,2-Dibromoethane (EDB)
1,2-Dibromo-3-chloropropane (DBCP)

Method 8021A is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Allyl chloride
Benzyl chloride
Bis (2-chloroethoxy)methane
Bis (2-chloroisopropyl)ether
Bromoacetone
Bromobenzene
Bromodichloromethane
Bromoform
Bromomethane
Carbon tetrachloride
Chlorobenzene
Chloroethane
2-Chloroethanol
Chloroform
1-Chlorohexane

2-Chloroethyl vinyl ether
Chloromethane
Chloromethyl methyl ether
Chloroprene
4-Chlorotoluene
Dibromochloromethane
1,2-Dibromo-3-chloropropane
Dibromomethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethylene (Vinylidene chloride)
trans-1,2-Dichloroethylene
Dichloromethane
1,2-Dichloropropane
1,3-Dichloro-2-propanol
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Epichlorhydrin
Ethylene dibromide
Methyl iodide
1,1,2,2-Tetrachloroethane
1,1,1,2-Tetrachloroethane
Tetrachloroethylene
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethylene
Trichlorofluoromethane
1,2,3-Trichloropropane
Vinyl chloride
Benzene
Chlorobenzene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Toluene
Ethyl benzene
Xylenes (Dimethyl benzenes)

Method 8015B is used to determine the concentration of the following nonhalogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Diethyl ether	Acrolein	n-butyl Alcohol
Ethanol	Acetonitrile	t-butyl Alcohol
Methyl ethyl ketone (MEK)	Acetone	Methanol
Methyl isobutyl ketone (MIBK)	Allyl Alcohol	1,4-Dioxane

Method 8031 is used to determine the concentration of the following volatile organic compound in groundwater, liquid, and solid matrices:

Acrylonitrile

All of the above Methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). Method 8021A analyzes for halogenated and aromatic volatile organics via GC/HECP and GC/PID (Electro Conductivity Detector and Photoionization detector), Method 8015B analyzes for nonhalogenated volatile organics via GC/FID (Flame Ionization Detector), and Method 8031 analyzes for the compounds acrylonitrile using GC/FID. Samples can be analyzed by these methods using direct injection, the headspace method (Method 5021) or the purge-and-trap method (Method 5030B and 5035). Groundwater samples should be determined using Method 5030B.

2.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

2.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.1.4 Sample Preparation

Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. Because of the large variability and complicated matrices of waste samples detection limits for this method may vary widely among samples.

Method 5030 is a purge-and-trap method applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, groundwater, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 5035 is a purge-and-trap method applicable to nearly all types of soil samples, regardless of water content, including oily wastes, soils, and sediments.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

2.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

2.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

2.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!). Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recoveries.

2.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.1.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.1.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.1.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.2 Semivolatiles (SW8041/8061A/8091/8310)

2.2.1 Applicability

Method 8041 is used to determine the concentration of the following phenolic compounds in groundwater, liquid, and solid matrices:

Phenol
2-Chlorophenol
2,4-Dichlorophenol
2,6-Dichlorophenol
Trichlorophenols
Tetrachlorophenols
Pentachlorophenol
Cresols (methyl phenols)
4-Chloro-3-methylphenol
2,4-Dimethylphenol
2-Nitrophenol
4-Nitrophenol
2,4-Dinitrophenol
2-sec-Butyl-4,6-dinitrophenol (DNBP)
2-Cyclohexyl-4,6-dinitrophenol
2-Methyl-4,6-dinitrophenol

Method 8061A is used to determine the concentration of the following phthalate esters in groundwater, liquid, and solid sample matrices:

Benzyl butyl phthalate
Bis(2-ethylhexyl)phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Diethyl phthalate
Dimethyl phthalate

Method 8091 is used to determine the concentration of the following nitroaromatic and cyclic ketone compounds in groundwater, liquid, and solid sample matrices:

Nitrobenzene
Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Isophorone
Naphthoquinone

Method 8310 is used to determine the concentration of the following polynuclear aromatic hydrocarbons (PAHs) in liquid and solid sample matrices:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene

Benzo(ghi)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Naphthalene
Phenanthrene
Pyrene

All of the above methods are gas chromatographic (GC), with the exception of Method 8310 which is a High Performance Liquid Chromatography (HPLC) technique, only (i.e., no mass spectrometer detector is employed). These methods use either an electron capture detector (ECD), a flame ionization detector (FID), a ultraviolet detector (UV), or a fluorescence detector.

2.2.2 Interferences

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source depending upon the waste being sampled. While general cleanup techniques such as Method 3530 are provided as part of these methods, unique samples may require additional cleanup.

If sample or matrix interferences occur, a secondary column may be employed in addition to the primary column so as to resolve any questionable compound results.

2.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using Method 3510 (separatory funnel extraction) or Method 3520 (continuous liquid-liquid extraction). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or Sonication (Method 3550) procedures.

2.2.5 Data Overview Prior to Validation

Before commencing validation the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.

- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

2.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water samples: 7 days
  - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Generally, positive results affected by noncompliances are qualified as estimated (J); nondetects (UJ). These results are considered to be biased low. Alternately, the bias qualifiers L and UL may be used. Nondetects may be rejected (R) when the sample was extracted after 14 days (28 days for solid samples). If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Refer to the appropriate data validation protocol for specific guidance.

2.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to

qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30%, and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or whose %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.2.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are

generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recovery.

2.2.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.2.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated, and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.3 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides (SW 8081A/8082/8141A/ 8151A)

2.3.1 Applicability

Methods 8081A/8082 are used to determine the concentration of the following organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices:

Aldrin
alpha-BHC
beta-BHC
delta-BHC
gamma-BHC (Lindane)
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
Aroclor-1016
Aroclor-1221
Aroclor-1232
Aroclor-1242
Aroclor-1248
Aroclor-1254
Aroclor-1260

Similarly, Method 8141A is used to determine the following pesticides in groundwater and waste samples:

Azinphos methyl
Bolstar (Sulprofos)
Chlorpyrifos
Coumaphos
Demeton-O
Demeton-S
Diazinon
Dichlorvos
Disulfoton
Ethoprop
Fensulfothion
Fenthion
Merphos
Mevinphos
Naled

Parathion methyl
Phorate
Ronnel
Stirophos (Tetrachlorvinphos)
Tokuthion (Prothiofos)
Trichloronate

Note that when Method 8141A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique if mass spectroscopy is not employed.

Method 8151A is used to determine the following chlorinated acid herbicides in groundwater and waste samples:

2,4-D
2,4-DB
2,4,5-T
2,4,5-TP (Silvex)
Dalapon
Dicamba
Dichloroprop
Dinoseb
MCPA
MCPP
4-Nitrophenol
Pentachlorophenol

Since these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A includes a hydrolysis step to convert the herbicide to the acid form prior to analysis. When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column; alternately, the compounds of interest can be confirmed by detection via a mass spectrometer.

All of the above Methods are Gas Chromatographic (GC) in which sample extracts are analyzed by direct injection. Methods 8081A and 8082 analyze for organochlorine pesticide compounds and PCBs via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used). Method 8141A analyzes for organophosphorous pesticide compounds via GC/FID (Flame Ionization Detector), and Method 8151A analyzes for chlorinated herbicide compounds via GC/ECD (alternately, a Microcoulometric Detector or Hall Electrolytic Conductivity Detector may be used).

2.3.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably, and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

2.3.3 General Laboratory Practices

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicate and laboratory duplicates should also be employed.

Note that herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, when performing Method 8151A, glassware and glass wool must be acid-rinsed and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

2.3.4 Sample Preparation

Prior to the use of Methods 8081, 8082, and 8141A, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid- liquid extractor (Method 3520). Solid samples are extracted with hexane:acetone (1:1) using either the Soxhlet extraction (Method 3540) or sonication (Method 3550) procedures.

Method 8151A provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetone and diethyl ether followed by esterification using diazomethane as a derivatizing agent.

2.3.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.

- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.3.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.3.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  Water samples: 7 days
  Solid samples: 14 days
- Analysis: 40 days from date of extraction

When the holding time criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected (R). These results are biased very low.

2.3.6.2 Calibration

Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

Method 8081A requires analysis of a DDT/Endrin breakdown check standard. The DDT/Endrin Breakdown should not exceed 20%. Generally, if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE and DDD following the in-last control standard until the next in-control standard (see analytical sequence). If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) nondetects for DDT in associated samples. Generally, if Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable standard. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) nondetect Endrin results.

2.3.6.3 Blank Contamination

When using the information provided below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific, and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; guidelines provided in the appropriate data validation protocol should be followed.

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

2.3.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs; circle any recoveries outside these limits on your working copies of these Forms.

No qualifications are made for surrogates which show zero recoveries because they were "diluted out." Generally, positive results affected by low surrogate recovery are qualified as estimated (J) or the (L) bias qualifier is used when applicable; nondetects are qualified (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; nondetects are not qualified based on high surrogate recovery. Because the surrogate recovery limits for these fractions are advisory, generally no results are rejected.

The pesticide/PCB surrogates decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII or equivalent must be 0.10 for DCB and 0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peak. The validator's professional judgment for qualifications should be used.

2.3.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate data validation guidelines for specific procedures for evaluating MS/MSD analyses.

2.3.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.3.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10%.

2.3.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.4 Explosives/Nitroaromatics/Nitroamines(SW 8330)

2.4.1 Applicability

Method 8330 is used to determine the concentration of the following explosives, nitroaromatics, and nitroamines in water, soil, or sediment matrices:

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
1,3,5-Trinitrobenzene (1,3,5-TNB)
1,3-Dinitrobenzene (1,2-DNB)
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
Nitrobenzene (NB)
2,4,6-Trinitrotoluene (2,4,6-TNT)
4-Amino-2,6-dinitrotoluene (4-Am-DNT)
2-Amino-4,6-dinitrotoluene (2-Am-DNT)
2,4-Dinitrotoluene (2,4-DNT)
2,6-Dinitrotoluene (2,6-DNT)
2-Nitrotoluene (2-NT)
3-Nitrotoluene (3-NT)
4-Nitrotoluene (4-NT)
Nitroguanidine
Nitroglycerin
Pentaerythritol Tetranitrate (PETN)

The analysis of the compounds listed above is conducted by High Performance Liquid Chromatography equipped with a 254 nm Ultra Violet (UV) detector. This method is capable of determining part per billion (ppb) detection levels in water and soil matrices.

The method requires the use of both a primary (C-18 reverse phase) and a confirmation (CN reverse phase) column.

2.4.2 Interferences

The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

2,4-Dinitrotoluene and 2,6-dinitrotoluene may co-elute. High concentrations of one of the two isomers may cause interference of the other isomer. In instances where this is applicable, both isomers should be reported as one. Baseline resolution should be present for all compounds.

Decomposition of Tetryl occurs rapidly and when exposed to heat. Samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

2.4.3 General Laboratory Practices

Method blanks and instrumentation blanks should be conducted to access laboratory contamination.

Matrix spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field and laboratory duplicates may also be employed.

2.4.4 Sample Preparation

Method 8330 provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetonitrile and a salting-out procedure for aqueous samples. Soil samples are air dried prior to preparation, thus percent moisture is not a consideration when calculating compound concentrations.

2.4.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package.
- If each sample was correctly analyzed and identified for the specified parameters.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and /or re-analyses, the validator should preview the data package contents to determine which analyses represent the best data quality.

Unless specifically directed by the client, never annotate the laboratory data package. Before beginning evaluation, prepare working copies(i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.4.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols, method requirements, and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this SOP is only intended as a general procedure for the data validation task.

Deficiencies, omissions, and/or other anomalies noted during the review require the data validator to contact the laboratory.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.4.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated form the date of collection to the date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
  - Water Samples: 7 days
  - Solid Samples: 14 days
- Analysis: 40 days from date of extraction

When the holding times criteria are not met, positive results in affected samples are generally qualified as estimated, (J); nondetected results, (UJ). These results are considered biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If holding times are exceeded by a factor of two or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected, (R). These results are considered to be biased very low.

2.4.6.2 Calibrations

Data pertaining to the initial calibration (i.e. evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels. The initial calibration should consist of a minimum of five concentration levels for each compound of interest.

In general, either the correlation coefficient (r) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is < 0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have %RSDs greater than 20%. Generally, associated data are qualified as estimated (J/UJ) if the calibration %RSD is >20%. Circle these noncompliances on working copies of calibration forms.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with each initial calibration by instrument. Write the affected samples on working copies of the appropriate continuing calibration forms. Spot-check(i.e. recalculate) a few of the %Ds to verify the laboratory's computation.

A continuing calibration or daily calibration must be performed at the beginning, midpoint and end of the analytical sequence. The continuing calibration response factor for each analyte must be compared to the response factor of the initial calibration. The continuing calibration response factor must agree within 15% of the initial response factor. Generally, positive and nondetected results are qualified as estimated (J/UJ) if the Percent Difference (%D) is >15%.

2.4.6.3 Blank Contamination

A review of all method and instrument blanks (if provided) is conducted to evaluate laboratory contaminants. An additional review of all relevant field quality control blanks is also conducted. Contaminants, if present, are summarized and the maximum concentration of each contaminant is selected and used to establish blank action levels.

An action level of 5X the maximum amount of each contaminant is used to evaluate sample data. Blank action levels must consider the aliquot used for analysis and sample dilution. Positive results less than the action level are qualified as false positives. The manner in which the qualifiers are applied varies [i.e., use of (U) or (B); replacement by the Reporting Limit]. General regional guidance procedures dictate the most appropriate validation action qualification.

2.4.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II or equivalent and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs. Circle any recoveries outside these limits on working copies.

Generally, positive results affected by low surrogate recoveries are estimated, (J) or (L), indicating low bias; nondetected results are qualified, (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated, (J), or the bias qualifier (K), is used when applicable. Nondetected results are not qualified based upon high surrogate recoveries. It should be noted that consideration of interferences may affect surrogate recoveries. If a trend of noncompliance is noted, an evaluation of sample chromatograms should be conducted when surrogate recoveries are noncompliant and a matrix effect is suspected.

No qualifications are made for surrogates which have been diluted out.

Generally, positive results associated with surrogate recoveries <10% are qualified as estimated, (J) or biased low (L). Nondetected results associated with surrogate recoveries <10 are considered unreliable and are qualified rejected (R).

2.4.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the unspiked sample. Typically, Percent Recoveries (%Rs) and the Relative Percent Difference (RPD) are evaluated based upon the laboratory provided control limits.

2.4.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked samples results with MS/MSD analyses result for unspiked compounds. Consider nondetected results and results reported at concentrations less than the reporting limit to be in agreement. Use professional judgment in determining whether to qualify sample results based upon the comparison. The comparison may be presented in terms of a %RSD or an RPD.

Likewise, compare positive compound results for field duplicate samples. Generally, an RPD between field duplicate results for the aqueous matrix should be <35%; for soil matrix results <50%. Qualification of the sample data is limited to specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetected results (UJ)

2.4.6.7 Quantitation

Verify and record the quantitation of at least one compound per fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. The validator and laboratory quantitations must agree within 10%. If quantitation differences are significant, the laboratory must be contacted to investigate and resolve the discrepancy.

2.2.4.7 Deliverable Guidance

In addition to any specific USEPA Regional requirements (i.e., data validation memorandum, data summary spreadsheets, USEPA Regional Worksheets), all laboratory data package quality summary forms, sample Form I reports method blank results and the Chain of Custody records must be included in the validation report.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the narrative is free of transcription and typographical errors before submitting all requested items for quality assurance review.

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1.0 INORGANICS (SW-846 6010B/7470A/7471A/9010A&B/7470/9010)

Inductively Coupled Plasma Emission Spectroscopy (ICP) - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium.

Cold Vapor Methodology - Mercury is commonly analyzed using cold vapor methodology.

Automated Colorimetric Technique - Cyanide is commonly analyzed using automated colorimetric methodology.

1.1 Applicability

These methods are applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

Detection limits for analytes are established on a quarterly basis and are both laboratory and instrument specific.

1.2 Data Overview Prior to Validation Process

1.2.1 Data Completeness

The data reviewer must initially verify that all forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required forms will include:

Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Calibration Summary (Form 2A or equivalent).

Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the blank summary (Form 3 or equivalent) (note, filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).

Verify that all ICP analytes are present in both ICSA and ICSAB solutions. Also, verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns.

Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels.

Verify that laboratory duplicate analyses were performed for each matrix. **NOTE**: Field quality control blanks are never to be designated for quality control analyses.

Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE**: An aqueous LCS is not required for mercury and cyanide analysis.

The Method of Standard Additions (MSA) (Form 8 or equivalent) may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 4.1.3.11 for further details.

Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE**: Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12 (or equivalent). Verify that no GFAA analytical results exceed the highest standard in the associated GFAA calibration.

Verify that the Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillation as applicable.

Examine the Form 14s (or equivalent) to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

1.3 Technical Evaluation Summary

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

1.3.1 Holding Times

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

Metals - 6 months; pH <2
Mercury - 28 days; pH <2
Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only. Solid samples do not receive preservative but require maintenance at 4 C (2 C) during shipment and storage.

The above holding times do not apply to leachate analyses. It is suggested that the data reviewer reference SW-846 Method 1311 for any questions regarding TCLP quality control requirements and analytical procedural requirements; these vary significantly from non-TCLP analyses.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE**: Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard
- **GFAA analyses** - must employ a blank and at least three standards. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e. r = 0.995 or greater)
- **Mercury analyses** - must employ a blank and at least three standards (r = 0.995 or greater).
- **Cyanide analyses** - must employ a blank and at least three standards (r = 0.995 or greater). **NOTE:** At least two additional standards (a high or low) must be distilled and compared to similar values on the curve. Values of distilled standards should agree within 10% of undistilled standards.

1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

The ICV/CCV %R quality control limits are 90-110% for ICP metals, 80-120% for GFAA metals and mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated, (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects remain unaffected. **NOTE:** Qualify results of only those samples associated with the noncompliant ICV or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

1.3.4 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours which ever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in a sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration

present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

1.3.5 ICP Interference Check Sample Results

Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. **NOTE:** Affected samples include all samples analyzed between the initial and final solutions or within the eight hour working shift whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 or equivalent and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample, while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\text{Estimated elemental intf.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}]\,[\text{Conc. Sample}]}{\text{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (**NOTE**: Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

1.3.6 Matrix Spike Sample Analysis (Pre-digestion)

Verify that at least one matrix spike was performed for each matrix for a given set of samples (maximum of 20 samples) within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Any deviations from the referenced method shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant, no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

1.3.7 Laboratory Duplicate Precision

Verify that one duplicate sample analysis was performed for each group of samples (maximum of 20 samples) of a similar matrix within an SDG. Control criteria used to evaluate the aqueous laboratory duplicates are as follows:

- a control limit of 20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of 35% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: Review Duplicate Summary (Form 6 or equivalent) carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

1.3.8 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances were field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

- a control limit of 30% for relative percent difference when sample and duplicate results are >5X CRDL

- a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a control limit of 50% for relative percent difference when sample and duplicate results are >5X CRDL

- a control limit of 4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, field duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

1.3.9 Laboratory Control Sample Results

Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. NOTE: An aqueous LCS is not required for mercury and cyanide analysis. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R <80%, qualify as estimated (J) positive results and (UJ) nondetects, If aqueous LCS %R >120, qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

1.3.10 Method of Standard Additions (MSA)

Review MSA Form 8 or equivalent and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance in the method.

Actions - If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.

1.3.11 ICP Serial Dilution Analysis

Verify that all ICP analytes are included on the Form 9 (or equivalent) with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50 IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of suppressed results exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

1.3.12 Analysis Run Logs Form 14

The Form 14 or equivalent serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify GFAA PDS percent recoveries within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS % R.

1.3.13 Further GFAA Evaluations

It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

1.4 <u>Deliverables Guidance</u>

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

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1.0 PURPOSE

To establish procedures for the use, maintenance, and calibration of the Photovac 2020 Photoionization Air Monitor.

2.0 SCOPE

Applies to each usage of the Photovac 2020 Photoionization Air Monitor by TtNUS personnel.

3.0 GLOSSARY

Electron volt (eV) - A unit of energy equal to the energy acquired by an electron when it passes through a potential difference of 1 volt in a vacuum. It is equal to $1.602192 \pm 0.000007 \times 10^{-19}$ volts.

Intrinsically Safe (I.S.) - Based on wiring, configuration, design, operation, gasketing, construction, this instrument may be employed within locations in which flammable gases and/or vapors may exist.

Ionization Potential (I.P.) - The energy required to remove an electron from a molecule yielding a positively charged ion and a negatively charged free electron. The instrument measures this energy level.

Photoionization Detector (PID) - Photoionization detector employed as general reference to air monitors of this type. PIDs detection method employs ultraviolet (UV) radiation as an energy source. As air and contaminant are drawn through the ionization chamber the UV light source causes the contaminant with ionization potentials equal to or less than the UV source to break into positive and negatively charge ions. The created ions are subjected to an electrostatic field. The voltage difference is measured in proportion to the calibration reference and the concentration of the contaminant.

Ultraviolet Radiation (UV) - Ultraviolet radiation is the energy source employed by the instrument to ionize collected sample gas streams. The UV lamp source is required to be equal to or greater than the ionization potential of the substance drawn through the instrument in order to create separate ionized species.

4.0 RESPONSIBILITIES

Office Managers – Office Managers are responsible for ensuring that personnel under their direction who may use this device are first provided with adequate training and information.

Project Managers – Project Managers are responsible for ensuring that appropriate health and safety requirements and resources are addressed for their assigned projects.

Health and Safety Manager (HSM) - The HSM shall ensure that appropriate training is available to users of the Photovac 2020 instrument.

Equipment Manager - The Equipment Manager shall ensure all air monitoring instrumentation slated for field activities has been operationally checked out, fully charged, and calibrated prior to issuing any instrument for field service. Maintenance deficiencies identified by the Equipment Manager will require those instruments to be pulled from service until repairs can be facilitated.

Field Operations Leader (FOL)/Field Team Leader (FTL) - The FOL/FTL shall ensure all field team members employing the monitoring instruments as part of their assigned duties are adequately trained in

the operation and limitations of this instrument. The FOL/FTL shall ensure that the air monitoring instruments are employed as directed by site guidance documents (i.e., Work Plan, Health and Safety Plan, etc.). Additionally, the FOL/FTL shall ensure that the appropriate documentation and recordkeeping requirements are fulfilled including Documentation of Calibration and Direct Reading Instrument Response Data Sheets for air monitoring activities. On projects where a dedicated SSO is not assigned, the FOL/FTL is responsible for assuming the duties of that position.

Health and Safety Officer (HSO) - The HSO is responsible for determining air monitoring requirements for the site activities, and providing direction for air monitoring during specific site activities. This identification of types of air monitoring and direction for use are indicated within the Site-Specific Health and Safety Plan (HASP).

Site Safety Officer (SSO) - The SSO shall ensure the instruments identified are employed in the manner directed by the HSO, and that any action levels specified are observed for the application of engineering controls, personal protective equipment (PPE) use, and administrative controls. Additionally, he/she shall ensure the instruments are properly maintained and calibrated prior to use in the field. The SSO, during specific air monitoring applications including STEL and TWA mode measurements, will be responsible for the operation and application of this specialty air monitoring device. The SSO is also responsible for addressing relevant Hazard Communication requirements (e.g., MSDS, chemical inventories, labeling, training, etc.) on each assigned project.

5.0 PROCEDURES

5.1 Principle of Operation

Direct-reading instruments such as a photoionization detector are typically used to monitor for airborne releases that could present an inhalation threat to personnel, and to screen and bias environmental samples. Proper use of these instruments by trained, qualified personnel is essential to the validity of any acquired results. Also essential is that the devices are properly calibrated according to manufacturers instructions (and the specifications of this SOP), and that users of the instrument properly document results.

The Photovac portable photoionizer detects many organic (and a few inorganic) vapors. The basis for detection of this instrument is the ionization of components of captured gaseous streams. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many gaseous compounds. Molecules are transformed into charged-ion pairs, creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively-charged ion and the free electron. The instrument measures this energy level.

This instrument measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual substances. Readings displayed represent the total concentration of all photoionizable chemicals present in the sample. This instrument is factory set to display concentration in units of ppm or mg/m^3.

The 2020 instrument is easy to operate. The meter display updates itself once per second. Concentrations are directly displayed on the readout.

The 2020 instrument also performs short-term exposure limit (STEL), time-weighted average (TWA) and PEAK calculations. Any of these results can be viewed, but only one mode may be viewed at a time.

2020 has 6 keys for alphanumeric entry and for accessing multiple functions. The keys are used to set up and calibrate the 2020 instrument. They allow for the manipulation of the data in various ways.

All information entered with the keys and stored in the instrument's memory. This is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time the instrument is turned on.

5.1.1 Displays

The 2020 instrument has a meter display for reporting detected concentration, and a display used to indicate status information and guide the user through configuration options. All functions of the instrument will be controlled or reported using one of these displays.

5.1.1.1 Meter Display

The meter has a 4-digit display. It will always be used for reporting detected concentrations. When the detector and pump are off, the meter display will be blank.

In order to accommodate the entire range of concentrations the 2020 can detect, the instrument has 2 resolution settings. The 0.1 resolution setting should be used for concentrations below 100 ppm, and the 1 resolution setting should be used for concentrations above 100 ppm.

5.1.1.2 Status Display

The status display is a 2 line by 16 character display. The top line is used to display status information and prompts the user for information. The bottom line is used for soft key names. Up to 3 names can be displayed for the 3 soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

5.1.2 Keys

5.1.2.1 Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key, and the last key is the ENTER key.

The ON/OFF key is used both to turn power on to the 2020 as well as to turn the power off. To turn on 2020, press the ON/OFF key. To turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent an accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow the user to advance and the EXIT key provides a way to go back. At the initial entry of the menu, EXIT will return the user to the default display.

The ENTER key has a context sensitive function. When operating or navigating through the function map, the ENTER key is used to exit the functions and return to the default display. When entering data such as a name, number, date, or time, ENTER is used to confirm the entry.

5.1.2.2 Soft Keys

The three soft keys on the 2020 are located directly below the status display. Each key has varying functions for configuring the 2020, editing the data logger, and controlling the display. Since only three soft keys are available, each function is broken down into a path.

5.1.2.3 Entering Text With the Soft Keys

For all information that must be entered, the left, center, and right soft keys correspond to the up, down, and right arrow.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character to the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the change, press the EXIT key.

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in the date are skipped when advancing the cursor.

All inputs 8 characters long, which is displayed on the right side of the status display line. The prompt, describing the input, occupies the left side of the top line. The soft keys are defined on the bottom line of the status display.

5.2 Default Display

The meter display shows the detected concentration. The resolution of the display automatically changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm or mg/m^3. A reading greater than 99.9 will be shown with a resolution of 1 ppm or mg/m^3. The meter will display concentrations up to 2000 ppm or 2(XX) mg/m^3.

The status display is used to indicate the instrument status, date, time, units, and active soft keys.

The default display provides the following information: instrument status, current detected concentration, time, date, and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

5.3 Monitoring

5.3.1 Use and Documentation of Results

As with any direct-reading instrument, understanding not only how – but when to use this instrument is essential to gathering relevant and valid data. This device will only respond to volatile substances in sampled air that have ionization potentials below the UV lamp strength. Inappropriate instrument selection, or use/interpretation of instrument results by an unqualified user not only can yield inaccurate

results, but could place personnel at risk of exposure to hazardous agents. Only personnel who are properly trained and authorized to use this device will be permitted to operate it.

It is essential that instrument operators understand and comply with the requirements to document results. This includes the need to document calibration results as well as operational readings. Calibration results must be recorded using Figure 5-1. Operational results can be recorded in several ways, including:

- Direct-Reading Instrument Response Data (Figure 5-2) – preferred method
- Boring Log Forms (Figure 5-3)
- Test Pit Log Forms (Figure 5-4)
- Log book entries

When using direct-reading instruments, it is important to monitor the air near the source of potential releases (e.g., drilling boreholes, tank entrances, drum openings, etc.) and at worker breathing zone areas. All readings should be recorded, including readings noted where background levels were not exceeded.

5.3.2 Instrument Status

The instrument status is shown on the left side of the first line of the status display and on the Table and Graph outputs. Each status has an assigned priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

5.3.3 Alarms

While operating the instrument, any one of three audible alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status.

The 2020 also has an audible alarm and a visual alarm LED. To conserve power, the 2020 alternates between these two alarm indicators, rather than operating both concurrently. Different alarms are identified by the frequency at which the 2020 alternates as follows: PEAK alarm-5 times per second; STEL alarm-2.5 times per second; and TWA alarm-1.25 times per second.

The left soft key is used for acknowledging alarm conditions, and is named "Ack." If no alarm conditions exist , then the "Ack" key is not shown. To clear an alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

The 2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and if exceeded, an alarm is triggered.

If the 15 minute average exceeds the selected STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average of concentration, since the TWA was last cleared, has exceeded the TWA exposure limit.

FIGURE 5-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ PROJECT NO.: _____

Date of Calibration	Instrument Name and Model	Tetra Tech Instrument I.D. Number	Person Performing Calibration	Instrument Settings/Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration Setting	Post-Calibration Reading		

FIGURE 5-2

DIRECT-READING INSTRUMENT RESPONSE DATA

SITE NAME: _____

PROJECT NO.:_____

PAGE OF

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn
Example 12-12-91/14:30	Joe Field	HNU 101	15 ppm/15 min.	breathing zone	sample location no. 12	Jim Technician/Level B George Grunt/Level B

FIGURE 5-2 (Continued)

SITE NAME: _____

PROJECT NO.: _____

PAGE _____ OF _____

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn

FIGURE 5-3

BORING LOG

Page ___ of ___

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

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

* When rock coring, enter rock brokeness.

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

Drilling Area Background (ppm): ____

Remarks: _____

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

FIGURE 5-4

Page ___ of ___

TEST PIT LOG

PROJECT NAME: _____ TEST PIT NUMBER: _____
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: _____

During calibration, all alarms are disabled. Once the calibration is complete the alarms are re-enabled.

5.4 STEL, TWA, MAX, and PEAK Operation

The 2020's display can be configured to show one of four values: STEL, TWA, PEAK, and MAX.

5.4.1 Short-term Exposure Limit (STEL) Mode

The Short-term Exposure Limit (STEL) mode displays the concentration as a 15 minute moving average. The 2020 maintains 15 samples, each representing a one-minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new one minute average. This moving average provides a 15-minute average of the last 15 minutes with a one-minute update rate. Since the average is calculated using 15 one-minute averages, the meter display will only update once every minute.

STEL is set to zero each time the instrument is turned on. Since STEL is a 15-minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by the 2020. The results of the calculations can be displayed by selecting STEL as the Display mode.

5.4.2 Time-weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While in the TWA mode, the time on the status display will show the number of minutes and hours of data that TWA data has been accumulated. When the sample time reaches 8 hours, the 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by the 2020. The results of the calculations can be displayed by selecting TWA as the Display mode. When the sampling period is less than 8 hours, record the TWA readout along with the sampling duration displayed on the meter.

5.4.3 MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is shown on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if you display the MAX reading, and you press "Clr," only the MAX value is cleared. The TWA is still accumulating in the background.

5.4.4 PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated every second. In the background, the 2020 data logger is sampling the concentration and measuring minimum, maximum, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the data logger until the data logger is full. Typically, the instrument is operated in the PEAK mode. Operation within the other specialized modes are the responsibility of the SSO.

5.5 Set Functions

Pre-set functions are used to setup the 2020. There are three functions which can be used: Calibration, Pump and Clock.

5.5.1 Pump

The Pump function is used to control the pump. After selecting Set Pump, the 2020 responds by displaying the new pump status.

The detector is also turned off when turning the pump off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and the 2020 will only be used for reviewing data or generating reports. Operating the instrument with the pump and detector turned off will conserve the lives of the battery and ultraviolet (UV) lamp.

1. Press the ENTER key. The top line of the status display changes to "Select?". The bottom line displays 3 soft key names: "Set," "Log," and "Disp."

2. Press the soft key below "Set."

3. The names of the soft keys change to reflect the Set options. The display now shows 3 devices which can be set: "Clock," "Pump," and "Cal." Press the "Pump" key.

4. If the pump is off, pressing the "Pump" key will turn the pump on.

5. A message will be displayed showing the status of the pump. The display reverts back to the previous menu after a few seconds.

6. To return to the default display, press the ENTER key.

5.5.2 Clock

The Clock function is used to set both the current date and time.

1. Press the ENTER key.

2. Press the "Set" key.

3. When the names of the soft keys change, press the "Clock" key.

The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character to the right. When the cursor is advanced past the right-most character, it wraps around to the first character again.

Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.

5. Press the ENTER key to confirm the time and move to the date option.

6. When setting the date, the 2020 prompts the user to input the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.

7. Press the ENTER key to confirm the date and return to the Set options.

5.5.3 Calibration (Cal)

The Cal function allows the user to setup and calibrate the 2020. Three options are available under the Cal function: "Zero," Span," and "Mem."

A calibration memory consists of a name, a response factor, and PEAK, TWA, and STEL alarm levels.

The "Zero" and "Span" keys are covered in detail in the manufacturer's operations manual for the instrument.

To edit the calibration memory, select "Mem" and then "Chng." The 2020 prompts the user with two new soft keys: "User" and "Lib."

5.5.4 Library (Lib)

Library selections simplify Cal Memory programming, and provide standard response factors for approximately 70 applications. "Lib" allows you to select an entry from a pre-programmed library. The name, response factor, and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set," "Cal," "Mem," "Chng," and "Lib."

2. Use the "Next" and "Prev" keys to scroll through the list. See the manufacturer's manual for a list of the library entries.

5.6 Preparing for Field Operation of the Photovac 2020

Turning 2020 On

1. Turn the 2020 on by pressing the ON/OFF key.

2. The instrument will display the software version number. Wait for the 2020 to proceed to the default display.

3. Allow 10 minutes for the instrument to warm up and stabilize.

4. Press the "Enter" Key. The default display will provide 3 soft key selections: "Set," "Log," and "Display."

5. Press "Set." From this option 3 other soft key selections will be offered: "Pump," "Clock," and "Cal."

6. Press "Cal." This will begin the calibration sequence. The first selection is to Zero the instrument.

7. Press "Enter," zeroing will begin. (Note: When employing zero gas attach and activate zero gas supply at this time.)

8. The next selection is for Span determination. Press "Enter" and the concentration will be requested. The isobutylene calibration gas employed under general service will be marked on the side of the container. Use the soft keys to toggle into position and to log the concentration. Once the concentration is logged press "Enter." The direction or status display will indicate spanning. At this time hook up the span gas with a regulator to the Photovac 2020, and open it to supply enough flow to elevate the flow rate indicator to the green indicator line (1/8" from the rest position).

9. Once spanning is complete, the alarms which have been disabled during calibration will activate indicating that calibration is complete.

10. Document this calibration procedure using a Documentation of Calibration form as illustrated in Figure 5-1.

This instrument is ready for use.

Calibration is to be performed daily or prior to each use in accordance with Sections 5.6 and 5.7 of this SOP, and with manufacturer's recommendations.

5.7 Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use. Complete Figure 5-4 for each calibration.
Factory Inspection and Calibration	Once a year, or when malfunctioning
Wipe Down the Outer Casing of the Unit	After each use
Clean UV Light Source	Every 24 hours of operation
Sample Inlet Filter	Change on a weekly basis or as required by level of use
Battery charging	After each use
Clean ionization chamber	Monthly

5.7.1 Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position. Use the instrument's multi-tool and remove lamp housing cover.

2. Tilt the lamp housing with one hand over the opening, slide the lamp out of the housing.

3. The lamp window may now be cleaned with any of the following compounds using lens paper:

   a. 11.7 eV Lamp - Dry Aluminum Oxide Powder (3.0 micron powder)
   b. All other lamps - HPLC Grade Methanol

   Observe manufacturer's MSDS requirements when handling these substances.

4. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using the multi-tool. (Do not over tighten).

5. Recalibrate as per Section 5.6.

5.7.2 Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and remove the lamp housing cover and lamp as per Section 5.7.1.

2. Using a gentle jet of compressed air, gently blow out any dust or debris.

3. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using the multi-tool. (Do not over tighten).

4. Recalibrate as per Sections 5.6 and 5.7.

5.8 Instrument Advantages

The Photovac 2020 is easy to use in comparison to many other types of monitoring instrumentation. Its detection limit is in the low parts-per-million range. Response time quickly reaches 90 percent scale of the indicated concentration (less than 3 seconds for benzene). This instrument's automated performance covers multiple monitoring functions simultaneously, and incorporates data logging capabilities.

5.9 Limitations of the Photovac 2020 Photoionization Monitor

- Since the 2020 is a nonspecific total gas/vapor detector, it cannot be used to identify unknown chemicals; it can only quantitate them in relationship to a calibration standard (relative response ratio).

- For appropriate application of the 2020, ionization potentials of suspected contaminants must be known.

- Because the types of compounds that the 2020 can potentially detect are only a fraction of the chemicals possibly present at a hazardous waste site, a background or zero reading on this instrument does not necessarily signify the absence of air contaminants.

- The 2020 instrument can monitor only certain vapors and gases in air. Many nonvolatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected.

- PID's are generally not compound-specific. Their response to different compounds is relative to the calibration gas used. This is generally referred to as the relative response ratio. Instrument readings may be higher or lower than the true concentration. This can be especially serious when monitoring for total contaminant concentrations if several different compounds are being detected at once.

- The 2020 is a small, portable instrument which cannot be expected to yield results as accurately as laboratory instruments.

5.9.1 Variables Affecting Monitoring Data

Monitoring hazardous waste site environments can pose a significant challenge in assessing airborne concentrations and the potential threats to site personnel. Several variables may influence both dispersion and the instrument's ability to detect actual concentrations. Some of the variables which may impact these conditions are as follows:

- Temperature Pressure - changes in temperature and/or pressure will influence volatization, and effect airborne concentrations. Additionally, an increase or decrease in temperature ranges may have an adverse effect on the instrument's ability to detect airborne concentrations. Significant changes in temperature or pressure from the time of calibration to the time of sample measurement may result in erroneous results.

- Humidity - excessive levels of humidity may interfere with the accuracy of monitoring results.

- Rainfall - through increased barometric pressure and water may influence dispersion pathways effecting airborne emissions.

- Electromagnetic interference - high voltage sources, generators, other electrical equipment may interfere with the operation and accuracy of direct-reading monitoring instruments.

6.0 TROUBLESHOOTING

6.1 Fault Messages

When the "Fault" status is displayed, the 2020's operation is comprised.

Fault 1: Signal from zero gas is too high.

Cause: If another fault occurred while the 2020 was setting its zero point, then this fault is displayed.

Action: Ensure no faults are occurring and calibrate the 2020 again.

Cause: Contamination of sample line, sample probe or fittings before the detector.

Action: Clean or replace the sample line, sample probe or the inlet filter.

Cause: Span gas and zero air are mixed up.

Action: Ensure clean air is used to zero the 2020. If you are using Tedlar bags, mark the calibration and zero gas Tedlar bags clearly.

Cause: Ambient air is contaminated.

Action: If the quality of ambient air is unknown, use a supply of commercial zero grade air.

Fault 2: Signal from span gas is too small.

Cause: Operator may have incorrectly used the span gas for the zero air source.

Action: Ensure clean air is used to zero the 2020. If you are using Tedlar bags, mark the calibration and zero gas Tedlar bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note: Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with the 2020.

Fault 3: UV lamp fault. UV lamp has not started.

Cause: UV lamp has not started immediately.

Action: This fault may be seen momentarily when the 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: UV lamp serial number label is blocking the photocell.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: The UV lamp has a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90 degree and then try to start the 2020 again. If the fault persists, replace the lamp.

Cause: UV lamp not installed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp.

Cause: UV lamp has failed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Fault 4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Action: Ensure the sample outlet, located on the underside of the 2020, is not obstructed.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: The pump has failed.

Action: Contact the Equipment Manager.

6.2 Specific Problems

Problem: Very low or no instrument response detected, yet compounds are suspected to be present.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in the User's Manual.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Equipment Manager.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Disconnect the battery charger before calibrating the 2020.

Cause: Calibration Memories have not been programmed correctly.

Action: Program all the calibration memories required for the intended application. You must use the correct calibration gas and concentration for each Cal Memory.

Cause: Response factor has been set to zero.

Action: Enter the correct response factor. Refer to the list of response factors. If the compound is not listed or you are measuring gas mixtures, then enter a value of 1.0. See User's Manual.

Cause: Not using the correct Cal Memory.

Action: Select the correct Cal Memory for the intended application.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detection lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly.

Action: Ensure the lamp cover has been tightened. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: Sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading.

The 2020 detector has been designed to operate under high humidity conditions. Under extreme humidity conditions, there may be a decreased response.

Cause: UV lamp is failing.

Action: Replace UV lamp.

Note: Do not remove or replace the detector lamp in a hazardous location.

Cause: High concentration of non-ionizable compounds suspected.

Action: Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly.

Application with high backgrounds of such materials, may be incompatible with the 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts which conduct a charge. The water vapor becomes an electrolytic solution which becomes ionized when it enters the detector.

Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants. The same effect may be seen when conducting ground water investigations in areas where the water contains a significant concentration of minerals.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Sections 5.6 and 5.7.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not contact the Equipment Manager.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories required for the intended application. The correct calibration gas and concentration must be used for each Cal Memory. See the User's Manual.

Cause: Not using the correct Cal Memory.

Action: Select the correct Cal Memory for the intended application. See the User's Manual.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Date and time settings are not retained.

Cause: The battery pack was removed before the 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack and reset the time and date. Ensure that the 2020 has been turned off before removing the battery pack.

Cause: The 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Connect the 2020 to the AC adapter and turn the instrument on. Turn the pump off. While the 2020 is running the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over."

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move the 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack or connect the 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on the 2020.

Cause: There is an undetermined problem.

Action: Reset the 2020. Leave the instrument on while disconnecting the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on the 2020, set the time and date and program all the calibration memories.

Action: Contact the Equipment Manager.

Problem: Sample flow rate is less than 300 ml/min.

Cause: Inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace inlet filter.

Cause: Inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet is not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the Equipment Manager.

Problem: Liquid has been aspirated.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the Equipment Manager.

Problem: Corrosive gases and vapors have been sampled.

Cause: The 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching of the window. If the 2020 is exposed to corrosive material, contact the Equipment Manager.

7.0 SHIPPING

- The Photovac may be shipped as cargo or carried on as luggage provided that there is no calibration gas cylinder accompanying the kit. When shipping or transporting the calibration gas, a Hazardous Materials (Dangerous Good) Airbill, including the information as stipulated in Figure 5-5 will be prepared. **Only personnel who have been properly trained are permitted to offer a hazardous material for shipment.** The "Shipping Hazardous Materials" course offered by Tetra Tech NUS is considered acceptable training for this purpose. Specific instructions on packaging, labeling, and otherwise preparing a hazardous material shipment are presented in the Student Manual that accompanies the course.

8.0 REFERENCES

Photovac 2020 Photoionization Monitor User's Manual, 1995.

Student Manual from "Shipping Hazardous Materials" course, Tetra Tech NUS, 1999.

FIGURE 5-5

FedEx *Dangerous Goods Airbill*

Sender's Copy
RETAIN FOR 1 YEAR
RETAIN THIS COPY FOR YOUR RECORDS

The World On Time

1 From (please print and press hard)

Date _____ Sender's FedEx Account Number _____

Sender's Name _____ Phone ( ).

Company _____

Address _____ Dept./Floor/Suite/Room

City _____ State _____ ZIP _____

2 Your Internal Billing Reference Information (Optional) (First 24 characters will appear on invoice)

3 To (please print and press hard)

Recipient's Name Tom Patton Phone (412) 262-4583

Company Tetra Tech NUS

Address Spring Run Road Extension, Suite 140 ☐ Check here if residence (Extra charge applies for FedEx Express Saver)
(To "HOLD" at FedEx location, print FedEx address here) (We Cannot Deliver to P.O. Boxes or P.O. ZIP Codes) Dept./Floor/Suite/Room

City Coraopolis State PA ZIP 15108

For HOLD at FedEx Location check here
☐ Hold Weekday ☐ Hold Saturday (Not available at all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

For WEEKEND Delivery check here (Extra Charge. Not available to all locations)
☐ Saturday Delivery (Available for FedEx Priority Overnight and FedEx 2Day only) ☐ NEW Sunday Delivery (Available for FedEx Priority Overnight only)

4a Express Package Service *Packages under 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Priority Overnight (Next business morning) ☐ FedEx Standard Overnight (Next business afternoon)
☐ FedEx 2Day (Second business day) ☐ FedEx Express Saver (Third business day)

4b Express Freight Service *Packages over 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Overnight Freight (Next business day) ☐ FedEx 2Day Freight (Second business day) ☐ FedEx Express Saver Freight (Up to 3 business days)
(Call for delivery schedule. Some restrictions apply. See back for detailed descriptions of freight services.)

Service Conditions, Declared Value, and Limit of Liability – By using this Airbill, you agree to the service conditions in our current Service Guide or U.S. Government Service Guide. Both are available on request. SEE BACK OF SENDER'S COPY OF THIS AIRBILL FOR INFORMATION AND ADDITIONAL TERMS. We will not be responsible for any claim in excess of $100 per package whether the result of loss, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely manner. Your right to recover from us for any loss includes intrinsic value of the package, loss of sales, interest, profit, attorney's fees, costs, and other forms of damage, whether direct, incidental, consequential, or special, and is limited to the greater of $100 or the declared value but cannot exceed actual documented loss. The maximum declared value for any FedEx Letter and FedEx Pak is $500. Federal Express may, upon your request, and with some limitations, refund all transportation charges paid.
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5 Packaging
☒ Other Packaging
Dangerous Goods cannot be shipped in FedEx packaging.

6 Special Handling
☒ Dangerous Goods as per attached Shipper's Declaration ☐ Cargo Aircraft Only

7 Payment
Bill to: ☐ Sender (Account No. in Section 1 will be billed) ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check
(Enter FedEx Account No. or Credit Card No. below)

FedEx Account No. _____

Credit Card No. _____ Exp. Date _____

Total Packages	Total Weight	Total Declared Value*	Total Charges
		$ .00	$

*When declaring a value higher than $100 per shipment, you pay an additional charge. See SERVICE CONDITIONS, DECLARED VALUE, AND LIMIT OF LIABILITY section for further information.

Signature Release Unavailable

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Page 1 of 1 Pages

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING
Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)
PASSENGER AND CARGO AIRCRAFT | ~~CARGO AIRCRAFT ONLY~~

Airport of Departure:

Airport of Destination:

Shipment type: *(delete non-applicable)*
NON-RADIOACTIVE | ~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification							
Proper Shipping Name	Class or Division	UN or I.D. No.	Packing Group	Subsidiary Risk	Quantity and Type of Packaging	Packing Inst.	Authorization
Compressed Gas N.O.S. (mixture Nitrogen and Oxygen)	2.2	UN 1956			1 Plastic box x 0.56 Kg	200	

Additional Handling Information

Prepared for AIR TRANSPORT according to: (Customer MUST check one)
☐ 49 CFR ☒ ICAO / IATA

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked, and labelled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Name/Title of Signatory
Place and Date
Signature *(see warning above)*

Emergency Telephone Number (Required for US Origin or Destination Shipments) 1-800-535-5053 InfoTRAC

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-1-1	Page 1 of 25
		Effective Date 09/03	Revision 5
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information regarding the sampling of groundwater wells.

2.0 SCOPE

This procedure provides information on proper sampling equipment, onsite water quality testing, and techniques for groundwater sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require 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, their total concentration, mobility, valence, and relative concentrations, and on temperature of measure. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 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.

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 standard hydrogen electrode.

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.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

Turbidity – Turbidity in water is caused by suspended matter, such as clay, silt, 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

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

Project Geologist/Field Sample Technician - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field technicians).

5.0 PROCEDURES

5.1 General

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

Methods for withdrawing samples from completed wells include the use of pumps, compressed air, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water due to sampling techniques. 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 the groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. To safeguard against collecting non-representative stagnant water in a sample, the following approach shall be followed prior to sample acquisition:

1. All monitoring wells shall be purged prior to obtaining a sample. Evacuation of three to five volumes is recommended prior to sampling. 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.

2. For wells that can be purged dry, the well shall be evacuated and allowed to recover to 75% full capacity prior to sample acquisition. If the recovery rate is fairly rapid, evacuation of more than one volume of water is required. Dewatering the well screen should be avoided. DW 3/3/08

3. For high-yielding monitoring wells which cannot be evacuated to dryness, there is no absolute safeguard against contaminating the sample with stagnant water. One of the following techniques shall be used to minimize this possibility:

   - A submersible pump or the intake line of a surface pump or bailer shall be placed just below the water surface when removing the stagnant water and lowered as the water level drops. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. Once this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

   - The intake line of the sampling pump (or the submersible pump itself) unless otherwise directed shall be placed near the center of the screened section, and approximately one casing volume of water shall be pumped from the well at a low purge rate, equal to the well's recovery rate (low flow sampling). Low flow sampling should only be performed in wells with screen length of 10 feet or less. The pump should be placed adjacent to the water-bearing fracture. DW 3/3/08

Stratification of contaminants may exist in the aquifer. Concentration gradients as a result of mixing and dispersion processes, layers of variable permeability, and the presence of separate-phase product (i.e.,

floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase the contaminant concentrations in the recovered sample compared to what is representative of the integrated water column as it naturally occurs at that point, thus the result is the collection of a non-representative sample.

5.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform with the guidelines expressed 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, ice, labels and chain-of-custody documents.
- Field tools and instrumentation - Multi-parameters water quality meter 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.
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with droplines, 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 and inert line with tripod-pulley assembly (if necessary).
- Pails - Plastic, graduated.
- Decontamination solutions - Deionized water, potable water, laboratory detergents, 10% nitric acid solution (as required), 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.

5.3 Calculations of Well Volume

To insure 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. 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 (see SOP SA-6.3):

- Obtain all available information on well construction (location, casing, screens, etc.).
- Determine well or inner casing diameter.
- Measure and record static water level (depth below ground level or top of casing reference point).
- Determine depth of well by sounding using a clean, decontaminated, weighted tape measure.

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

- Calculate one static well volume in gallons $V = (0.163)(T)(r^2)1$

where: V	=	Static volume of well in gallons.
T=		Thickness of water table in the well measured in feet (i.e., linear feet of static water).
r	=	Inside radius of well casing in inches.
0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.

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

5.4 Evacuation of Static Water (Purging)

5.4.1 General

The amount of purging a well shall receive prior to sample collection 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 that 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 the 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, field notebook, or on standardized data sheets.

5.4.2 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. Note that all of these techniques involve equipment which is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe with a sealed bottom (bucket-type bailer) or, as is more useful and favored, with a ball check-valve at the bottom. An inert line is used to lower the bailer and retrieve the sample.

Advantages of bailers include:

- Few limitations on size and materials used for bailers.
- No external power source 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.
- Transfer of sample may cause aeration.
- Use of bailers is physically demanding, especially in warm temperatures at protection levels above Level D.

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 a flexible tubing, thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination.

These 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 significant loss of dissolved gases and volatile organics.

Air-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force the water 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, leading to 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. The operation principles vary and the 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:

- They may have low delivery rates.
- Many models of these pumps are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time-consuming.

5.5 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
- Dissolved Oxygen (DO)
- Oxidation-Reduction Potential (ORP)

- Turbidity
- Salinity

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

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Since 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 6.3 for example equipment calibration log.

5.5.1 Measurement of pH

5.5.1.1 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.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and 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, 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.

5.5.1.2 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 pH range hydrion 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 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.

5.5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Stand-alone portable pH meter, or combination meter (e.g., Horiba U-10), or combination meters 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.
- pH indicator paper, to cover the pH range 2 through 12.
- Manufacturer's operation manual.

5.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

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

- Inspect the instrument and batteries prior to initiation of the field effort.
- 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.
- 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).
- Calibrate on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on an equipment calibration log sheet.
- 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. This must be clearly noted in the logbook.
- Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH unit. Also record the sample temperature.
- Rinse the electrode(s) with deionized water.
- Store the electrode(s) in an appropriate manner when not in use.

Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

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 adequately determined.

5.5.2 Measurement of Specific Conductance

5.5.2.1 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 the 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, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance. Most conductivity meters in use today display specific conductance (SC); units of milliSiemens per centimeter, which is the conductivity normalized to temperature @ 25°C. This format (SC) is the required units recorded on the groundwater sample log field form (Attachment B).

5.5.2.2 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, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or 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 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.

5.5.2.3 Equipment

The following equipment is needed for taking specific conductance (SC) measurements:

- Stand alone portable conductivity meter, or combination meter (e.g., Horiba U-10), or combination meters 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 which may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirement of the sampling program.

5.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet. Potassium chloride solutions with a SC closest to the values expected in the field shall be used for calibration.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.
- Immerse the electrode in the sample and measure the conductivity.
- Read and record the results in a field logbook or sample log sheet.
- Rinse the electrode with deionized water.

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

5.5.3 Measurement of Temperature

5.5.3.1 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. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

5.5.3.2 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 dissolved oxygen meters, which 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.

5.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is used, the instrument shall be calibrated according to manufacturer's recommendations.

5.5.4 Measurement of Dissolved Oxygen

5.5.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration 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 dissolved oxygen meters only. 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, colloidal material or suspended matter.

5.5.4.2 Principles of Equipment Operation

Dissolved oxygen 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.

Since 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, while leaving the surface of the solution undisturbed.

Dissolved oxygen 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, which are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

5.5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- Stand alone portable dissolved oxygen meter, or combination meter (e.g., Horiba U-10), or combination meters 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.

5.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked before going to the field.
- The probe shall be conditioned 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.
- The instrument shall be calibrated in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
- Record all pertinent information on an equipment calibration sheet.
- Rinse the probe with deionized water.
- 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 can be moved up and down.
- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.
- Rinse the probe with deionized water.
- 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, since sample handling is not involved. This however, may not always be practical.

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

5.5.5 Measurement of Oxidation-Reduction Potential

5.5.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendency of organic or inorganic compounds 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 oxidized to reduced species in the sample.

5.5.5.2 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 dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

5.5.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Combination meters 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.

5.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be checked using the manufacturer's recommended reference solution and have its batteries checked before going to the field.
- Thoroughly rinse the electrode with deionized water.
- If the probe does not respond properly to the recommended reference solution, then verify the sensitivity of the electrodes by noting the change in millivolt reading 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 if the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added (i.e., pH is raised) thus indicating 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.
- Record all pertinent information on an equipment calibration log sheet.

5.5.6 Measurement of Turbidity

5.5.6.1 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, finely divided organic and inorganic matter, soluble colored organic compounds, and microscopic organisms, including plankton.

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

5.5.6.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method. This method 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 turbidmeter. Therefore, nephelometric turbidity units (NTU) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

5.5.6.3 Equipment

The following equipment is needed for turbidity measurement:

- Light meter (e.g., LaMotte 2020) which calibrates easily using test cells with standards of 0.0 NTUs, and 10 NTUs, or combination meter (e.g., Horiba U-10), 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.

5.5.6.4 Measurement Techniques for Turbidity

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

- Check batteries and calibrate instrument before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the electrode with one or more portions of the sample to be tested or with deionized water (applies to "e").
- Fill the light meters glass test cell with ~5 ml of sample, screw on cap, wipe off glass, place test cell in light meter and close the lid (applies to "l").
- Immerse the electrode in the sample and measure the turbidity (applies to "e").
- The reading must be taken immediately as suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
- Read and record the results in a field logbook or sample log sheet. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
- Rinse the electrode or test cell with deionized water.

5.5.7 Measurement of Salinity

5.5.7.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Note: Most field meters determined salinity automatically from conductivity and

temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

5.5.7.2 Principles of Equipment Operation

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

5.5.7.3 Equipment

The following equipment is needed for Salinity measurements:

- Multi-parameter water quality meter capable of measuring conductive, temperature and converting them to salinity (e.g., Horiba U-10 or YSI 600 series).
- Calibration Solution, as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.7.4 Measurement Techniques for Salinity

The steps involved in taking Salinity measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the cell with the sample to be tested.
- Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or sample log sheet.
- Rinse the probes with deionized water.

5.6 Sampling

5.6.1 Sampling Plan

The sampling approach consisting of the following, shall be developed as part of the project plan documents which are 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 degrees of contamination between wells is unknown or insignificant, a sampling sequence which 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 as a result of the sampling procedures.

- 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, requirement for split samples, access problems, location of keys, etc.

5.6.2 Sampling Methods

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

1. The site Health & Safety Officer (or designee) will first open the well cap and use volatile organic detection equipment (PID or FID) on the escaping gases at the well head to determine the need for respiratory protection.

2. 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 (see Attachment B); then calculate the fluid volume in the well pipe (as previously described in this SOP).

3. Calculate well volume to be removed as stated in Section 5.3.

4. Select the appropriate purging equipment (see Attachment A). If an electric submersible pump with packer is chosen, go to Step 10.

5. Lower the purging equipment or intake into the well to a short distance below the water level and begin water removal. Collect the purged water and dispose of it in an acceptable manner (as applicable). Lower the purging device, as required, to maintain submergence.

6. Measure the rate of discharge frequently. A graduated bucket or cylinder and stopwatch are most commonly used.

7. Observe the peristaltic pump 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.

8. Purge a minimum of three to five casing volumes before sampling. In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Purged water shall be collected in a designated container and disposed in an acceptable manner.

9. If sampling using a pump, lower the pump intake to midscreen (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.

10. (For pump and packer assembly 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.

11. In the event that 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.

12. Fill sample containers (preserve and label as described in SOP SA-6.1).

13. Replace the well cap and lock as appropriate. Make sure the well is readily identifiable as the source of the samples.

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

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

5.7 Low Flow Purging and Sampling

5.7.1 Scope & Application

Low flow purging and sampling techniques are sometimes 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. The minimum stress procedure emphasizes negligible water level drawdown and low pumping rates in order 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, or open interval, length of ten feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semi-volatile organic compounds, pesticides, PCBs, metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This procedure is not designed to collect non-aqueous phase liquids samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs), using the low flow pumps.

The 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 NTU and to achieve a water level drawdown of less than 0.3 feet during purging and sampling. If these goals cannot be achieved, sample collection can take place provided the remaining criteria in this procedure are met.

5.7.2 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 may be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing - Teflon, Teflon-lined polyethylene, polyethylene, 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, 0.01 foot accuracy, (electronic devices are preferred for tracking water level drawdown during all pumping operations).
- Interface probe, if needed.
- 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 dissolved oxygen, flow-through cell is required. Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s), and other forms (see Attachments B and C).
- 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 VOCs (volatile organic compounds).

5.7.3 Purging and Sampling Procedure

Open monitoring well, measure head space gases using PID/FID. If there is an indication of off gassing when opening the well, wait 3-5 minutes to permit water level an opportunity to reach equilibrium.

Measure and record the water level immediately prior to placing the pump in the well.

Lower 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 two 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 three feet or less of standing water in the well.

Start with the initial pump rate set at approximately 0.1 liters/minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 feet during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters/minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below

the top of the well screen, purging will cease or the well will be pumped to dryness and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be colleted from these wells within the same workday and no later than 24 hours after the start of purging.

Measure the well water level using the water level meter every 5 to 10 minutes. Record the well water level on the Low-Flow Purge Data Form (Attachment C).

Record on the Low-Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, dissolved oxygen and salinity or as specified by the approved site specific work plan) 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.

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

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.

After stabilization is achieved, sampling can begin when 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: DW 2/29/08

- pH ±~~0.2~~ 0.1 standard units
- Specific conductance ±~~10%~~ 3% DW 2/29/08
- Temperature ±10%
- Turbidity less than 10 NTUs
- Dissolved oxygen ±10%

If the above conditions have still 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.

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.

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: (1) Collect the non-VOCs samples first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample and record the new flow rate; (2) 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; (3) 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, collect sample from the narrow diameter tubing.

Prepare samples for shipping as per SOP SA-6.1.

the top of the well screen, purging will cease or the well will be pumped to dryness and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be colleted from these wells within the same workday and no later than 24 hours after the start of purging.

Measure the well water level using the water level meter every 5 to 10 minutes. Record the well water level on the Low-Flow Purge Data Form (Attachment C).

Record on the Low-Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, dissolved oxygen and salinity or as specified by the approved site specific work plan) 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.

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

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.

After stabilization is achieved, sampling can begin when 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:

- pH ~~±0.2 standard units~~ 0.1 standard units
- Specific conductance ±~~10%~~ 3%
- Temperature ±10%
- Turbidity less than 10 NTUs
- Dissolved oxygen ±10%

If the above conditions have still 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.

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.

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: (1) Collect the non-VOCs samples first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample and record the new flow rate; (2) 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; (3) 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, collect 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:

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

ATTACHMENT C
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 procedure describes methods and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing, or for subsequent laboratory analysis.

2.0 SCOPE

The information presented in this guideline is generally applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions and equipment different from those described herein.

3.0 GLOSSARY

Environmental Sample - a sample containing (or suspected to contain) low-level concentrations of contaminants, which does not require special handling or transport considerations as detailed in SOP SA-6.1.

Hazardous Waste Sample - a sample containing (or suspected to contain) higher concentrations of contaminants thus requiring special handling and/or transport considerations per SOP SA-6.1.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel.

Field Operations Leader - The Field Operations Leader (FOL) is responsible for the supervision of onsite water quality analyses, ensuring proper sample collection, handling, and the completion and accuracy of all field documentation, and making sure that custody of all samples obtained is maintained according to proper procedures.

5.0 PROCEDURES

5.1 Introduction

Collecting a representative sample from surface water or sediments is difficult because of water movement, stratification, or patchiness. To collect representative samples, one must standardize sampling bias related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification.

Representativeness is a qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been taken. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples.

5.2 Defining the Sampling Program

Many factors must be considered in developing a sampling program for surface water or sediments including study objectives, accessibility, site topography, physical characteristics of the water body (such as flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on the vertical and lateral mixing within the body of water. For sediments, dispersion depends on bottom current or flow characteristics, sediment characteristics (density, size) and geochemical properties (which affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes, but also must understand the role of fluvial-sediment transport, deposition, and chemical sorption.

5.2.1 Sampling Program Objectives

The objective of surface water sampling is to determine the surface water quality entering, leaving or remaining within the site. The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., spills). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the waterbody, direct waste disposal (solid or liquid) into the water body; and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) which encompass the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the location of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc., shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation are: (1) move the site far enough downstream to allow for adequate mixing, or (2) collect integrated samples in a cross section. Also, nonhomogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and the heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

5.2.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled

by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream, because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes and reservoirs, as well as those on larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Sampling in marshes or tidal areas may require the use of an all-terrain vehicle (ATV). The same precautions mentioned above with regard to sediment disturbance apply.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed in order to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of streamflow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Streamflow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining streamflow data by direct or indirect methods.

5.2.3 Frequency of Sampling

The sampling frequency and the objectives of the sampling event will be defined by the project plan documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of the contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples shall be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly, and during droughts and floods). Samples of bottom material shall be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water-quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

5.3 Surface Water Sample Collection

5.3.1 Streams, Rivers, Outfalls and Drainage Features (Ditches, Culverts)

Methods for sampling streams, rivers, outfalls, and drainage features at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode, shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited.

Generally, the number and type of samples to be taken depend on the river's width, depth, discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of DO, pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project plan documents.

5.3.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained.

The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, the measurement of DO, pH, temperature, etc., is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally-formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample. These verticals are often taken along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline which is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer which is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several verticals with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality since it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in-situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, dissolved oxygen, some cations and anions, and light penetration.

5.3.3 Estuaries

Estuarine areas are by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Estuaries are generally categorized into three types dependent upon freshwater inflow and mixing properties. Knowledge of the estuary type is necessary to determine sampling locations. Each type of estuarine area is described below:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically this type of estuary is shallow and is found in major freshwater sheetflow areas. Being well mixed, the sampling locations are not critical in this type of estuary.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally, back and forth, with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small with the preponderance of the fresh-saline water mixing occurring near, or at, the shore line.

Sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical dissolved oxygen and temperature profiles.

5.3.4 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. The most frequently used samplers are:

- Open tube.
- Dip sampler.
- Weighted bottle.
- Hand pump.
- Kemmerer.
- Depth-Integrating Sampler.

The dip sampler and the weighted bottle sampler are used most often, and detailed discussions for these devices only (and the Kemmerer sampler) are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.
4. Nonreactive/noncontaminating properties - Teflon-coated, glass, stainless-steel or PVC sample chambers are preferred (in that order).

As specified above, each sample (grab or each aliquot collected for compositing) shall be measured for but not limited to:

- Specific conductance.
- Temperature.
- pH.
- Dissolved oxygen (optional).

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field

notebook or on sample logsheets (see Attachment A). These analyses will provide information on water mixing/stratification and potential contamination.

Dip Sampling

Water is often sampled by filling a container either attached to a pole or held directly, from just beneath the surface of the water (a dip or grab sample). Constituents measured in grab samples are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration that is distributed throughout the water column and in the cross section. Therefore, whenever possible, it is recommended to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

Weighted Bottle Sampling

A grab sample can also be taken using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of a stopped glass or plastic bottle, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The procedure for sampling with this device is:

- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
- Pull out the stopper with a sharp jerk of the stopper line.
- Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
- Raise the sampler and cap the bottle.
- Decontaminate the outside of the bottle. This bottle can be used as the sample container as long as the bottle is an approved container type.

Kemmerer

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless-steel or acrylic cylinder, with rubber stoppers that leave the ends open while being lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles.

5.3.5 Surface Water Sampling Techniques

Most samples taken during site investigations are grab samples. Typically, surface water sampling involves immersing the sample container in the body of water; however, the following suggestions are made to help ensure that the samples obtained are representative of site conditions:

- The most representative samples are obtained from mid-channel at a 0.6 foot stream depth in a well-mixed stream.
- Even though the containers used to obtain the samples are previously laboratory cleaned, it is suggested that the sample container be rinsed at least once with the water to be sampled before the sample is taken. This is not applicable when sample containers are provided "pre-preserved."
- For sampling moving water, it is suggested that the farthest downstream sample be obtained first, and that subsequent samples be taken as one works upstream. In general, work from zones suspected of low contamination to zones of high contamination.
- To sample a pond or other standing body of water, the surface area may be divided into grids. A series of samples taken from each grid node is combined into one sample, or several grid nodes are selected at random.
- Care should be taken to avoid excessive agitation of the water, as loss of volatile constituents could result.
- When obtaining samples in 40 mL septum vials for volatile organics analysis, it is important to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. The vial can be turned upside down to check for air bubbles.
- Do not sample at the surface, unless sampling specifically for a known constituent which is immiscible and on top of the water. Instead, the sample container should be inverted, lowered to the approximate depth, and held at about a 45-degree angle with the mouth of the bottle facing upstream. When sample containers are provided "pre-preserved," use a dedicated, clean, un-preserved bottle for sampling and transfer to an appropriately-preserved container.

5.4 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

5.5 Sediment Sampling

5.5.1 General

Sediment samples are usually collected at the same verticals at which water samples were collected. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body.

Generally, the coarser grained sediments are deposited near the headwaters of the reservoir. Bed sediments near the center of a water body will be composed of fine-grained materials which may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathometry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled while areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials, shall be avoided.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if

water column concentrations are below detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B.

5.5.2 Sampling Equipment and Techniques

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

The following health and safety provisions apply when working on/over/near water.

Size of Work Team:

1) Never less than 2 persons [who are wearing USCG approved Personal Flotation Devices (PFDs)]

2) A minimum of 3 persons if *any* of the following conditions are anticipated or observed:

- Depth is greater than 3 feet
- Involves a waterway that is turbulent *or* swift
- The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples would include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.)
- Waterway is tidal, and conditions such as those listed above could change

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered PFD (life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect bottom materials:

- Scoop sampler.
- Dredge samplers.

Each type of sampler is discussed subsequently.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if it can be waded, the easiest and best way to collect a sediment sample is to use a scoop sampler. This reduces the potential for cross-contamination. This method is accomplished by reaching over or wading into the water body and, while facing upstream (into the current), scooping the sampler along the bottom in an upstream direction. It is very difficult not to disturb fine-grained materials of the sediment-water interface when using this method.

Dredges

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., coarse-grained or partially-cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger". Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. There are three major types of dredges: Peterson, Eckman and Ponar dredges.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave". The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

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ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA

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

SAMPLE COLLECTION INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (~~y~~261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (~~y~~261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration			Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER						
Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR						
Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11], [12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11], [14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

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

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

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

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

_____

Field Operations Leader

ATTACHMENT B

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

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

DATE YEAR ___	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - Is a solution selected/identified within the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

Potable Water - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

Solvent - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Failure to meet these objectives are sufficient to restrict equipment from entering the site/exiting the site/ or moving to a new location on the site until the objectives are successfully completed.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide the following characteristics

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

  - Liner – Depending on the types of equipment and the decontamination method the liner should be of sufficient thickness to provide a puncture resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. Achieving the desired thickness maybe achieved through layering lighter constructed materials. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner a light coating of sand maybe applied to provide traction as necessary.

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.

2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable*.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

---

* - The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

1) Wash with soap and water
2) Rinse with tap water
3) Rinse with deionized water

Note: In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer's instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.

- Cleanliness – As per protocol only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or associated with noticeable odors it should be decontaminated prior to use.

  1) Wash with soap and water
  2) Rinse with tap water
  3) Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole Drilling Equipment

This includes any portion of the drill rig that is over the borehole including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

During the course of field investigations disposable/single use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- Rinsate Blanks – It is recommended that Rinsate samples be collected to

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Low-Flow Purging and Sampling of Groundwater Monitoring Wells

Bulletin No. QAD023 October 15, 1997

RECOMMENDED PROCEDURE FOR LOW-FLOW PURGING AND SAMPLING OF GROUNDWATER MONITORING WELLS

1.0 OBJECTIVE AND APPLICATION

This directive provides a procedure for collection of ground-water samples in small-diameter wells with short-screened intervals using low-flow purging and sampling. While these procedures pertain to the Superfund program in Region 3, they were based on recommendations presented in the EPA Ground Water Issue paper entitled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures". The low-flow purging and sampling method is not appropriate for use in all hydrogeologic regimes, and particular groundwater monitoring well designs may make the method unsuitable (e.g. open hole and long screen monitoring wells in bedrock and stratified sand and clay where the water bearing zones have not been characterized). Therefore, please confer with a Region 3 hydrogeologist or geologist before using these procedures at a site.

2.0 BACKGROUND

Past scientific research (Barcelona et al., 1983; Nielson and Yeates, 1985) and EPA guidance have discussed sampling devices in terms of their compatibility with contaminants being sampled, and well construction, depth, and diameter. Although some sampling devices have been used in order to provide more representative groundwater samples in certain situations, most of these incorporate high-volume withdrawal techniques (i.e., in excess of the "natural" recharge rate of groundwater flow through the well screen) for both purging and sampling.

Research conducted by Puls et al. (1992), Puls and Powell (1992), and Powell and Puls (1993) has shown that high-volume purging and sampling cause significant turbidity and suspended particulate artifacts that can result in biased-high metals results. Additionally, purging can cause pressure changes and bailing can cause aeration that can strip VOCs from the sample (Pennino, 1988). The use of low-flow pumping devices (preferably dedicated) for purging and sampling minimizes both the disturbance of water in well casing and the potential for mobilization of colloidal material (Barcelona et al., 1994). Low-flow purging with maintenance of water level in the well and stabilization of indicator parameters (especially turbidity) allows collection of groundwater samples that are more representative of conditions without filtering (U.S. EPA, 1993; Backhus et al., 1993). In many cases, use of a low-flow pump to purge and sample monitoring wells decreases sampling time, reduces the need to handle large volumes of purge water and lowers the cost associated with its disposal, and allows collection of samples for inorganic analyses without filtering. This procedure is designed to be used in conjunction with groundwater sampling and analyses for the most common types of groundwater contaminants (volatile and semi-volatile organic compounds, pesticides, PCBs and inorganic compounds).

3.0 EQUIPMENT

Adjustable rate, positive displacement pumps (e.g. low flow-rate submersible centrifugal or bladder pumps constructed of stainless steel or Teflon). Low flow-rate electrical submersible pumps are recommended because (1) they are not subject to cyclical flow/arrest and consequent potential for mobilizing fine-grained material, and (2) they may be less prone to

operator error, thereby reducing potential error resulting from application by different personnel. The pump should be easily adjustable and capable of operating reliably at lower flow rates. Peristaltic pumps may be used only for inorganic sample collection. Bailers are inappropriate for use in this procedure.

- Tubing: Tubing used in purging and sampling each well must be dedicated to the individual well. Once properly located, moving the pump in the well should be avoided. Consequently, the same tubing should be used for purging and sampling. Teflon or Teflon-lined polyethylene tubing must be used to collect samples for organic analysis. For samples collected for inorganic analysis, Teflon or Teflon lined polyethylene, PVC, Tygon or polyethylene tubing may be used. The tubing wall thickness should be maximized (3/8 to ½ inch) and the tubing length should be minimized (i.e. do not have excess tubing outside of the well).
- Polyethylene sheeting and sampling gloves.
- Water level measuring device, 0.01 feet accuracy, (electronic preferred for tracking water level drawdown during all pumping operations).
- Flow measurement supplies (e.g. graduated cylinder and stop watch).
- Interface probe, if needed.
- Power source (e.g. generator, located downwind; nitrogen tank, etc). The generator should not be oversized for the pump.
- In-line flow-through cell containing purge criteria parameter monitoring instruments for pH, turbidity, specific conductance, temperature, Eh and dissolved oxygen (DO). The in-line device should be bypassed or disconnected during sample collection.
- Photoionization detector (PID), or equivalent.
- Nylon stay-ties.
- Decontamination supplies.
- Logbook(s).
- Sample Bottles. It is recommended that preservatives are added to sample bottles prior to field activities to reduce potential error or introduction of contaminants.
- Sample preservation supplies (as required by the analytical method; see previous bullet).
- Sample tags or labels, chain of custody.
- Well construction data, location map, field data from last sampling event.
- Approved Field Sampling Plan/QA Project Plan.

4.0 PRELIMINARY SITE ACTIVITIES

1) Check the condition of the monitoring well for damage and evidence of tampering, and record pertinent observations.

2) In order to maintain a clean work area, lay out a sheet of polyethylene to place sampling and monitoring equipment.

3) Remove well cap and measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the field logbook.

4) If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing) make one.

5) Measure and record the depth to water (to 0.01 feet) in all wells to be sampled before any purging begins. Care should be taken to minimize disturbance to the water column and to any particulate attached to the sides or at the bottom of the well. Consequently, in order to avoid disturbing any accumulated sediment and to prevent mixing of stagnant water with water in the screened interval, the total depth of a well should be measured well in advance (one to two weeks) of purging and sampling or after sampling is completed. Obtain depth to bottom of well information from the well construction log and calculate standing water volume as: depth of water column times cross-sectional area of the well.

6) For wells where an Light Non-aqueous Phase Liquid (LNAPL) has been detected, a stilling tube should be inserted into the well prior to purging. Refer to Section 7.2.4 of EPA (1992) for the procedure to follow. If the wells are constructed so that DNAPLs could accumulate, their detection and /or sampling should occur, at a minimum, a week before groundwater purging

and sampling. Measurement and sampling of potential DNAPL should be conducted as a separate event to minimize disturbance of any sediments which have accumulated in the bottom of the well. A double check valve, bottom loading bailer is recommended for sampling. Light non-aqueous phase liquid (LNAPL) measurement may be conducted (with an interface probe), with care to avoid disturbance of the water column within the well.

5.0 PURGING AND SAMPLING PROCEDURES

The following describes the procedure for the low-flow purging and sampling method. Equipment calibration, logbook documentation, sample bottle filling and preservation, and shipping will be conducted in accordance with the site-specific Quality Assurance Project Plan (QAPjP). Personal protective equipment will be donned in accordance with the requirements of the site-specific Health and Safety Plan. Wells should be sampled in the order of least contaminated to most contaminated.

1) Attach and secure the polyethylene tubing to the low-flow pump. As the pump is slowly lowered into the well, secure the safety drop cable, tubing, and electrical lines to each other using nylon stay-ties.

2) Pump, safety cable, tubing and electrical lines should be lowered slowly into the well to a depth corresponding to the center of the saturated screen section of the well, or at a location determined to either be a preferential flow path or zone where contamination is present. The pump intake should be kept above the bottom of the well to prevent mobilization of any sediment or DNAPL present in the bottom of the well. It is recommended that the pump be placed in the well 12 to (preferably) 48 hours prior to purging/sampling to minimize the effects of turbidity and mixing in the well from introducing the pump.

3) Measure the water level again with the pump in the well before starting the pump. Start pumping water from the well at a rate of 100 to 400 milliliters per minute (mL/min). Avoid surging. Observe air bubbles displaced from discharge tube to assess progress of steady pumping until water arrives at the surface. The pumping rate should cause little or no water level drawdown in the well (less than 0.2 ft) and the water level should stabilize. Water level measurements should be made continuously. Precautions should be taken to avoid pump suction loss or air entrainment. Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to avoid pumping the well dry and ensure stabilization of indicator parameters. If the recharge rate of the well is very low, purging should be interrupted so as not to cause the drawdown within the well to advance below the pump intake but the operator should attempt to maintain a steady flow rate with the pump to the extent practicable. In these low-yielding wells, where 100 mL/min exceeds the entrance rate of groundwater into the well, it is important to avoid dewatering the well screen interval. In these cases, the pump should remain in place and the water level should be allowed to recover repeatedly until there is sufficient volume in the well to permit collection of samples. An alternative means of sample collection may be necessary under these conditions.

4) While purging the well, monitoring of in-line water quality indicator parameters should include turbidity, specific conductance, pH, dissolved oxygen (DO) and redox potential (Eh) which must be collected every three to five minutes until all of the parameters have stabilized. Stabilization is achieved when three successive readings are within ± 0.1 for pH, $\pm 3\%$ for conductivity, ± 10mv for redox potential (Eh), and $\pm 10\%$ for turbidity and DO. A minimum subset of these parameters that can be used to determine stabilization during purging in this procedure are pH, specific conductivity and turbidity or DO. Turbidity and DO are typically the last parameters to stabilize. If the parameters have stabilized, but the turbidity is not in the range of 5-10 NTU, then follow step 6.

5) Once stabilization has been documented, VOC and gas sensitive (e.g. Fe^{+2}, CH_4, H_2S/HS) parameter samples should be immediately collected first and directly into pre-preserved sample containers. All sample containers should be filled by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

Samples requiring pH adjustment should have their pH checked to assure that the proper pH has been obtained. For VOC samples, this will require that a test sample be collected to determine the amount of preservative required to be added to the sample containers prior to sampling.

6) If the turbidity measurements do not approach the range of that of natural groundwater (10 NTU), both filtered and unfiltered samples should be collected for metals analysis. Filtered metal samples are to be collected with an in-line filter. A high capacity, in-line 0.45 micron particulate filter must be pre-rinsed according to the manufacturer's recommendations, or with approximately 1 liter of groundwater following purging and prior to sampling. After the sample is filtered it must be preserved immediately.

7) As each sample is collected, the sample should be labeled as defined in the QAPjP. All samples should be placed into a cooler with proper temperature control as outlined in the QAPjP.

After collection of the samples, the tubing from the pump should be properly discarded or dedicated to the well for re-sampling (by hanging the tubing inside the well).

8) Measure and record well total depth.

9) Secure the well (close and lock it up).

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APPENDIX B

LABORATORY SOPs

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1 Scope and Application

1.1 This standard operating procedure is adapted from "Test Methods for Evaluating Solid Waste Physical/Chemical Methods" (SW-846), Method 8260B, Revision 2, December 1996, Method 5035A, Revision 1, July 2002, and Method 5030C, Revision 3, December 2003. The method detection limits (MDLs) can be found in the most current GC/MS method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.2 Method 8260B is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, 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. The following compounds can be determined by this method:

Analyte	CAS No.[b]	Appropriate Technique Purge-and-Trap
Acetone	67-64-1	pp
Acetonitrile	75-05-8	pp
Acrolein (Propenal)	107-02-8	pp
Acrylonitrile	107-13-1	pp
Allyl alcohol	107-18-6	ht
Allyl chloride	107-05-1	a
Benzene	71-43-2	a
Benzyl chloride	100-44-7	a
Bromacetone	598-31-2	pp
Bromochloromethane	74-97-5	a
Bromodichloromethane	75-27-4	a
4-Bromofluoraobenzene	460-00-4	a
Bromoform	75-25-2	a
Bromomethane	74-83-9	a
n-Butanol	71-36-3	ht
2-Butanone (MEK)	78-93-3	pp
Carbon disulfide	75-15-0	pp
Carbon tetrachloride	56-23-5	a
Chloral hydrate	302-17-0	pp
Chlorobenzene	108-90-7	a
Chlorobenzene	126-99-8	a
Chlorodibromomethane	124-48-1	a
Chloroethane	75-00-3	a
2-Chloroethanol	107-07-3	pp
bis-(2-Chloroethyl) sulfide	505-60-2	pp
2-Chloroethyl vinyl ether	110-75-8	a
Chloroform	67-66-3	a
Chloromethane	74-87-3	a
Chloroprene	126-99-8	a
3-Chloropropene	107-05-1	a
3-Chloropropionitrile	542-76-7	I
1,2-Dibromo-3-chloropropane	96-12-8	pp
1,2-Dibromoethane	106-93-4	a
Dibromomethane	74-95-3	a
1,2-Dichlorobenzene	95-50-1	a
1,3-Dichlorobenzene	541-73-1	a
1,4-Dichlorobenzene	106-46-7	a
cis-1,4-Dichloro-2-butene	1476-11-5	a
trans-1,4-Dichloro-2-butene	110-57-6	pp
Dichlorodifluoromethane	75-71-8	a
1,1-Dichloroethane	75-34-3	a
1,2-Dichloroethane	107-06-2	a
1,1-Dichloroethene	75-35-4	a
trans-1,2-Dichloroethene	156-60-5	a
1,2-Dichloropropane	78-87-5	a
1,3-Dichloro-2-propanol	96-23-1	pp
cis-1,3-Dichloropropene	10061-01-5	a
trans-1,3-Dichloropropene	10061-02-6	a
1,2,3,4-Diepoxybutane	1464-53-5	a
Diethyl ether	60-29-7	a
1,4-Difluorobenzene	540-36-3	a

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1,4-Dioxane	123-91-1	pp
Epichlorohydrin	106-89-8	i
Ethanol	64-17-5	i
Ethyl acetate	141-78-6	i
Ethylbenzene	100-41-4	a
Ethylene oxide	75-21-8	pp
Ethyl methacrylate	97-63-2	a
Hexachlorobutadiene	87-68-3	a
Hexachloroethane	67-72-1	i
2-Hexanone	591-78-6	pp
2-Hydroxypropionitrile	78-97-7	i
Iodomethane	74-88-4	a
Isobutyl alcohol	78-83-1	pp
Isopropylbenzene	98-82-8	a
Malononitrile	109-77-3	pp
Methacrylonitrile	126-98-7	pp
Methylene chloride (DCM)	75-09-2	a
Methyl methacrylate	80-62-6	a
4-Methyl-2-pentanone (MIBK)	108-10-1	pp
Naphthalene	91-20-3	a
Nitrobenzene	98-95-3	a
2-Nitropropane	79-46-9	a
Pentachloroethane	76-01-7	i
2-Picoline	109-06-8	pp
Propargyl alcohol	107-19-7	pp
β-Propiolactone	57-57-8	pp
Propionitrile (ethyl cyanide)	107-12-0	ht
Propylamine	107-10-8	a
Pyridine	110-96-1	i
Styrene	100-42-5	a
1,1,1,2-Tetrachlorethane	630-20-6	a
1,1,2,2-Tetrachlorethane	79-34-5	a
Tetrachloroethene	127-18-4	a
Toluene	108-88-3	a
1,2,4-Trichlorobenzene	120-82-1	a
1,1,1-Trichloroethane	71-55-6	a
1,1,2-Trichloroethane	79-00-5	a
Trichloroethene	79-01-6	a
Trichlorofluoromethane	75-69-4	a
1,2,3-Trichloropropane	96-18-4	a
Vinyl acetate	108-05-4	a
Vinyl chloride	75-01-4	a
o-Xylene	95-47-6	a
m-Xylene	108-38-3	a
p-Xylene	106-42-3	a

a	Adequate response by this technique.
b	Chemical Abstract Services Registry Number.
ht	Method analyte only when purged at 80°C.
i	Inappropriate technique for this analyte.
pc	Poor chromatographic behavior
pp	Poor purging efficiency resulting in high EQLs.

NOTE: For the preparation of soils and solids, see the 19-5035 SOP.

1.3 Method 8260B can be used to quantitate most volatile organic compounds that have 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, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides. The following analytes are also amenable to analysis by Method 8260B:

Bromobenzene	1-Chlorohexane
n-Butylbenzene	2-Chlorotoluene
sec-Butylbenzene	4-Chlorotoluene
tert-Butylbenzene	Crotonaldehyde
Chloroacetonitrile	Dibromofluoromethane
1-Chlorobutane	cis-1,2-Dichloroethene
1,3-Dichloropropane	Methyl-t-butyl ether
2,2-Dichloropropane	Pentafluorobenzene
1,1-Dichloropropene	n-Propylbenzene
Fluorobenzene	1,2,3-Trichlorobenzene
p-Isopropyltoluene	1,2,4-Trimethylbenzene
Methyl acrylate	1,3,5-Trimethylbenzene

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1.4 The estimated quantitation limit (EQL) of Method 8260B for an individual compound is somewhat instrument dependent. Using standard quadruple instrumentation, limits shall be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water. EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.5 Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

1.6 An additional method for sample introduction is direct injection. This technique has been tested (by agencies other than ALSI) for the analysis of waste oil diluted with hexadecane 1:1 (vol/vol) and may have application for the analysis of some alcohols and aldehydes in aqueous samples. ALSI does not use the direct injection technique and the technique will not be covered by this standard operating procedure.

1.7 This standard operating procedure also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes and soils/sediments for analysis by the purge-and-trap procedure.

1.8 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.9 The Method Detection Limits can be found in the current GC/MS Volatiles method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.10 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

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3 Interferences

3.1 Impurities in the purge gas, organic compounds outgassing from the plumbing ahead of the trap and solvent vapors in the laboratory, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.4. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system shall be avoided.

3.2 Samples may be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on contamination.

3.3 Contamination by carry-over can occur whenever a low level sample is analyzed immediately after a high level sample. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, one or more cleaning blanks shall be analyzed to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash the purging device with a soap solution, rinse with organic-free reagent water, and then dry in an oven at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent baking and purging of the entire system may be needed. In extreme situations, the whole purge and trap device may require dismantling and cleaning.

3.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area shall be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Laboratory clothing worn by analysts shall be clean since exposure to methylene chloride fumes during extraction procedures can contribute to sample contamination.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

4.2 Analysts shall consult the material safety data sheets (MSDS) for each chemical used in the analysis. ALSI maintains MSDSs on all chemicals used in this procedure.

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MSDSs are available to all staff and are located in the QA office.

4.3 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene and vinyl chloride. Primary standards of these toxic compounds shall be prepared in a hood.

4.4 Since the chemical makeup of the samples is not known, analysts shall treat the samples with extreme caution. Precautionary steps would include using Latex gloves, wearing a fully-buttoned lab coat, and safety glasses.

5 Apparatus and Materials

5.1 Purge-and-trap device (Example system provided below)

5.1.1 Purge and Trap concentrator: Tekmar 3000, Model #14-30000-000, Serial #93133003.

5.1.2 Autosampler: Archon-EST, Model #D4-505220-16, Serial #12543.

5.1.3 Trap: VocarbJ 3000, Purge Trap K, purchased from Supelco, catalog #2-4920, or equivalent.

5.2 Gas chromatography/mass spectrometer/data system. (Example system provided below.)

5.2.1 Gas chromatograph: Hewlett Packard 5890 Series II, Serial # 3336A50415.

5.2.2 Gas chromatographic column: 75 m x 0.53 mm ID megabore capillary column coated with DB624 (J & W Scientific), 3 um film thickness, or equivalent.

5.2.3 Mass spectrometer: Hewlett Packard 5970 Series Mass Selective Detector, Model #5970B, Serial #3004A12574.

5.2.4 Electron Multiplier: K and M Model #7596M, purchased from CPI, part #4200-01.

5.2.5 GC/MS Interface

5.2.5.1 Jet separator if necessary: purchased from SIS, part #13505

5.2.5.2 Transfer line: 0.53 ID fused silica guard column, phenyl methyl deactivated, purchased from Restek, catalog #0045. Not needed with

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split injector and EPC (electronic pressure control).

Note: Currently, none of the GCMS instruments have need of a jet separator but may require one in the future.

5.2.6 GC Inlet: O-I Low-Dead-Volume Injector Port kit, O-I-Analytical, Part #176926.

5.2.7 Data System

5.2.7.1 Hewlett Packard MS DOS Chemstation, used for instrument tuning and data collection.

5.2.7.2 Hewlett Packard 4920 ChemServer with Envision and Target 4.13 software.

5.2.8 See the instrument maintenance logbooks, located in the data review area of the GC/MS laboratory, for serial number and all pertinent information relating to all other GC/MS instruments used for the analysis of 8260B volatile samples.

5.3 Microsyringes: Hamilton gastight, various volumes between 10µL and 100µL: VWR #60376-220,230,241,252,263,274, or equivalent.

5.4 Syringe valve: two-way, with Luer ends.

5.5 Syringes: 5 mL Hamilton Gastight: VWR # 60376-321, or equivalent.

5.6 Balance – ACCULAB VI-200; 200 gm capacity; 0.01 gm resolution or equivalent.

5.7 Micro Reaction Vessels: 1.0 mL, Supelco #3-3293 or equivalent.

5.8 Mininert Valves: 15 mm, Supelco #614160 or equivalent.

5.9 Vials: 40 mL I-CHEM: VWR # IRS136-0040, or equivalent.

5.10 Vials: 20 mL I-CHEM: VWR # IRS126-0020, or equivalent.

5.11 Teflon faced liners: VWR # 66001-236, or equivalent.

5.12 Disposable pipettes: Pasteur, VWR 5 ¾” # 14672-200, or equivalent.

5.13 Volumetric flasks: Class A, various volumes, with ground-glass stoppers.

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5.14 Spatula: stainless steel VWR # 57952-107, or equivalent

5.15 NOTE: In the above listings, the item noted may be replaced by a similar item of equivalent quality and function by another supplier if the need arises.

6 Reagents

6.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of The Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Reagent water: Reagent water is water in which an interference is not observed at the analyte of interest. For this purpose, tap water is used. De-ionized water shall not be used for this method as it has been shown to contain interferences due to cartridge bleed. NOTE: Once resolved, DI water may be used.

6.3 Compressed Helium gas: ultrahigh purity grade.

6.4 Methanol: EM Science Purge & Trap grade, # MX0482-6 or equivalent. This methanol is stored in the solvent storage cabinet in the GC department.

6.5 Primary Stock Solutions: Primary stock solutions may be prepared from pure standard materials or purchased as certified solutions. Standards for all 8260B compounds are purchased as certified solutions. These certified solutions are stored in flame-sealed ampules in the small freezer marked “Volatile Standards” located in the GCMS volatile area of the laboratory at -10°C to -20°C. Each certified solution has an expiration date and needs to be properly discarded if that date is exceeded. For all secondary working solutions, vials are labeled with the name of the standard, the date prepared, the expiration date, the preparer’s initials and the reference to the volume and page number in the GCMS VOC Standards Logbook. All standard preparations must be documented in the GCMS VOC Standards Logbook. Storage location is the volatile standards freezer.

6.5.1 If the primary stock solutions are to be prepared from pure standard materials, follow the instructions in Section 5.7 of Method 8260B.

6.5.2 As an alternative to Section 5.7 of Method 8260B, the following procedure may be used to prepare standards from pure standard materials that are a liquid at room temperature.

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6.5.2.1 Determine the desired concentration, **C**, in µg/mL of the stock solution.

6.5.2.2 Determine the desired volume, **V**, in mL of the stock solution.

6.5.2.3 Lookup the density, **D**, of the liquid analyte.

6.5.2.4 Find the purity, **P**, in percent of the chemical. If it is 96% or greater, assume the purity is 100%.

6.5.2.5 Determine the volume, **VA**, in µL of chemical necessary to prepare the standard using the following equation.

$$\mathbf{VA} = (\mathbf{C}*\mathbf{V})/(10*\mathbf{P}*\mathbf{D})$$

6.5.2.6 Partially fill a **V** mL volumetric flask with purge and trap methanol.

6.5.2.7 Add **VA** µL of the chemical. Note more than one chemical may be added to a solution using this procedure.

6.5.2.8 Dilute to volume and invert three times to mix.

6.6 Tuning Solution: The tuning solution is prepared containing 50 µg/mL of 4-Bromofluorobenzene in P+T (purge and trap) Methanol.

6.6.1 Place about 48 mL of P+T methanol in a 50 mL Class A volumetric flask.

6.6.2 Add 1.25 mL of Restek's 2000 µg/mL 4-Bromofluorobenzene mix, Cat. #30026, to the methanol, dilute mixture to volume, and invert 3 times to mix.

6.6.3 Note: Preparations of varied amounts are acceptable for this standard as long as the ratio of BFB to methanol is 1:40 (50 µg/mL).

6.6.4 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.7 Internal Standard (IS) solution: The internal standard solution is prepared containing 150 µg/mL of the 8260A internal standards. This standard is abbreviated as 826IS. Note, since the auto sampler adds the spiking solution, the sample loop must be calibrated according to manufacturer's instructions and the IS solution may need to be prepared at a slightly different concentration to spike the standards at 30 ppb. Record the preparation of the alternate solution in the standard logbook with the identification of the system it will be used with.

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6.7.1 The IS solution contains the following compounds at 150 µg/mL: chlorobenzene-d5, fluorobenzene, and 1,4-dichlorobenzene-d4.

6.7.2 IS solution preparation. (Note: The ratio of standard to methanol will remain the same if different volumes are prepared.)

6.7.2.1 Place about 23 mL of P+T methanol in a 25 mL Class A volumetric flask.

6.7.2.2 Add 1.5 mL of Restek's 2500 µg/mL 8260A Internal Standard Mix, cat. #30241, dilute mixture to volume, and invert 3 times to mix.

6.7.2.3 This solution has a six-month expiration from the date prepared and is stored in the volatile standards freezer.

6.8 8260 Surrogate Solution. The 8260 surrogate solution is prepared containing 150 µg/mL of the 8260A surrogate compounds. This solution is used to prepare samples. This standard is abbreviated as **826SS**. Note: since the auto sampler adds the spiking solution, the sample loop must be calibrated according to manufacturer's instructions and the SS solution may need to be prepared at a slightly different concentration to spike the standards at 30 ppb. Record the preparation of the alternate solution in the standard logbook with the identification of the system it will be used with.

6.8.1 The 8260 surrogate solution contains the following compounds at 150 µg/mL: 4-bromofluorobenzene, 1,2-dichloroethane-d4, dibromofluoromethane, and toluene-d8.

6.8.2 8260 surrogate solution preparation. Note: Any volume can be prepared as long as the ratio of surrogate mix to methanol is 1:50.

6.8.3 Place about 48 mL of P+T methanol in a 50 mL Class A volumetric flask.

6.8.4 Add 1.0 mL of Restek's 2500 µg/mL 8260A Surrogate Standard Mix, cat. #30240, dilute mixture to volume, and invert 3 times to mix.

6.8.5 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.9 High 8260 Surrogate Solution. The 8260 surrogate solution is prepared containing 250 µg/mL of the 8260A surrogate compounds. This solution is used to prepare initial calibrations. This standard is abbreviated as **H826SS**.

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6.9.1 The H8260 surrogate solution contains the following compounds at 250 µg/mL: 4-bromofluorobenzene, 1,2-dichloroethane-d4, dibromofluoromethane, and toluene-d8.

6.9.2 H8260 surrogate solution preparation. Note: Any volume can be prepared as long as the ratio of surrogate mix to methanol is 1:10.

6.9.2.1 Place slightly less than 9 mL of P+T methanol in a 10 mL volumetric flask.

6.9.2.2 Add 1.0 mL of Restek's 2500 µg/mL 8260A surrogates mix, Cat. #30240, to the methanol.

6.9.2.3 Dilute mixture to volume.

6.9.2.4 Invert the flask 3 times to mix and place the solution into labeled sub-vials.

6.9.2.5 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.10 Calibration Solutions.

6.10.1 The following stock solutions are purchased to prepare the calibration and calibration checkstandards:

Name	***Catalog #***	***Abbreviation***
502.2 CAL2000 MEGA MIX	30431	VCSMEGA
Custom V standard Acrolein	54588	VCS Acrolein
8260B Acetates Mix	30489	VCS Acetates
Custom Ketones Mix (10,000 µg/mL)	559848	VCS Ketones
2-Chloroethylvinylether Standard	30265	VCS 2 CEVE
502.2 Calibration mix 1A	30439	V Gas
Custom VOA Additions Mix (2000-50,000 µg/mL)	559847	VCS Additions

6.10.2 The concentrations of the stock solutions and their concentration in the calibration solutions are recorded in appendix A.

6.10.3 Preparation of VOA NEW, a secondary stock standard.

6.10.3.1 Add approximately 3.0 mL (but no more than 4.0 mL) of P+T methanol to a 10 mL volumetric flask.

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6.10.3.2 Add 1.0 mL of the following stock standards: VCSMEGA, VCS Acetates, VCS Ketones, VCS Additions, VCS Acrolein, and VCS 2 CEVE.

6.10.3.3 Dilute mixture to volume.

6.10.3.4 Invert the flask 3 times to mix and place the solution into labeled sub vials.

6.10.3.5 This solution has a two-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.11 Quality control sample solutions.

6.11.1 The following stock solutions are purchased to prepare the matrix spikes and blank spikes:

Name	***Catalog #***	***Abbreviation***
502.2 CAL200 MEGA Mix	30432	QCS MEGA
Custom Acetates Mix	560215	QCS Acetates
Custom Q Acrolein	54589	QCS Acrolein
Custom 2-Chloroethylvinylether Std	560216	QCS2CEVE
Custom Q Gases	52911	QGAS
Custom Ketones Mix (1000µg/mL)	560214	QCS Ketones
Custom VOA Additions Mix (200-5000 µg/mL)	560213	QCS Additions

6.11.2 The concentrations of the stock solutions and their concentration in the calibration verification solution are recorded in Appendix B.

6.11.3 Preparation of QVOALCS, a secondary stock standard.

6.11.3.1 Add approximately 6.0 mL (but no more than 7.0 mL) of P+T methanol to a 10 mL volumetric flask.

6.11.3.2 Add 0.5 mL of the following stock standards: QCSMEGA, QCS Acetates, QCS Ketones, QCS Additions, QCS Acrolein, and QCS2CEVE.

6.11.3.3 Dilute mixture to volume.

6.11.3.4 Invert flask 3 times and place into labeled sub-vials.

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6.11.3.5 This solution has a two-month expiration from the date prepared and is stored in the volatile standards freezer.

6.12 Antifoam Agent (Sigma – SE-15, product #A8582 or equivalent).

6.12.1 10 µL of antifoam is added to 5 mL of sample. An antifoam blank shall precede any samples run with antifoam to prove that there are no target analytes present in the antifoam solution. If using an Archon sampler, add 100 µL to the 40mL vial.

6.13 DPD Free Chlorine Reagent, HACH # 21055-60, or equivalent.

7 Instrument Calibration

7.1 The specific configuration of each volatile instrument (ms01, ms03, ms05, and ms07) is recorded in the instrument's maintenance logbook.

7.2 The purge and trap program of each volatiles instrument is recorded in the instrument maintenance logbook.

7.3 The GC/MS methods are printed out with each analytical batch. This includes the tune reports and the methods. NOTE: If no changes have been made to the methods, photocopies of the latest updated method can be produced from the originals. These copies are archived with the associated raw data.

7.4 Tuning Requirements. Before the beginning of the analysis of samples, blanks, MS/MSDs, duplicates, or standards; the instrument must be hardware tuned to meet the requirements stated below.

7.4.1 Inject or purge 1.0 µL (50 ng of BFB) of the tuning solution into the instrument.

7.4.2 When the run is complete, process the data on the Target system using the BFB method.

7.4.3 BFB performance may be evaluated using the following scans: apex, left of the apex, right of the apex, average of the apex (left, apex, and right), average of the entire BFB peak, or any of the preceding with background subtraction. Note: if background subtraction is performed, the background scan must elute within 10 scans before BFB begins to elute.

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NOTE: **The current software criteria set up in our BFB method uses the average of three scans: the apex, scan left of the apex, and scan right of the apex. These criteria satisfy DoD specifications.**

7.4.4 Compare the performance of BFB to the following table:

Mass (m/z)	Abundance Criteria
50	15 to 40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% Relative abundance
96	5-9% of m/z 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

7.4.5 When the instrument meets the above requirements analysis may begin and continue for 12 hours from the injection of the tuning solution. For example, if the tuning solution is injected at 0100 on 12/1/98, the last sample may inject at 1300 on12/1/98.

7.5 Initial calibration.

7.5.1 Each instrument in the laboratory could have a specific initial calibration curve analyzed on it but mainly use the following concentrations.

7.5.1.1 The following calibration standards are analyzed for the initial calibration, VSTD001, VSTD005, VSTD020, VSTD050, VSTD100, and VSTD200.

7.5.2 Prepare calibration solutions following the table in appendix A. For the Archon auto sampler, simply place these solutions in a 40 mL VOC vials, the auto sampler will add the IS solution automatically.

NOTE: Appendix A allows for surrogate standards to be elevated along with the same concentration of each individual calibration standard resulting in a multi-point calibration.

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7.5.4 Calibration Criteria.

7.5.4.1 The average fit must have a %RSD of less than 15% over at least five calibration points (Six points are normally analyzed). It is acceptable to drop calibration points from either end of the calibration. Note: if the high calibration standard is dropped for a compound, the limit of quantitation must be lowered to the next calibration concentration for that compound.

7.5.4.2 The calibration curves are generated with the Target software. A primary or secondary curve fit may also be used for any compound with a %RSD greater than 15%, but shall be less than 50%. (**DoD requires less than %30**)

7.5.4.2.1 Power and quadratic fits require 6 points. The R value must be 0.99 or greater. **(DoD requires ≥ 0.995)**Our Target software uses R^2 so it must be ≥ 0.98 and 0.99 for DoD.

7.5.4.2.2 The linear primary fit requires at least 5 points. The R value must be 0.99 or greater. **(DoD requires ≥. 995)** Our Target software uses R^2 so it must be ≥ 0.98 and 0.99 for DoD.

7.5.4.2.3 A %RSD of <30% must be achieved for the Calibration Check Compounds (CCCs) before the curve passes. If it is between 15% and 30% RSD, a primary or secondary curve fit must be used. The CCCs are as follows:

1,1-Dichloroethane
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

7.5.5 Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time can be explicitly identified only if the resolution between isomers in a standard is acceptable. Acceptable resolution is achieved if the baseline to valley height between isomers is less than 25 % of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. In other words, sum the isomers and report any results as total of the isomer X and Y. Isomers of this type need to be calibrated as a sum also.

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7.5.5.1 After an initial calibration, a blank spike (LCS) must be analyzed before the analysis of samples can begin. The recoveries of the LCS must fall within laboratory acceptable limits for each compound of interest. If these recovery limits are met, samples may be run under the initial calibration to finish the 12-hour tune. **DoD requires** the second source verification meet 25% recovery of expected value.

7.6 GC/MS calibration verification

7.6.1 Prior to the analysis of samples, inject the BFB standard. The resultant mass spectra for the BFB must meet all of the criteria as stated in the standard operating procedure for tuning the GC/MS system. These criteria must be demonstrated each twelve hours of operation.

7.6.1.1 When the analysis of **DoD samples** is to take place, BFB must meet criteria by the average of three (3) scans. The scans include the apex, the scan left of the apex, and the scan right of the apex.

7.6.2 The initial calibration curve for each compound of interest must be checked and verified once every twelve hours during analysis with the introduction technique used for samples. Analyzing a 50 ppb calibration standard and checking the SPCCs and CCCs accomplish this.

Note: When DoD samples are to be analyzed for 15 or less analytes, all of the target analytes shall meet the same criteria as the CCCs.

7.6.2.1 When the analysis of **DoD samples** is to take place, a continuing calibration standard may need to be analyzed following the samples in a 12-hour period.

7.6.3 System Performance Check Compounds (SPCCs) - Once the continuing calibration check standard is analyzed (Section 7.6.2), the SPCCs are checked. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check applied during the initial calibration. If the minimum relative response factors are not met, the system must be evaluated and corrective action must be taken 4 before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

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7.6.3.1 The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.6.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed below are used to check the validity of the initial calibration. If the %RSD for each CCC is less than or equal to 20%, the initial calibration is assumed valid. If the criterion is not met (> 20% RSD), for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new six-point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

1,1-Dichloroethane
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

7.6.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the midpoint (50) standard of the last initial calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to +100%) from the midpoint (50) standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary. NOTE: During the course of a 12-hour tune period, all samples and blanks must also follow these criteria when referenced against the continuing calibration standard run in that tune period.

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8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 ALSI operates a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory shall maintain records to document the quality of the data generated. Ongoing quality checks are compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard shall be analyzed to confirm that the measurements were performed in an in-control mode of operation. (i.e.: If the MS/MSD fails, an LCS shall be analyzed.) It is the practice of the GC/MS department to analyze a laboratory control sample in every 12-hour tune period.

8.3 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.9. If the analyst meets the acceptance criteria, they are now capable of running actual samples. Ongoing proficiency must be established annually as specified in the QA plan, Technical Training.

8.4 Each day a reagent water blank must be analyzed to demonstrate that interferences from the analytical system are under control.

8.5 The method blank shall be performed at a frequency of one per 12-hour tune period per matrix type or preparation method. The results of this analysis shall be one of the QC measures to be used to assess tune period acceptance. The source of method blank contamination shall be investigated, and measures taken to correct, minimize, or eliminate the problem if the concentration exceeds one-half the reporting limit. If one-half the reporting limit (RL) is exceeded, the laboratory shall evaluate whether reanalysis of the samples are necessary, based on the following criteria:

8.5.1 The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparatory batch, or

8.5.2 The blank contamination is greater than 1/10 of the project specified limit.

8.5.3 Any samples associated with a blank that fail these criteria shall be reanalyzed, except when the sample analysis resulted in a non-detect. If no sample volume remains for reanalysis, the results shall be reported with appropriate data qualifying codes.

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8.5.4 The current laboratory practice is to comment on a sample associated with a method blank in which one or more analytes were detected at or above the reporting limit in the blank and also in the sample. (i.e. If j-values are detected, no comment is necessary)

8.6 The laboratory must, on an ongoing basis, analyze a spike and spike duplicate on a minimum of 5 % of all samples to monitor and evaluate laboratory data quality. It is the policy of the GC/MS department to spike one sample per every 20 samples. Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.6.1 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter.

8.6.2 Spike a second 40 mL sample vial with 86 µL and 43 µL of the QVOALCS/QGAS(see Section 6.11 and Appendix B) using an 100 µL gastight syringe and analyze it twice, and determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as:

$$P = \frac{\text{Spiked sample conc. - unspiked sample conc.}}{T} \times 100\%$$

where T = the known true value of the spike

8.6.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in the most current listing of QC recovery limits.

8.6.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. However, since some failures may occur due to sample matrix interferences, if the LCS (Section 8.7) passes the set criteria for those failing compounds, the system performance is acceptable. In this case, a comment needs to go on the background sample stating that one or more compounds failed in the MS/MSD associated but passed in the associated LCS.

8.7 If any parameter fails the acceptance criteria for recovery in Section 8.6, a Laboratory Control Sample (LCS) containing each parameter that failed must be prepared and analyzed. Note: The current practice is to run an LCS at the same frequency as the method blank, which is one per 12-hour tune period. This is more than the method requires but allows for QC to be more closely associated with each sample in that 12-

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hour tune period.

8.7.1 Prepare a Laboratory Control Sample (LCS) by using a 100 or 250 µL gastight microliter syringe and adding 50 µL of QGAS and 100 µL of QVOALCS listed in Section 6.11 to 50 mL of reagent water and analyze.

8.7.2 Analyze the Laboratory Control Sample (LCS) to determine the concentration measured (A) of each failed parameter. Calculate each percent recovery (P_s).

8.7.3 Compare the percent recovery (P_s) for each failed parameter with the corresponding LCS acceptance criteria found in the latest control charts generated for the Method 8260 LCS. **DoD requires** specific acceptance criteria found in the DoD Quality Systems Manual. If the recovery of any such parameter, which failed in the MS/MSD, falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter, if detected in any samples associated with that LCS, is suspect and shall not be reported for regulatory compliance purposes. If any results are reported, a comment must accompany that result stating the failure and the possibility of a low or high bias to the data. If the LCS fails for one or more compounds that met criteria in the associated MS/MSD, the MS/MSD can prove that the instrument performance is valid as long as the acceptance criteria are as stringent as the criteria for the LCS.

8.7.4 It will be the judgment of the analyst and/or supervisor to approve the data acquired using the initial calibration. If evaluation of the system in addition to the failed QCs indicates a lack of integrity of the data, it will be reanalyzed.

8.8 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solution and calculate the percent recovery of each surrogate compound. Recoveries must fall within the calculated limits. See Section 8.13 for the development of surrogate control limits. If the surrogate recoveries do not fall within the calculated limits, the sample shall be re-analyzed and the system shall be evaluated for malfunctions. If the surrogate recovery is acceptable in the re-analysis, report the data from the re-analysis. If data must be reported in which a surrogate(s) is out, report with a comment.

8.8.1 HORIZON LIMS standard verbiage comment V8L - One or more 8260 surrogates were recovered outside of the recovery limits. Then it lists the current limits.

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8.8.2 HORIZON LIMS standard verbiage comment VSM - One or more volatile surrogate(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

NOTE: A sample with a surrogate out will normally be re-analyzed followed by reporting with a comment VSM. However, if holding time is expired, comment V8L will be used, and the sample will not be re-analyzed.

8.9 To establish the ability to generate accuracy and precision, the analyst must perform the following operation as an initial demonstration of capability.

8.9.1 A quality control (QC) check sample concentrate is prepared containing each parameter of interest. The concentrate must be from an external source, different from the source used for the calibration standards.

8.9.2 Using a 100 or 250 µL gastight syringe, inject 50 µL and 100 µL of the QGAS/QVOALCS solutions into a 50 mL volumetric flask containing reagent water. This is done four (4) times. See Appendix B.

8.9.3 Calculate the average recovery ($\overline{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter of interest using the four results.

8.9.4 For each parameter, compare s and X with the DOC forms of the corresponding acceptance criteria in the latest control charts generated for 8260B MS/MSDs, respectively. If s and X for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of samples can begin. If any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, the system performance is unacceptable for that parameter.

8.10 If one or more of the parameters tested fail at least one of the acceptance criteria from Section 8.9, the analyst must proceed according to Section 8.10.1 or 8.10.2.

8.10.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.9.2.

8.10.2 Beginning with Section 8.9.2, repeat the test only for those parameters that failed. Repeated failure will confirm a general problem with the measurement system. If this occurs, locate and correct the problem and repeat the test for all compounds of interest beginning with Section 8.9.2.

8.11 The laboratory must, on an ongoing basis, demonstrate through the analysis of

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Laboratory Control Samples (LCSs) that the operation of the measurement system is in control. The procedure is described in Section 8.7. The frequency of the check standard analysis is equivalent to 5 % of all samples analyzed. This 5% is equivalent to 1 in 20 samples, which is what the GCMS department defines as a batch. This may be reduced if spike recoveries from samples (see Section 8.6) meet all specific control criteria. It is the practice of the GCMS department to analyze a LCS every 12-hour tune period even though the method does not require it. A batch will typically consist of two or more 12-hour tune periods until 20 samples have been analyzed including all QC.

8.12 The laboratory must maintain performance records to document the quality of data generated.

8.13 As part of the QC program, control limits for samples must be assessed and records must be maintained. After the analysis of at least 20 spiked samples as in Section 8.6, calculate the average percent recovery (P) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from P - $3s_p$ to P + $3s_p$ (i.e., If P = 100% and s_p = 10%, the accuracy interval is expressed as 70 - 130%). Update the accuracy assessment for each parameter at least annually.

8.14 MDL Studies. Method detection limit studies are performed annually to statistically determine the concentration levels an analytical system is capable of determining. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent. Reporting limits are set approximately 3-5 times the method detection limit, but not lower than the lowest initial calibration standard. (For DoD reporting purposes, reporting limits are set at least 3 times the MDL, and not more than 10 times the MDL.)

8.14.1 The group leader will determine at what level (concentration) the MDL study will be performed. At least seven replicates are to be analyzed. All replicates analyzed must be included in the MDL study. Note: The current concentration used for MDL studies is 0.8 µg/L.

8.14.2 The QLCS and the QGAS standards can be used to prepare the MDL studies, but using the initial calibration standards, VOANEW and VGAS, is a more commonly used method.

8.14.3 Use the Target software to generate an MDL study report.

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9 Sample Collection, Preservation and Handling

9.1 Sample Collection

9.1.1 Standard 40 mL glass screw-cap VOA vials with Teflon-lined silicone septa may be used for liquid samples.

9.1.2 When collecting the samples, liquids shall be introduced into the vials gently to reduce agitation, which might drive off volatile compounds. In general, liquid samples shall be poured into the vial without introducing any air bubbles within the vial as it is being filled. Shall bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. The vials shall be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The sample shall be hermetically sealed in the vial at the time of sampling, and must not be opened prior to analysis to preserve their integrity.

9.1.2.1 Due to differing solubility and diffusion properties of gases on liquid matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and shall not invalidate a sample for volatile analysis.

9.1.2.2 The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial (e.g., carbonate-containing samples preserved with acid). Studies conducted by the USEPA (EMSL-Ci, unpublished data) indicate the Pea-sized bubbles (i.e., bubbles not exceeding 1/4 inch or 6 mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are more susceptible to variations in gas solubility than are groundwater samples. NOTE: **For DoD samples**, any size air bubble will be commented on the lab report. See Section 9.3.2.

9.1.3 If the aromatic compounds benzene, toluene, and ethyl benzene are to be determined; a second separate sample shall be collected as follows because refrigeration alone may not preserve these compounds for more than seven days. Collect about 500 mL of sample in a clean container. Adjust the pH to about 2 while stirring vigorously by adding 1+ 1 HCl. Check the pH with narrow range (1.4 to 2.8) pH paper. Fill the sample vial as described in Section 9.1.2.

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9.2 Sample Preservation

9.2.1 Preserve aqueous samples using HCl to a pH <2. Sample preservation shall be performed immediately upon sample collection. The sample shall then be iced above the freezing point of water up to 6°C in transport.

9.2.2 Ascorbic acid preservative is added to the vial prior to shipping to the sample site if the sample contains residual chlorine. Immediately following collection of the sample, shake the vial vigorously for one minute. Maintain the hermetic seal until the time of sample analysis.

9.2.3 Once samples are received, they must be refrigerated above the freezing point of water up to 6°C until analysis.

9.3 Sample Handling

9.3.1 All samples must be analyzed within 14 days of collection. All samples not analyzed within this time frame must be discarded and re-sampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.

9.3.1.1 If a dilution of the sample was analyzed after the hold time due to compounds exceeding the calibration range in the initial (and reportable) analysis, the standard verbiage comment VDL in the HORIZON LIMS may be used.

9.3.2 Check the run logbook to see if there was headspace in the sample. Add a comment to the report if it was present. The standard verbiage code for this in the HORIZON LIMS is HSP.

9.3.3 Check the run logbook to see if the pH of the sample was greater than 2. Add comment to the report if it exceeded 2. The standard verbiage code for this in the HORIZON LIMS is PH>.

9.3.4 Chlorine: Check the run logbook to see if there was free chlorine in the sample. Add a comment to the report if it was present.

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10 Procedure

10.1 Daily tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

10.2 All samples must be allowed to warm to ambient temperature before analysis.

10.3 Sample preparation and analysis.

10.3.1 Blanks must be free from target analytes above the established reporting limits.

10.3.1.1 Using the Archon autosampler, fill a 40 mL VOC vial with reagent water and cap, load on the autosampler and run. The Archon will add the IS/SS automatically.

10.3.2 Laboratory Control Samples (LCSs).

10.3.2.1 Using the Archon autosampler, place slightly less than 50 mL of reagent water in a 50 mL volumetric flask. Add 100 µL of QVOA LCS and add 50 µL of QGAS. Dilute to volume, invert 3 times, and place in a 40 mL VOC vial. The Archon will add the IS/SS automatically.

10.3.3 Samples:

10.3.3.1 Note in the run logbook whether or not the sample has headspace. Mark a Y in the HSP column if there is an air bubble bigger than a large pea (one quarter inch in diameter). Otherwise, mark an N in this column.

10.3.3.2 In order to prevent system overload it is a good practice to check each sample's history in the LIMS before analyzing the sample. If the sample has no history, then immediately before loading the instrument, open the vial, lift to within approximately 2-3 inches of the nose, wave a hand across the top of the sample towards the nose. Run the sample at a dilution if it has a polluted or organic chemical odor.

10.3.3.3 To composite samples, gently pour the sample containers into a clean appropriately sized beaker. Swirl the beaker gently to mix, and pour contents back into the sample vials. Mark the sample vials with a "C" to denote that they were composited. NOTE: Only at a client's request are 8260 samples ever composited.

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10.3.3.4 Prepare sample dilutions according to the following table (Note: This table may not be inclusive of every dilution that will need to be performed. It may be necessary to perform intermediate dilutions or to use a larger volumetric flask to perform the larger dilutions): Note: **For DoD samples;** dilutions shall be limited to steps that are less than 100-fold.

Dilution factor	*Water volume*	*Sample added*	*Final Volume*
2	2.5mL	2.5mL	5.0mL
2	25mL	25mL	50mL
4	3.75mL	1.25mL	5.0mL
4	3.75mL	12.5mL	50mL
5	4.0mL	1.0mL	5.0mL
5	40mL	10mL	50mL
10	4.5mL	0.5mL	50mL
10	45mL	5.0mL	50mL
20	4.75mL	250µl	5.0mL
20	47.5mL	2.5mL	50mL
50	4.9mL	100µl	5.0mL
50	49mL	1.0mL	50mL
100	4.95mL	50µl	5.0mL
100	49.5mL	500µl	50mL
200	4.975m	25µl	5.0mL
200	49.75mL	250µl	50mL
500	4.99mL	10µl	5.0mL
500	49.9mL	100µl	50mL
1000	5.0mL	5.0µl	5.0mL
1000	50mL	50µl	50mL
2000	5.0mL	2.5µl	5.0mL
2000	50mL	25µl	50mL
5000	5.0mL	1.0µl	5.0mL
5000	50mL	10µl	50mL
10000	50mL	5.0µl	50mL
20000	50mL	2.5µl	50mL
50000	50mL	1.0µl	50mL

10.3.3.5 Dilutions with a 5.0 mL final volume are prepared in the following manner.

10.3.3.5.1 Rinse a 5.0 mL syringe several times with reagent water.

10.3.3.5.2 Fill the syringe with reagent water and adjust the water volume to the appropriate mark.

10.3.3.5.3 Move the plunger back to 5 mL.

10.3.3.5.4 Quickly add the volume of sample, then prepare for the appropriate autosampler.

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10.3.3.6 Prepare dilutions in Class A volumetric flasks in the following manner.

10.3.3.6.1 Partially fill a clean volumetric with reagent water.

10.3.3.6.2 Quickly add the appropriate volume of sample.

10.3.3.6.3 Quickly dilute to volume. Invert 3 times to mix. Prepare for the appropriate autosampler.

10.3.3.7 Using the Archon autosampler, place the sample vial (it may be necessary to remove the label) or a capped 40 mL VOC vial filled with diluted sample on the autosampler, and run. The Archon will add the IS/SS automatically.

10.3.3.8 Antifoam may be added to prevent foaming. It is added 100 µL at a time to a 40 mL vial or 50 mL flask, or 10 µL to a 5 mL syringe.

10.3.3.9 Check the pH of the unused sample (this includes sample spikes if they were taken from a different vial) with pH paper. Record in column of the run logbook either the pH or whether or not the pH was less than or equal to 2 denoted "<2", if greater than 2 it is denoted ">2".

10.3.3.10 Check for the presence of free chlorine with an aliquot of DPD free chlorine reagent added to 10 mL of sample. It will turn pink if chlorine is present. In the Cl column of the run logbook, record a "Y" if free chlorine was detected, record an "N" if it was not. Note: We use a dispenser purchased from HACH to determine the aliquot size.

10.3.3.11 If running low or med level soils by 8260B, see the SOP for the 5035 Method (19-5035).

10.3.4 Sample spikes (MS/MSD).

10.3.4.1 Using the Archon auto sampler, inject 86 µL of QVOALCS and 43 µL of QGAS through the vial's septum. Shake or roll the vial to mix contents. Place the sample vial (it may be necessary to remove the label) or a capped 40 mL VOC vial filled with diluted sample on the auto sampler and run. The Archon will add the IS/SS automatically.

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10.3.5 Duplicates.

10.3.5.1 Prepare duplicates the same way as normal samples. Duplicates shall only be prepared on samples that have historically had target hits. Otherwise, matrix spike duplicates shall be analyzed. The RPD acceptable limit is <40 %. **DoD requires** the RPD acceptable limit is <30%.

10.4 Data analysis.

10.4.1 Tune time. All analyses must have an injection time within 12 hours of the injected 50 ng bfb tuning solution, which met method criteria. The only exception to this is ending calibration checks that are requested by some clients. They shall run within 24 hours of the bfb (immediately after the last sample in the sequence).

10.4.2 Internal standard areas and retention times.

10.4.2.1 The internal standard areas in each analysis shall be within a factor of two of their abundance in the calibration verification standard. Note, this is not only a method requirement, but also a good laboratory practice as it helps to ensure that the instrument system is functioning normally.

10.4.2.2 The retention times of internal standards in each analysis shall be within 0.5 minutes of their retention times in the calibration verification standard. Note, this is not only a method requirement, but also a good laboratory practice as it helps to ensure that the instrument system is functioning normally.

10.4.2.3 The internal standard areas may vary due to the following reasons: instrument malfunction, wrong amount of IS/SS mix added, partially open sample valve, sample overload (i.e., shall have been run very diluted), etc.

10.4.2.3.1 ALSI LIMS standard verbiage comment VIS – One or more volatile internal(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

10.4.2.4 The retention times may vary due to the following reasons: sample overload, sample foaming, plug or obstruction in the instrument, instrument malfunction, unstable room temperature, etc.

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10.4.3 Surrogates.

10.4.3.1 The surrogates shall be recovered within the recovery limits. If they are not the sample shall be re-analyzed and the system shall be evaluated for malfunctions. If the surrogate recovery is acceptable in the re-analysis, report the data from the re-analysis. If data must be reported in which a surrogate(s) is out, report with a comment.

10.4.3.1.1 HORIZON LIMS standard verbiage comment V8 - One or more 8260 surrogates recovered outside of the recovery limits. Then it lists the current limits.

10.4.3.1.2 HORIZON LIMS standard verbiage comment VSM - One or more volatile surrogate(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

NOTE: A sample with a surrogate out will normally be re-analyzed followed by reporting with a comment VSM. However, if holding time is expired, comment V8 will be used, and the sample will not be re-analyzed.

10.4.4 Target hits.

10.4.4.1 Positive hits. Identified by comparing the mass spectrum of the compound with a reference mass spectrum of the compound from a standard, which was analyzed on the same instrument. Obtain EICPs (the overlays on the right of the Target review window) for the primary (quantitating) mass and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification.

10.4.4.1.1 The characteristic masses of each parameter of interest must maximize in the same scan or within one scan of each other. Beware of co eluting interferences.

10.4.4.1.2 The retention time must fall within ± 30 seconds of the retention time of the compound in the daily QC calibration verification check.

10.4.4.1.3 The relative peak heights of the characteristic masses in the EICPs must fall within ± 20 % of the relative intensities of the masses in a reference mass spectrum.

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10.4.4.2 Negative hits. Hits which do not meet the requirements for a positive hit are marked as unknown on Target.

10.4.4.3 Over-range hits. If a requested target analyte is a positive hit (be aware that if an analyte is present at a high enough concentration, its mass spectrum may be distorted) and exceeds the instrument's initial calibration range, the sample shall be rerun at a dilution until the analyte is within the instrument's calibration range (preferably in the upper half of the calibration range). If internal and surrogate recoveries are acceptable in the original analysis, all compounds except those that exceeded the calibration range will be reported from this run. Those that did exceed the calibration range will be reported from the dilution analysis (DL).

10.4.4.4 If manual integrations are required, the procedure in SOP 99-Integration is followed.

10.4.5 Blanks: Blanks shall have no target hits present at the reporting limit. A blank's surrogates shall be within the surrogate recovery limits. If a requested analyte is present above the reporting limit in the blank and it is present above the reporting limit in a sample(s), the sample shall be rerun. If that is not possible fill out a corrective action form explaining the situation and report the data with a comment similar to the following: "This sample had a hit of 8 µg/L of TCE which was present in the associated method blank at 1 µg/L." **NOTE**: **For DoD samples,** blanks shall have no target hits present above 2 times the calculated MDL. Blanks must not exceed one-half the reporting limit. See Section 8.5 under quality control for additional information.

10.4.6 QC samples (MS/MSD, blank spikes, duplicates).

10.4.6.1 Generate a MS/MSD report (form 3) using Quickforms in the Target software.

10.4.6.2 Compare the percent recovery, P, of each parameter with the corresponding QC acceptance criteria. If the spike sub-list MS/MSD.spk is used, these shall be the limits present on MS/MSD report generated with Quickforms.

10.4.6.3 If any individual P falls outside the designated range for recovery in either the MS or MSD, that parameter has failed the acceptance criteria. A blank spike containing each parameter that failed shall have been analyzed.

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10.4.6.3.1 Analyze the blank spike to determine the concentration measured (A) of each failed parameter. Compounds which did not fail in the MS/MSD are not considered. The target software will calculate the percent recoveries of the blank spike. Use the spike sub-list WATERQC. The equation to calculate percent recovery (P) in a blank spike follows. T is the known true value of the spike.

$$P = A * 100\% / T$$

10.4.6.3.2 Compare the percent recovery, P, of each parameter with the corresponding QC acceptance criteria found in Appendix C. If the spike sub-list QATERQC is used, these shall be the limits present on blank spike report generated with Quickforms.

10.4.6.3.3 If the recovery of any such parameter, P, falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes. If the data must be reported due to either the samples hold time or a lack of sufficient sample for re-analysis, fill out a corrective action form explaining the problem, and comment on the sample report.

10.4.6.3.4 It will be the judgment of the analyst and/or supervisor to approve the data acquired using this initial calibration. If evaluation of the system in addition to the failed QCs indicates a lack of integrity of the data, the samples will be reanalyzed.

10.4.6.4 Duplicates (including MSD) shall have a % Repeatability of 40 % or less. NOTE: **For DoD samples**, control limits calculated by using LCS data will be used to determine acceptable Relative Percent Difference.

10.4.7 Library searches.

10.4.7.1 The selection and quantitation of non-target peaks is performed automatically by the Target software. Note: The sample shall be processed for client specific compounds only. Otherwise, the Target

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software will not pick any other target compounds as library peaks. If a sublist containing client specific analytes is not used, the analyst must add, to the library search form, any compounds that were detected in the analysis that the client does not want reported. Also, if any changes have been made to the sample such as deleting or integrating peaks, the library search must be redone by selecting "process unknowns" in Target.

10.4.7.2 If the software selects any internal standard, surrogate, or target compounds, delete them in Target. The air peak (the first large peak on the chromatogram usually, its primary m/e will be 44) shall be deleted also. Also, early eluters with mass 40 can be deleted.

10.4.7.3 The Target software will perform a library search on and estimate the concentration of the 20 largest non-target peaks.

10.4.7.4 Only after visual comparison of sample spectra with the nearest library searches shall a tentative identification be assigned to the peak. The analyst will need to search each non-target peak on the Target system to view the library searches. Consider the following sets of guidelines before making a tentative identification.

10.4.7.5 Guidelines for making tentative identification of non-target compounds. In other words, making a specific identification of non-target compounds, i.e., limonene, hexamethylbenzene. These guidelines are from a contract laboratory program statement of work.

10.4.7.5.1 Major ions (ions greater than 10% of the most abundant ion) in the reference spectrum shall be present in the sample spectrum.

10.4.7.5.2 The relative intensities of the major ions shall agree within plus or minus 20%. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

10.4.7.5.3 Molecular ions present in the reference spectrum shall be present in the sample spectrum.

10.4.7.5.4 Ions present in the reference spectrum but not in the sample spectrum shall be reviewed for possible

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subtraction from the sample spectrum because of background contamination or co eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.7.5.5 If in the analyst's technical judgment, no valid identification is possible, the compound shall be reported as unknown. If possible give an additional classification to the compound (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound, etc.). If the probable molecular weights can be determined, include them.

10.4.7.6 Guidelines for making tentative ID based on match quality.

10.4.7.6.1 If a non-target compound is present in the calibration mixes it can be identified as that compound no matter how good or bad the match quality is as long as the criteria for identifying the spectra of target compounds are met for those compounds, i.e., hexane, benzyl chloride, 1,2,4-trimethylbenzene, etc.

10.4.7.6.2 If a tentatively identified compound has a match of greater than 90% and the next closest match is greater than 30 % less, tentatively identify the peak as that compound.

10.4.7.6.3 If the match quality of 2 or more isomers are very close together and greater than 70% with no other unrelated compounds within 10 %, identify that peak as _____ isomer. Be as specific as possible.

10.4.7.6.4 Identify classes of compounds if all the compounds above 50 % match belong to the same class, or if the 2 or 3 closest matches belong to one chemical class and the next matches have significantly different match quality.

10.4.7.6.5 Use the analyst's experience when possible. Also, be consistent throughout a group of samples, referring back to retention times as a guide.

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11 Calculations

11.1 All calculations are performed by the Target software.

12 Reporting Results

12.1 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.2 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

12.3 Any sample compound with a result less than the reporting limit is reported with the actual result in the Horizon LIMS. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit. The client may request that "J values" be reported. J values are hits between the reporting limit and the method detection limit. They are reported with a "J" flag.

12.4 If the primary analysis of a sample was diluted, the reporting limits must be raised proportionate to the dilution factor. The following standard verbiage comments in the LIMS may be added to explain to the client why the reporting limits are elevated.

12.4.1 VLE - Sample was run at a dilution due to late eluting non-target compounds.

12.4.2 VNT - Sample was run at a dilution due to the level of non-target compounds.

12.4.3 VTC - Sample was run at a dilution due to the level of target compounds.

12.5 Any errors must be marked through with a single line with the analyst's initial, the date, and the correction.

12.6 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

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14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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TABLE 1
BFB KEY M/Z ABUNDANCE CRITERIA

MASS	M/Z 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	<2% OF MASS 174
174	>50% OF MASS 95
175	5 TO 9% OF MASS 174
176	>95% BUT <101% OF MASS 174
177	5 TO 9% OF MASS 176

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APPENDIX A

Theoretical Standard Concentrations

Initial Calibration
SW846 Method 8260B

VOANEW = 1.0 mL of VCSMEGA, VCS Acetates, VCS Ketones, VCS Additions, VCS Acrolein, and VCS2CEVE to a final volume of 10 mL in P & T methanol.

Prepare the 6 initial calibration standards from the following table:

Standard ID	Volume Added			Flask Volume (mL)
	VOANEW	VGas	H826SS	
VSTD200	50 µL	50 µL	40 µL	50
VSTD100	25 µL	25 µL	20 µL	50
VSTD050	25 µL	25 µL	20 µL	100
VSTD020	20 µL	20 µL	16 µL	200
VSTD005	12.5 µL	12.5 µL	10 µL	500
VSTD001	2.5 µL	2.5 µL	2 µL	500

Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
Benzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromochloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromodichloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromoform	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
n-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Sec-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Tert-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Carbon tetrachloride	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroform	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2-Chlorotoluene (o)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
40Chlorotoluene (p)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dibromochloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dibromo-3-chloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dibromoethane (EDB)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dibromomethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,4-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cis-1,2-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trans-1,2-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

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Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
1,2-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2,2-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cis-1,3-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trans-1,3-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Hexachlorobutadiene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Isopropylbenzene (Cumene)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
p-Isopropyltoluene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methylene Chloride	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Naphthalene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
n-Propylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Styrene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,1,2-Tetrachloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,2,2-Tetrachloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Tetrachloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Toluene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,3-Trichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,4-Trimethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3,5-Trimethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
m-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
p-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
o-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,1-Trichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,3-Trichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,4-Trichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,2-Trichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Vinyl Acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acetone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Butanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
4-Methyl-2-pentanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Hexanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1,1-Dichloro-2-propanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Pentane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
3-Chloroprene (allyl chloride)	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Di-isobutylene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1-Chlorohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl-tert-butyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

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Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
Ethyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Freon 113 (1,1,2-TCTFE)	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Hexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Heptane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cyclohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Benzyl chloride	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Iodomethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Carbon Disulfide	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroprene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Octane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acrylonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Nitropropane	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Tetrahydrofuran	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Tert-Butyl alcohol	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Trans-1,4-Dichloro-2-butene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl methacrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Isobutyl alcohol	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Hexachloroethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl methacrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2-Propanol	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1-Propanol	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Propionitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Methacrylonitrile	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,4-Dioxane	VCS Additions	50000	5000 ppb	2500 ppb	1250 ppb	500 ppb	125 ppb	25 ppb
Pentachloroethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Nitrobenzene	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Methyl acrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroacetonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1-Chlorobutane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dichlorofluoromethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
tert-amyl methyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl tert-butyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Di-isopropyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl cyclohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acetonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1,2,3-Trimethylbenzene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acrolein	V Acrolein	50000	5000 ppb	2500 ppb	1250 ppb	500 ppb	125 ppb	25 ppb
2-Chloroethyl vinyl ether	VCS2CEVE	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromomethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dichlorodifluoromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trichlorofluoromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Vinyl Chloride	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

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APPENDIX B

Theoretical Standard Concentrations
Quality Control Standard / Spike
EPA Method 524.2

QVOALCS = 0.5 mL of QCS MEGA, QCS Acetates, QCS Ketones, QCS Additions, QCS Acrolein, and QCS 2CEVE to a final volume of 10.0 mL in P & T methanol.

Stock mix ID	Volume Added		
	5 mL Final Volume	**50 mL Final Volume**	**100 mL Final Volume**
QVOALCS	10 µL	100 µL	200 µL
QGASES	5 µL	50 µL	100 µL

Compound Name	Standard Mix	Stock (ppm)	Working Conc.
Benzene	QCSMEGA	200	20 ppb
Bromobenzene	QCSMEGA	200	20 ppb
Bromodichloromethane	QCSMEGA	200	20 ppb
Bromoform	QCSMEGA	200	20 ppb
n-Butylbenzene	QCSMEGA	200	20 ppb
Sec-Butylbenzene	QCSMEGA	200	20 ppb
Tert-Butylbenzene	QCSMEGA	200	20 ppb
Carbon tetrachloride	QCSMEGA	200	20 ppb
Chlorobenzene	QCSMEGA	200	20 ppb
Chloroform	QCSMEGA	200	20 ppb
2-Chlorotoluene (o)	QCSMEGA	200	20 ppb
40Chlorotoluene (p)	QCSMEGA	200	20 ppb
Dibromochloromethane	QCSMEGA	200	20 ppb
1,2-Dibromo-3-chloropropane	QCSMEGA	200	20 ppb
1,2-Dibromoethane (EDB)	QCSMEGA	200	20 ppb
Dibromomethane	QCSMEGA	200	20 ppb
1,2-Dichlorobenzene	QCSMEGA	200	20 ppb
1,3-Dichlorobenzene	QCSMEGA	200	20 ppb
1,4-Dichlorobenzene	QCSMEGA	200	20 ppb
1,1-Dichloroethene	QCSMEGA	200	20 ppb
Cis-1,2-Dichloroethene	QCSMEGA	200	20 ppb
Trans-1,2-Dichloroethene	QCSMEGA	200	20 ppb
1,2-Dichloropropane	QCSMEGA	200	20 ppb
1,3-Dichloropropane	QCSMEGA	200	20 ppb
2,2-Dichloropropane	QCSMEGA	200	20 ppb
1,1-Dichloropropene	QCSMEGA	200	20 ppb
Cis-1,3-Dichloropropene	QCSMEGA	200	20 ppb
Trans-1,3-Dichloropropene	QCSMEGA	200	20 ppb
Ethylbenzene	QCSMEGA	200	20 ppb
Hexachlorobutadiene	QCSMEGA	200	20 ppb
Isopropylbenzene (Cumene)	QCSMEGA	200	20 ppb

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Compound Name	Standard Mix	Stock (ppm)	Working Conc.
p-Isopropyltoluene	QCSMEGA	200	20 ppb
Methylene Chloride	QCSMEGA	200	20 ppb
Bromochloromethane	QCSMEGA	200	20 ppb
1,1-Dichloroethane	QCSMEGA	200	20 ppb
1,1,1-Trichloroethane	QCSMEGA	200	20 ppb
1,2-Dichloroethane	QCSMEGA	200	20 ppb
1,2,3-Trichloropropane	QCSMEGA	200	20 ppb
1,2,4-Trichlorobenzene	QCSMEGA	200	20 ppb
1,1,2-Trichloroethane	QCSMEGA	200	20 ppb
Trichloroethene	QCSMEGA	200	20 ppb
Naphthalene	QCSMEGA	200	20 ppb
n-Propylbenzene	QCSMEGA	200	20 ppb
Styrene	QCSMEGA	200	20 ppb
1,1,1,2-Tetrachloroethane	QCSMEGA	200	20 ppb
1,1,2,2-Tetrachloroethane	QCSMEGA	200	20 ppb
Tetrachloroethene	QCSMEGA	200	20 ppb
Toluene	QCSMEGA	200	20 ppb
1,2,3-Trichlorobenzene	QCSMEGA	200	20 ppb
1,2,4-Trimethylbenzene	QCSMEGA	200	20 ppb
1,3,5-Trimethylbenzene	QCSMEGA	200	20 ppb
m-xylene	QCSMEGA	200	20 ppb
p-xylene	QCSMEGA	200	20 ppb
o-xylene	QCSMEGA	200	20 ppb

Methyl acetate	QCS Acetates	200	20 ppb
Ethyl acetate	QCS Acetates	200	20 ppb
Vinyl Acetate	QCS Acetates	200	20 ppb

Acetone	QCS Ketones	1000	100 ppb
2-Butanone	QCS Ketones	1000	100 ppb
4-Methyl-2-pentanone	QCS Ketones	1000	100 ppb
2-Hexanone	QCS Ketones	1000	100 ppb
1,1-Dichloro-2-propanone	QCS Ketones	1000	100 ppb

Pentane	QCS Additions	200	20 ppb
3-Chloroprene (allyl chloride)	QCS Additions	200	20 ppb
Di-isobutylene	QCS Additions	200	20 ppb
1-Chlorohexane	QCS Additions	200	20 ppb
Methyl-tert-butyl ether	QCS Additions	200	20 ppb
Ethyl ether	QCS Additions	200	20 ppb
Freon 113 (1,1,2-TCTFE)	QCS Additions	200	20 ppb
Hexane	QCS Additions	200	20 ppb
Heptane	QCS Additions	200	20 ppb
Cyclohexane	QCS Additions	200	20 ppb
Benzyl chloride	QCS Additions	200	20 ppb
Iodomethane	QCS Additions	200	20 ppb
Carbon Disulfide	QCS Additions	200	20 ppb
Chloroprene	QCS Additions	200	20 ppb

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Compound Name	Standard Mix	Stock (ppm)	Working Conc.
Octane	QCS Additions	200	20 ppb
Acrylonitrile	QCS Additions	1000	100 ppb
2-Nitropropane	QCS Additions	1000	100 ppb
Tetrahydrofuran	QCS Additions	1000	100 ppb
Tert-Butyl alcohol	QCS Additions	1000	100 ppb
Trans-1,4-Dichloro-2-butene	QCS Additions	200	20 ppb
Methyl methacrylate	QCS Additions	200	20 ppb
Isobutyl alcohol	QCS Additions	2000	200 ppb
Hexachloroethane	QCS Additions	200	20 ppb
Ethyl methacrylate	QCS Additions	200	20 ppb
2-Propanol	QCS Additions	1000	100 ppb
1-Propanol	QCS Additions	2000	200 ppb
Propionitrile	QCS Additions	1000	100 ppb
Methacrylonitrile	QCS Additions	200	20 ppb
1,4-Dioxane	QCS Additions	5000	500 ppb
Pentachloroethane	QCS Additions	200	20 ppb
Nitrobenzene	QCS Additions	2000	200 ppb
Methyl acrylate	QCS Additions	200	20 ppb
Chloroacetonitrile	QCS Additions	1000	100 ppb
1-Chlorobutane	QCS Additions	200	20 ppb
Dichlorofluoromethane	QCS Additions	200	20 ppb
tert-amyl methyl ether	QCS Additions	200	20 ppb
Ethyl tert-butyl ether	QCS Additions	200	20 ppb
Di-isopropyl ether	QCS Additions	200	20 ppb
Methyl cyclohexane	QCS Additions	200	20 ppb
Acetonitrile	QCS Additions	1000	100 ppb
Acrolein	Q Acrolein	1500	150 ppb
2-Chloroethyl vinyl ether	QCS2CEVE	200	20 ppb
Bromomethane	Qgas mix	20	20 ppb
Chloroethane	Qgas mix	20	20 ppb
Chloromethane	Qgas mix	20	20 ppb
Dichlorodifluoromethane	Qgas mix	20	20 ppb
Trichlorofluoromethane	Qgas mix	20	20 ppb
Vinyl Chloride	Qgas mix	20	20 ppb

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APPENDIX C

TABLE 2

CALIBRATION AND QC ACCEPTANCE CRITERIA

PARAMETER	RANGE FOR Q (ug/L)	LIMIT FOR s (ug/L)	RANGE FOR X (ug/L)	RANGE FOR P, P, (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon Tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
Chloroethane	7.6-32.4	11.4	8.4-40.4	14-230
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.5	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.5	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethylbenzene	11.8-28.2	7.5	17.4-26.7	47-150
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.6-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

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Appendix D

GC/MS VOLATILE SAMPLE INFORMATION

Instrument MS03

DATE(S):	CALIBRATION VERIFIED BY:	BATCH CONTINUED FROM PAGE:
DATA FILE NAME:	BFB INJECTED: ICAL DATE:	BATCH CONTINUED TO PAGE:
BATCH NAME:	12-HOUR ENDING:	METHOD:

METH #	POS. #	RUN #	SAMPLE	CLIENT	SAMPLE INFORMATION	DIL.	ANALYST	TEST	pH / %M	HDSP / Wt.	Cl-	First Review	COMMENT	SDG

IS: _____ SS: _____ Page: _____

Prev. _____ Current _____ Total _____

Batch: _____ Prev.: _____ Current: _____ Total: _____

Rev. 6/02

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APPENDIX E

Batch Review Checklist

TARGET BATCH: **HORIZON BATCH:**

INSTRUMENT: **METHOD:** **ICAL DATE(S):**

Reviewer 1	Reviewer 2	
_____	_____	BFB Reviewed
_____	_____	Calibration/Continuing Calibration reviewed
_____	_____	Continuing Calibration passes +/- ISTD area - CCAL is zeroed
_____	_____	Correct ICAL referenced (Y__/N__) (correct start/end times/dates (Y__/N__)
_____	_____	Any raised reporting limits due to ICAL (Y__/N__) - See attached spreadsheet
_____	_____	Blank reviewed - Any detections (Y__/N__) If yes, what_____
_____	_____	LCS/second source reviewed - List failures in comment section
_____	_____	MS/MSD reviewed - List failures on unspiked sample
_____	_____	All samples within 12/24-hour tune period (Y__/N__)
_____	_____	Compound retention times reviewed
_____	_____	ISTDs for each sample reviewed
_____	_____	Surrogates for each sample reviewed
_____	_____	Soil weights/volumes correct
_____	_____	Samples checked for over-range detections and put back in for reanalysis
_____	_____	Spectra and Ions have been reviewed for each analyte detected
_____	_____	Library search has been performed for clients requesting it
_____	_____	Container IDs in LIMS batch (Y__/N__) - If no, which samples

REPORTS:

_____	Photocopy of runlog
_____	Batch summary printed
_____	ICAL Summary printed
_____	BFB Reports
_____	Calibration/CCAL reports
_____	Reports printed for all samples (include library search if necessary)
_____	Logbook complete - All standard information included
_____	Tests scheduled in LIMS
_____	Data review signed
_____	Data reported signed
_____	Data approved signed

COMMENTS:

_____

_____

_____

_____

_____

_____

_____

Rev. 8/06

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SOP Change History Sheet

Section No.	Section	Reason for Change
5.2.5	Apparatus and Materials	Correction to current SOP
6.10 – 6.11	Reagents	Correction to current SOP
7.5.1	Instrument Calibration	Correction to current SOP
7.5.3.2	Instrument Calibration	DoD audit response
7.6.2	Instrument Calibrations	Correction to current SOP
8.6.4	Quality Control	Correction to current SOP
8.7.3	Quality Control	Correction to current SOP
8.5.4	Quality Control	Correction to current SOP
8.11	Quality Control	Correction to current SOP
10.3.1	Procedure	Correction to current SOP
10.3.2	Procedure	Correction to current SOP
10.3.3.4	Procedure	DoD audit response
10.3.7.7 & 9	Procedure	Correction to current SOP
10.3.7.9	Procedure	Correction to current SOP
10.3.3.12	Procedure	Correction to current SOP
10.3.4.1 & 3	Procedure	Correction to current SOP
A & B	Appendix	Correction to current SOP

Revision 8:03/06/2006 **(**Revisions made throughout to update Section references)

1.1	Scope and Application	Updated method revision
1.10	Scope and Application	Added project criteria requirements verbiage

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
4.2	Safety	Added MSDS availability
5.3, 5.5, 5.12	Apparatus and Materials	Added vendor information
5.9-5.11	Apparatus and Materials	Revised vendor information
5.14	Apparatus and Materials	Removed pipette reference
6.4	Reagents	Added vendor information
6.5	Reagents	Revised temperature range
6.6.2, 6.7.2.2, 6.8.4, 6.9.2.3, 6.10.3.3, 6.11.3.1, 6.11.3.3	Reagents	Added word “mixture”
6.7, 6.9	Reagents	Added abbreviation verbiage
6.8	Reagents	Added abbreviation verbiage and note
6.10.1	Reagents	Added calibration check standard verbiage, revised catalog #’s
6.10.3.1	Reagents	Revised preparation volumes
6.10.3.2, 6.11.3.2	Reagents	Revised preparation volumes, added standards
6.10.4.2	Reagents	Added volume amount of Acrolein
6.11.1	Reagents	Removed calibration check standard verbiage, revised catalog #’s
6.13	Reagents	Added DPD Free Chlorine to list
7.1	Instrument Calibration	Added instrument and recording location

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
7.4.2, 10.4.4.2, 10.4.7.2, 10.4.7.4, 11.1	Instrument Calibration Procedure Calculations	Updated reference from "Chemserver" to read "Target"
7.4.3, 7.6.1.1, 7.6.2.1, 8.14	Instrument Calibration Quality Control	Revised "USACE" to read "DoD"
7.5.4.2, 7.5.5.1, 8.7.3, 10.3.5.1, 10.4.5	Instrument Calibration Quality Control Procedure	Added DoD requirements
7.6.1	Instrument Calibration	Revised wording from "purge" to "inject"
7.6.1.1	Instrument Calibration	Added verbiage concerning scanning
8.3	Quality Control	Added verbiage about ongoing proficiency
8.6.2	Quality Control	Removed reference to third aliquot, added/revised volumes
8.7.1, 8.9.2	Quality Control	Added LCS and QC check sample preparation details
8.14	Quality Control	Added reference to SOP 99-MDL/reference method
9.3, 10.4.3	Sample Collection… Procedure	Replaced "AMS" with "Horizon", revised comment
10.3.3.5	Procedure	Removed references for performing dilutions in 50mL flasks
10.3.3.6	Procedure	Added reference to Class A flasks
10.3.3.10	Procedure	Removed reference to vendor
10.3.4	Procedure	Added mixing directions

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
10.4.4.3	Procedure	Added sample reruns to be rerun "at a dilution"
10.4.4.4	Procedure	Added reference to SOP 99-Integration
10.4.6.3.1, 10.4.6.3.2	Procedure	Revised spike sublist reference
10.4.7.5	Procedure	Added verbiage to include "guidelines"
12.3	Reporting Results	Added verbiage for reporting limits and J-values
12.6	Reporting Results	Added instructions to date and initial 1st and 2nd reviews
16	Troubleshooting	Added Section
A, B	Appendix	Substantial revisions throughout both tables

Revision 9: 08/17/2006

6.10-6.11	Reagents	Made current with lab practice as per internal audit findings
A, B, E	Appendix	Made current with lab practice as per internal audit findings

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Semi-volatile Organic Compounds by GC/MS

Document Control Number: _____

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Deborah Fatula, PhD, Date
GC/MS Supervisor/Validator

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1 Scope and Application

1.1 This standard operating procedure is adapted from "Test Methods for Evaluating Solid Waste Physical/Chemical Methods" (SW-846), Method 8000C, Revision 3, March 2003, and Method 8270D, Revision 4, January, 1998. The method detection limits (MDLs) can be found in the most current GC/MS MDL spreadsheet. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.2 Method 8270D is used to determine the concentration of semi-volatile organic compounds in extracts prepared from all types of solid waste matrices, soil, water and air sampling media. The following table from Method 8270D lists compounds that can be determined by this method.

		Appropriate Preparation Techniques[1]				
Compounds	**CAS No[a]**	**3510**	**3520**	**3540**	**3550**	**3580**
Acenaphthene	83-32-9	X	X	X	X	X
Acenaphthene-d_{10} (I.S.)		X	X	X	X	X
Acenaphthylene	208-96-8	X	X	X	X	X
Acetophenone	98-86-2	X	ND	ND	ND	X
2-Acetylaminofluorene*	53-96-3	X	ND	ND	ND	X
1-Acetyl-2-thiourea*	591-08-2	LR	ND	ND	ND	LR
Aldrin*	309-00-2	X	X	X	X	X
2-Aminoanthraquinone*	117-79-3	X	ND	ND	ND	X
Aminoazobenzene*	60-09-3	X	ND	ND	ND	X
4-Aminobiphenyl*	92-67-1	X	ND	ND	ND	X
3-Amino-9-ethylcar-bazole*	132-32-1	X	X	ND	ND	ND
Anilazine*	101-05-3	X	ND	ND	ND	X
Aniline	62-53-3	X	X	ND	X	X
o-Anisidine*	90-04-0	X	ND	ND	ND	X
Anthracene	120-12-7	X	X	X	X	X
Aramite*	140-57-8	HS(43)	ND	ND	ND	X
Aroclor-1016*	12674-11-2	X	X	X	X	X
Aroclor-1221*	11104-28-2	X	X	X	X	X
Aroclor-1232*	11141-16-5	X	X	X	X	X
Aroclor-1242*	53469-21-9	X	X	X	X	X
Aroclor-1248*	12672-29-6	X	X	X	X	X
Aroclor-1254*	11097-69-1	X	X	X	X	X
Aroclor-1260*	11096-82-5	X	X	X	X	X
Azinphos-methyl*	86-50-0	HS(62)	ND	ND	ND	X
Barban*	101-27-9	LR	ND	ND	ND	LR
Benzidine	92-87-5	CP	CP	CP	CP	CP

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Benzoic acid	65-85-0	X	X	ND	X	X
Benz(a)anthracene	56-55-3	X	X	X	X	X
Benzo(b)fluoranthene	205-99-2	X	X	X	X	X
Benzo(k)fluoranthene	207-08-9	X	X	X	X	X
Benzo(g,h,i)perylene	191-24-2	X	X	X	X	X
Benzo(a)pyrene	50-32-8	X	X	X	X	X
p-Benzoquinone*	106-51-4	OE	ND	ND	ND	X
Benzyl alcohol	100-51-6	X	X	ND	X	X
α-BHC*	319-84-6	X	X	X	X	X
β-BHC*	319-85-7	X	X	X	X	X
δ-BHC*	319-86-8	X	X	X	X	X
γ-BHC (Lindane)*	58-89-9	X	X	X	X	X
Bis(2-chloroethoxy) methane	111-91-1	X	X	X	X	X
Bis(2-chloroethyl) ether	111-44-4	X	X	X	X	X
Bis(2-chloroisopropyl) ether	108-60-1	X	X	X	X	X
Bis(2-ethylhexyl) phthalate	117-81-7	X	X	X	X	X
4-Bromophenyl phenyl ether	101-55-3	X	X	X	X	X
Bromoxynil*	1689-84-5	X	ND	ND	ND	X
Butyl benzyl phthalate	85-68-7	X	X	X	X	X
2-sec-Butyl-4,6-dinitro-phenol*	88-85-7	X	ND	ND	ND	X
Captafol*	2425-06-1	HS(55)	ND	ND	ND	X
Captan*	133-06-2	HS(40)	ND	ND	ND	X
Carbaryl*	63-25-2	X	ND	ND	ND	X
Carbofuran*	1563-66-2	X	ND	ND	ND	X
Carbophenothion*	786-19-6	X	ND	ND	ND	X
Chlordane*	57-74-9	X	X	X	X	X
Chlorfenvinphos*	470-90-6	X	ND	ND	ND	X
4-Chloroaniline	106-47-8	X	ND	ND	ND	X
Chlorobenzilate*	510-15-6	X	ND	ND	ND	X
5-Chloro-2-methyl-aniline*	95-79-4	X	ND	ND	ND	X
4-Chloro-3-methyl-phenol	59-50-7	X	X	X	X	X
3-(Chloromethyl) pyridine hydrochloride	6959-48-4	X	ND	ND	ND	X
1-Chloronaphthalene	90-13-1	X	X	X	X	X
2-Chloronaphthalene	91-58-7	X	X	X	X	X
2-Chlorophenol	95-57-8	X	X	X	X	X

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4-Chloro-1,2-phenylenediamine	95-83-0	X	X	ND	ND	ND
4-Chloro-1,3-phenylenediamine	5131-60-2	X	X	ND	ND	ND
4-Chlorophenyl phenyl ether	7005-72-3	X	X	X	X	X
Chrysene	218-01-9	X	X	X	X	X
Chrysene-d_{12} (I.S.)		X	X	X	X	X
Coumaphos	56-72-4	X	ND	ND	ND	X
p-Cresidine	120-71-8	X	ND	ND	ND	X
Crotoxyphos	7700-17-6	X	ND	ND	ND	X
2-Cyclohexyl-4,6-dinitrophenol	131-89-5	X	ND	ND	ND	LR
4,4'-DDD	72-54-8	X	X	X	X	X
4,4'-DDE	72-55-9	X	X	X	X	X
4,4'-DDT	50-29-3	X	X	X	X	X
Demeton-O	298-03-3	HS(68)	ND	ND	ND	X
Demeton-S	126-75-0	X	ND	ND	ND	X
Diallate (cis or trans)	2303-16-4	X	ND	ND	ND	X
2,4-Diaminotoluene	95-80-7	DC,OD(42)	ND	ND	ND	X
Dibenz(a,j)acridine	224-42-0	X	ND	ND	ND	X
Dibenz(a,h)anthracene	53-70-3	X	X	X	X	X
Dibenzofuran	132-64-9	X	X	ND	X	X
Dibenzo(a,e)pyrene	192-65-4	ND	ND	ND	ND	X
1,2-Dibromo-3-chloropropane	96-12-8	X	X	ND	ND	ND
Di-n-butyl phthalate	84-74-2	X	X	X	X	X
Dichlone	117-80-6	OE	ND	ND	ND	X
1,2-Dichlorobenzene	95-50-1	X	X	X	X	X
1,3-Dichlorobenzene	541-73-1	X	X	X	X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X	X
1,4-Dichlorobenzene-d_4 (I.S.)		X	X	X	X	X
3,3'-Dichlorobenzidine	91-94-1	X	X	X	X	X
2,4-Dichlorophenol	120-83-2	X	X	X	X	X
2,6-Dichlorophenol	87-65-0	X	ND	ND	ND	X
Dichlorovos	62-73-7	X	ND	ND	ND	X
Dicrotophos	141-66-2	X	ND	ND	ND	X
Dieldrin	60-57-1	X	X	X	X	X
Diethyl phthalate	84-66-2	X	X	X	X	X
Diethylstilbestrol	56-53-1	AW,OS(67)	ND	ND	ND	X
Diethyl sulfate	64-67-5	LR	ND	ND	ND	LR
Dihydrosaffrole	56312-13-1	ND	ND	ND	ND	ND

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Dimethoate	60-51-5	HE,HS(31)	ND	ND	ND	X
3,3'-Dimethoxy-benzidine	119-90-4	X	ND	ND	ND	LR
Dimethylaminoazo-benzene	60-11-7	X	ND	ND	ND	X
7,12-Dimethylbenz-(a)anthracene	57-97-6	CP(45)	ND	ND	ND	CP
3,3'-Dimethylbenzidine	119-93-7	X	ND	ND	ND	X
α,α-Dimethyl-phenethylamine	122-09-8	ND	ND	ND	ND	X
2,4-Dimethylphenol	105-67-9	X	X	X	X	X
Dimethyl phthalate	131-11-3	X	X	X	X	X
1,2-Dinitrobenzene	528-29-0	X	ND	ND	ND	X
1,3-Dinitrobenzene	99-65-0	X	ND	ND	ND	X
1,4-Dinitrobenzene	100-25-4	HE(14)	ND	ND	ND	X
4,6-Dinitro-2-methylphenol	534-52-1	X	X	X	X	X
2,4-Dinitrophenol	51-28-5	X	X	X	X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X	X	X	X	X
Dinocap	39300-45-3	CP,HS(28)	ND	ND	ND	CP
Dinoseb	88-85-7	X	ND	ND	ND	X
Dioxathion	78-34-2	ND	ND	ND	ND	ND
Diphenylamine	122-39-4	X	X	X	X	X
5,5-Diphenylhydantoin	57-41-0	X	ND	ND	ND	X
1,2-Diphenylhydrazine	122-66-7	X	X	X	X	X
Di-n-octyl phthalate	117-84-0	X	X	X	X	X
Disulfoton	298-04-4	X	ND	ND	ND	X
Endosulfan I	959-98-8	X	X	X	X	X
Endosulfan II	33213-65-9	X	X	X	X	X
Endosulfan sulfate	1031-07-8	X	X	X	X	X
Endrin	72-20-8	X	X	X	X	X
Endrin aldehyde	7421-93-4	X	X	X	X	X
Endrin ketone	53494-70-5	X	X	ND	X	X
EPN	2104-64-5	X	ND	ND	ND	X
Ethion	563-12-2	X	ND	ND	ND	X
Ethyl carbamate	51-79-6	DC(28)	ND	ND	ND	X
Ethyl methanesulfonate	62-50-0	X	ND	ND	ND	X
Ethyl parathion	56-38-2	X	X	ND	ND	ND
Famphur	52-85-7	X	ND	ND	ND	X
Fensulfothion	115-90-2	X	ND	ND	ND	X
Fenthion	55-38-9	X	ND	ND	ND	X
Fluchloralin	33245-39-5	X	ND	ND	ND	X

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Fluoranthene	206-44-0	X	X	X	X	X
Fluorene	86-73-7	X	X	X	X	X
2-Fluorobiphenyl (surr.)	321-60-8	X	X	X	X	X
2-Fluorophenol (surr.)	367-12-4	X	X	X	X	X
Heptachlor	76-44-8	X	X	X	X	X
Heptachlor epoxide	1024-57-3	X	X	X	X	X
Hexachlorobenzene	118-74-1	X	X	X	X	X
Hexachlorobutadiene	87-68-3	X	X	X	X	X
Hexachlorocyclo-pentadiene	77-47-4	X	X	X	X	X
Hexachloroethane	67-72-1	X	X	X	X	X
Hexachlorophene	70-30-4	AW,CP(62)	ND	ND	ND	CP
Hexachloropropene	1888-71-7	X	ND	ND	ND	X
Hexamethylphosphor-amide	680-31-9	X	ND	ND	ND	X
Hydroquinone	123-31-9	ND	ND	ND	ND	X
Indeno(1,2,3-cd) pyrene	193-39-5	X	X	X	X	X
Isodrin	465-73-6	X	ND	ND	ND	X
Isophorone	78-59-1	X	X	X	X	X
Isosafrole	120-58-1	DC(46)	ND	ND	ND	X
Kepone	143-50-0	X	ND	ND	ND	X
Leptophos	21609-90-5	X	ND	ND	ND	X
Malathion	121-75-5	HS(5)	ND	ND	ND	X
Maleic anhydride	108-31-6	HE	ND	ND	ND	X
Mestranol	72-33-3	X	ND	ND	ND	X
Methapyrilene	91-80-5	X	ND	ND	ND	X
Methoxychlor	72-43-5	X	ND	ND	ND	X
3-Methylcholanthrene	56-49-5	X	ND	ND	ND	X
4,4'-Methylenebis(2-chloroaniline)	101-14-4	OE,OS(0)	ND	ND	ND	LR
4,4'-Methylenebis(N,N-dimethylaniline)*	101-61-1	X	X	ND	ND	ND
Methyl methanesulfonate*	66-27-3	X	ND	ND	ND	X
2-Methylnaphthalene	91-57-6	X	X	ND	X	X
2-Methyl-5-nitroaniline	*99-55-8	X	X	ND	ND	ND
Methyl parathion*	298-00-0	X	ND	ND	ND	X
2-Methylphenol	95-48-7	X	ND	ND	ND	X
3-Methylphenol	108-39-4	X	ND	ND	ND	X
4-Methylphenol	106-44-5	X	ND	ND	ND	X
2-Methylpyridine*	109-06-8	X	X	ND	ND	ND
Mevinphos*	7786-34-7	X	ND	ND	ND	X
Mexacarbate*	3115-18-4	HE,HS(68)	ND	ND	ND	X
Mirex*	2385-85-5	X	ND	ND	ND	X

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Monocrotophos*	6923-22-4	HE	ND	ND	ND	X
Naled*	300-76-5	X	ND	ND	ND	X
Naphthalene*	91-20-3	X	X	X	X	X
Naphthalene-d_8 (I.S.)		X	X	X	X	X
1,4-Naphthoquinone*	130-15-4	X	ND	ND	ND	X
1-Naphthylamine*	134-32-7	OS(44)	ND	ND	ND	X
2-Naphthylamine	91-59-8	X	ND	ND	ND	X
Nicotine*	54-11-5	DE(67)	ND	ND	ND	X
5-Nitroacenaphthene*	602-87-9	X	ND	ND	ND	X
2-Nitroaniline	88-74-4	X	X	ND	X	X
3-Nitroaniline	99-09-2	X	X	ND	X	X
4-Nitroaniline	100-01-6	X	X	ND	X	X
5-Nitro-o-anisidine*	99-59-2	X	ND	ND	ND	X
Nitrobenzene	98-95-3	X	X	X	X	X
Nitrobenzene-d_5 (surr.)		X	X	X	X	X
4-Nitrobiphenyl*	92-93-3	X	ND	ND	ND	X
Nitrofen*	1836-75-5	X	ND	ND	ND	X
2-Phenol	88-75-5	X	X	X	X	X
4-Phenol	100-02-7	X	X	X	X	X
5-Nitro-o-toluidine*	99-55-8	X	ND	ND	ND	X
Nitroquinoline-1-oxide*	56-57-5	X	ND	ND	ND	X
N-Nitrosodibutylamine	924-16-3	X	ND	ND	ND	X
N-Nitrosodiethylamine	55-18-5	X	ND	ND	ND	X
N-Nitrosodimethyl-amine	62-75-9	X	X	X	X	X
N-Nitrosomethylethyl-amine*	10595-95-6	X	ND	ND	ND	X
N-Nitrosodiphenyl-amine	86-30-6	X	X	X	X	X
N-Nitrosodi-n-propylamine	621-64-7	X	X	X	X	X
N-Nitrosomorpholine*	59-89-2	ND	ND	ND	ND	X
N-Nitrosopiperidine*	100-75-4	X	ND	ND	ND	X
N-Nitrosopyrrolidine	930-55-2	X	ND	ND	ND	X
Octamethyl pyrophosphoramide*	152-16-9	LR	ND	ND	ND	LR
4,4'-Oxydianiline*	101-80-4	X	ND	ND	ND	X
Parathion*	56-38-2	X	ND	ND	ND	X
Pentachlorobenzene	608-39-5	X	ND	ND	ND	X
Pentachloronitro-benzene*	82-68-8	X	ND	ND	ND	X
Pentachlorophenol	87-86-5	X	X	X	X	X
Perylene-d_{12} (I.S.)		X	X	X	X	X
Phenacetin*	62-44-2	X	ND	ND	ND	X

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Phenanthrene	85-01-8	X	X	X	X	X
Phenanthrene-d_{10} (I.S.)		X	X	X	X	X
Phenobarbital*	50-06-6	X	ND	ND	ND	X
Phenol	108-95-2	DC(28)	X	X	X	X
Phenol-d_6 (surr.)		DC(28)	X	X	X	X
1,4-Phenylenediamine	*106-50-3	X	ND	ND	ND	X
Phorate*	298-02-2	X	ND	ND	ND	X
Phosalone*	2310-17-0	HD(65)	ND	ND	ND	X
Phosmet*	732-11-6	HS(15)	ND	ND	ND	X
Phosphamidon*	13171-21-6	HE(63)	ND	ND	ND	X
Phthalic anhydride*	85-44-9	CP,HE(1)	ND	ND	ND	CP
2-Picoline*	109-06-8	ND	ND	ND	ND	ND
Piperonyl sulfoxide*	120-62-7	X	ND	ND	ND	X
Pronamide*	23950-58-5	X	ND	ND	ND	X
Propylthiouracil*	51-52-5	LR	ND	ND	ND	LR
Pyrene	129-00-0	X	X	X	X	X
Pyridine	110-86-1	ND	ND	ND	ND	ND
Resorcinol	108-46-3	DC,OE(10)	ND	ND	ND	X
Safrole*	94-59-7	X	ND	ND	ND	X
Strychnine*	60-41-3	AW,OS(55)	ND	ND	ND	X
Sulfallate*	95-06-7	X	ND	ND	ND	X
Terbufos*	13071-79-9	X	ND	ND	ND	X
Terphenyl-d_{14} (surr.)	1718-51-0	X	X	ND	X	X
1,2,4,5-Tetrachloro-benzene	95-94-3	X	ND	ND	ND	X
2,3,4,6-Tetrachloro-phenol	58-90-2	X	ND	ND	ND	X
Tetrachlorvinphos*	961-11-5	X	ND	ND	ND	X
Tetraethyl dithiopyrophosphate*	3689-24-5	X	X	ND	ND	ND
Tetraethyl pyrophosphate*	107-49-3	X	ND	ND	ND	X
Thionazine*	297-97-2	X	ND	ND	ND	X
Thiophenol (Benzenethiol)*	108-98-5	X	ND	ND	ND	X
Toluene diisocyante*	584-84-9	HE(6)	ND	ND	ND	X
o-Toluidine*	95-53-4	X	ND	ND	ND	X
Toxaphene*	80001-35-2	X	X	X	X	X
2,4,6-Tribromophenol (surr.)		X	X	X	X	X
1,2,4-Trichlorobenzene	120-82-1	X	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	ND	X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X	X

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Trifluralin*	1582-09-8	X	ND	ND	ND	X
2,4,5-Trimethylaniline*	137-17-7	X	ND	ND	ND	X
Trimethyl phosphate*	512-56-1	HE(60)	ND	ND	ND	X
1,3,5-Trinitrobenzene*	99-35-4	X	ND	ND	ND	X
Tris(2,3-dibromopropyl) phosphate*	126-72-7	X	ND	ND	ND	LR
Tri-p-tolyl phosphate*	78-32-0	X	ND	ND	ND	X
O,O,O-Triethyl phosphorothioate*	126-68-1	X	ND	ND	ND	X

[1]Methods 3545 and T013 are also in use in this laboratory
*Compounds not currently in ALSI Calibration Standard
[a]Chemical Abstract Service Registry Number

AW = Adsorption to walls of glassware during extraction and storage
CP = Nonreproducible chromatographic performance
DC = Unfavorable distribution coefficient (number in parenthesis is percent recovery)
HE = Hydrolysis during extraction accelerated by acidic or basic conditions (number in parenthesis is percent recovery)
HS = Hydrolysis during storage (number in parenthesis is percent stability)
LR = Low response
ND = Not determined
OE = Oxidation during extraction accelerated by basic conditions (number in parenthesis is percent recovery)
OS = Oxidation during storage (number in parenthesis is percent stability)
X = Greater than 70 percent recovery by this technique

1.3 Method 8270D can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. See Appendix A for a list of the compounds for which ALSI regularly analyzes.

1.4 The following compounds may require special considerations when being determined by this method.

1.4.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

1.4.2 α-BHC, γ-BHC, Endosulfan I and II, Endrin are subject to decomposition in alkaline conditions of the extraction step from aqueous matrices. Neutral extraction shall be performed if these compounds are expected.

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1.4.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the GC, chemical reaction in acetone solution, and photochemical decomposition.

1.4.4 1,4-Dioxane and N-nitrosodimethylamine are difficult to separate from the solvent. The chromatographic conditions described provide adequate separation.

1.4.5 N-nitrosodiphenylamine decomposes in the GC inlet to Diphenylamine and cannot be distinguished from Diphenylamine. 1,2-Diphenylhydrazine decomposes in the GC inlet to Azobenzene and cannot be distinguished from Azobenzene.

1.4.6 Pentachlorophenol, 2,4-dinitophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic behavior, especially if the GC system is contaminated with high boiling material.

1.4.7 Pyridine may perform poorly at high GC injection port temperatures. Lowering the injection port temperature may reduce the amount of degradation, but may also cause losses of the late eluting compounds.

1.4.8 The base-neutral extraction may cause significantly reduced recovery of phenol, 2-methylphenol, and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

1.4.9 Resorcinol is very soluble in water and only slightly soluble in Methylene Chloride therefore exhibits very poor recoveries in 3510 extractions. If Resorcinol is to be analyzed by 8270 an alternative extraction procedure must be developed.

1.4.10 Anilines degrade slowly in acetone which is a solvent used in 3550 extractions, and therefore may exhibit low recoveries in 3550 extracts

1.4.11 Atrazine and Benzaldehyde degrade slowly in Methanol which is the solvent used for the Spiking solution, therefore may exhibit low recoveries

1.5 The estimated quantitation limit (EQL) of Method 8270D for determining an individual compound is approximately 1 mg/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for ground water samples. EQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer (GC/MS) and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.5.

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1.7 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.8 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Prior to using this method, the samples shall be prepared for chromatography using the appropriate sample preparation methods. This method describes chromatographic conditions that will allow for the separation of the compounds in the extract and for their qualitative and quantitative analysis by mass spectrometry**.** Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of two or three characteristic masses (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

3 Interferences

3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples and take corrective action to eliminate the problem. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profile. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.6.1

3.2 Glassware must be scrupulously clean. This is accomplished by following the glassware cleaning SOP.

3.3 The use of high purity reagents and solvents will help to minimize interference problems.

3.4 Matrix interferences may be caused by contaminants co-extracted from the sample. The extent of matrix interferences will vary depending on the origin of the sample. GPC cleanup may remove some of these interferences..

3.5 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, sample syringes are rinsed out between samples with solvent. Deactivated inlet liners may also help to reduce carryover. Analytical sequences shall be set up so that samples with suspected high levels are at the end of the sequence.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely

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defined; therefore, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. This includes use of proper protective wear and fume hoods.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available to all staff and are located in the QA office. Analysts shall consult the material safety data sheets (MSDS) for each chemical used in the analysis.

4.3 The following parameters covered in this method have been tentatively classified as known or suspected, human or mammalian carcinogens: Benzo(a)anthracene; benzidine; 3,3'-dichlorobenzidine; benzo(a)pyrene; α-BHC; β-BHC; δ-BHC, γ-BHC; dibenzo(a,h)anthracene; N-nitrosodimethylamine; 4,4'-DDT; and polychlorinated biphenyls (PCBs). Standards of these toxic compounds shall be prepared in a fume hood.

4.4 Since the chemical makeup of these samples is not known, analysts shall treat the samples with extreme caution. Precautionary steps would include PVC gloves, fully buttoned lab coat and safety glasses.

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5 Apparatus and Materials

5.1 Gas chromatograph/mass spectrometer/data system

5.1.1 Gas chromatograph: Hewlett Packard 5890 Series II Plus, with electronic pressure control, or equivalent.

5.1.2 Gas chromatographic column: 30 M x 0.25 mm ID ZB-5 with Guardian capillary column, 0.5 µm film thickness, manufactured by Phenomenex, Part #7HG-G002-17-GGC or equivalent

5.1.3 Mass spectrometer: Hewlett Packard Mass Selective Detector, Model 5971 or 5972, or equivalent.

5.1.4 Autosampler: Hewlett Packard 7673 Automatic sampler, or equivalent

5.1.5 Electron Multiplier: Model #4200-010200, purchased from CPI, or equivalent.

5.1.6 GC/MS Interface: Since a narrow-bore capillary column is used, the interface is capillary-direct into the mass spectrometer source.

5.1.7 Data system

5.1.7.1 Hewlett Packard MS DOS ChemStation, used for instrument tuning and data collection, or equivalent.

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5.1.7.2 Thermoquest Chemserver with Target4 and Exceed software from Thruput, or equivalent.

5.2 Microsyringes: Hamilton gas-tight, various sizes, purchased from VWR, or equivalent.

5.3 Disposable pipets: Pasteur, purchased from VWR, or equivalent.

5.4 Vials: 2 mL, 12 x 32 mm wide mouth amber crimp-top autosampler vial, purchased from National Scientific Company, catalog #C4011.2W, or equivalent.

5.5 Crimp-top caps: 11 mm Aluminum Seal with Teflon over Natural Red Rubber Septum, purchased from National Scientific Company, or equivalent

5.6 Low volume inserts for 2 mL autosampler vials: purchased from Agilent, part #5181-3377, or equivalent

5.7 Inlet liners: 4 mm Splitless Sleeve, purchased from Restek Corp. catalog #20774, or equivalent.

5.8 Fused silica wool: for inlet liner, purchased from Restek Corp. catalog #20790, or equivalent.5.9 5% dimethyldichlorosilane in toluene (Sylan-CT) for deactivating inlet liners, purchased from Supelco, catalog #33065U, or equivalent.

5.10 Septa: for inlet, Thermogreen LB-2, 11 mm, purchased from Supelco, catalog #2-0654, or equivalent.

5.11 Gold Plated Seals: purchased from Agilent, P/N 18740-20885, or equivalent.

5.12 Autosampler syringe, purchased from SGE, P/N 002814, or equivalent.

5.13 Vials: 8 mL amber Wheaton, purchased from VWR catalog #66011-926, or equivalent.

5.14 Caps: Wheaton, without liner, purchased from VWR catalog #66012-598, or equivalent.

5.15 Teflon faced liners: Wheaton, purchased from VWR catalog #66012-725, or equivalent.

6 Reagents

6.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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6.2 Compressed Helium gas: ultrahigh grade, purchased from MG Industries, or equivalent.

6.3 Methylene Chloride: Burdick & Jackson B& J Brand™ High Purity Solvent, purchased from Baxter catalog #300-4WRL, or equivalent, storage at room temperature, expiration as indicated by the manufacturer,

6.4 Primary Stock Solutions:
Primary stock solutions are purchased as pre-made standards. Certificates of Analysis for Primary Stock Solutions are in Appendix B. Expiration for unopened standards is as indicated by the manufacturer. When a new ampoule is opened, the solution is transferred to a properly labeled 2 mL amber vial for storage. Opened standards expire 6 months after the date opened or sooner if problems occur. They shall be stored as directed by the manufacturer, either in a refrigerator at 4C ± 2C or in a freezer at –10 to -20C or as indicated. Other equivalent standards may be used. These alternative solutions may be named differently and may contain the same analyses in different mixes, and concentrations may vary. The following primary stock solutions are used.

6.4.1 Decafluorotriphenylphosphine (DFTPP) Stock: EPA 8270 GC/MS Tuning Solution purchased from Ultra P/N GCM-150. This solution contains pentachlorophenol, decafluorotriphenylphosphine (DFTPP), benzidine and 4,4'-DDT at 1000 µg/mL in methylene chloride. See the Certificate of Analysis in Appendix B

6.4.2 Mega Mix: 1000 µg/mL in methylene chloride, purchased from Restek, part #31686, or equivalent. See the Certificate of Analysis in Appendix B for composition of Mega Mix. Note: Two different lots of Mega Mix are used. One lot is used for the preparation of Calibration Standards, the other lot is used as a Second Source Standard (Section 7.2.16)

6.4.3 Phenols Mix: 2000 µg/mL in methylene chloride, purchased from Restek, part #31494. See the Certificate of Analysis in Appendix B for composition of Phenols Mix.

6.4.4 Benzidines Mix: 2000 µg/mL in methylene chloride, purchased from Restek, part #31834. See the Certificate of Analysis in Appendix B for composition of Benzidines Mix..

6.4.5 Custom Acids Mixture: 2000 µg/mL in methylene chloride, purchased from Restek, part #559802. See the Certificate of Analysis in Appendix B for composition of Custom Acids Mixture. .

6.4.6 Custom Base/Neutral Mixture: 2000 µg/mL in methylene chloride, purchased from Restek, part #559801. See the Certificate of Analysis in Appendix B for composition of Custom Base/Neutral Mixture.

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6.4.7 Custom Resorcinol Mixture: 100000 µg/mL in methyl t-butyl ether, purchased from Restek, part #559803. See the Certificate of Analysis in Appendix B for composition of Custom Resorcinol Mixture

6.4.8 BNA Surrogates: 4000 µg/mL in methylene chloride, purchased from Supelco, part # 47960-U. See the Certificate of Analysis in Appendix B for composition of BNA Surrogates Mix.

6.4.9 Internal Standard Solution: 2000 µg/mL in methylene chloride, purchased from Restek, catalog #31206. See the Certificate of Analysis in Appendix B for composition of Internal Standard Solution. Internal Standard solution is stored at room temperature to prevent precipitation.

6.4.10 Custom BNA Mix : 100 µg/mL in methylene chloride, purchased from NSI, catalog #Q-4570. See the Certificate of Analysis in Appendix B for composition of Custom BNA Mix.

6.5 Decafluorotriphenylphosphine (DFTPP) Standard: EPA 8270 GC/MS Tuning Solution is diluted to 50 µg/mL for analysis. See Appendix C for preparation of 50 µg/mL DFTPP Standard. Store refrigerated at 4 ± 2°C for up to 6 months.

6.6 Secondary Stock Solution for Calibration Standards: See Appendix D for preparation of Secondary Stocks. The standard ID is recorded in the analysis log book and can be cross-referenced with the BNA Standards Logbook. Prepare as needed. Due to project requirements it may be necessary to add additional standards to the Secondary Stock Solution. The volume of methylene chloride added will be adjusted accordingly. This will be documented in the BNA standards logbook.

6.7 Calibration Standards: See Appendix D for preparation of Calibration Standards. Calibration standards are stored refrigerated at 4 ± 2°C. For analysis of DoD samples, calibration check standards are prepared weekly. For analysis of all other samples, calibration standards are prepared monthly or sooner as needed, depending on visual inspection of standards.

6.8 Second Source Standards are prepared as described in Appendix D.

6.9 Spike Solutions: The spike solutions are prepared by the Prep Department. See the Prep SOPs for more information including storage and expiration. BNA Spike Mix and Benzidine Spike Mix are used: as QC check samples to verify initial calibrations (Section 7.2); to spike samples for the Initial Demonstration of Capability (IDOC, Section 8.5); to spike Laboratory Control Samples (LCS, Section 8.6.2.2); and to spike Matrix Spike and Matrix Spike Duplicate samples (MS/MSD, Section 8.6.2.1). Surrogate Spike Solution is added to all samples.

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6.9.1 BNA Spike Mix: at 100 µg/mL in methanol, custom standard purchased from NSI, catalog #Q4114. See Appendix E for a list of compounds found in the mix.

6.9.2 Benzidine Spike Mix: contains benzidine and 3,3'-dichlorobenzidine at 2000 µg/mL, purchased from NSI, catalog #C-402P. This standard is diluted in methanol at a 1:20 ratio for use.

6.9.3 Surrogate Spike Solution: The surrogate spike solution is prepared by the Prep Department. See the Prep SOPs for more information including storage and expiration. Surrogates are added to all samples and all QC samples

7 Instrument Calibration

7.1 Instrument Performance Check

7.1.1 The GC/MS must be hardware tuned to meet tuning criteria before analysis can begin. In addition, it must be demonstrated that the GC/MS system is sufficiently inert. These checks are performed by injecting 1 µL of Decafluorotriphenylphosphine (DFTPP) Standard (Section 6.5) which contains 50 µg/mL DFTPP, pentachlorophenol, benzidine, and DDT.

7.1.2 Use the following guidelines to acquire the mass spectrum of DFTPP when performing the DFTPP tune check

7.1.2.1 When the analysis of non-DoD samples is to take place, DFTPP must meet tuning criteria by using a scan of the apex, a scan immediately preceding the apex, a scan immediately following the apex, the average of the three (3) scans, or the average of the entire peak. Background subtraction is optional.

7.1.2.2 When the analysis of DoD samples is to take place, DFTPP must meet tuning criteria by using the average of three (3) scans. The three (3) scans to be used are the apex of the peak, the scan immediately preceding the apex, and the scan immediately following the apex. Background subtraction is required. The background spectrum must be a single scan, be no more than 20 scans prior to the elution of DFTPP, and not be a part of the DFTPP peak

7.1.3 Acceptable tuning criteria must be demonstrated at the beginning of any 12-hour period in which analysis takes place. The start time of the 12-hour period begins at the time of injection of the Decafluorotriphenylphosphine (DFTPP) Standard (6.5). If the injection of a sample exceeds the 12-hour period it will require reanalysis.

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7.1.4 Use the DFTPP mass intensity criteria in Table 1 as tuning acceptance criteria. If criteria are not met, the GC/MS system must be re-tuned. Once tuned, DFTPP standard shall be re-injected and re-evaluated. Once a set of criteria has been chosen and used for an initial multi-point calibration, the same criteria must be used for all samples analyzed under that calibration, including CCAL standards, QC samples, and samples.

7.1.5 DDT criteria: The % breakdown of DDT to DDE and DDD shall not exceed 20% using the following calculation.

$$\%DDT\ breakdown = \frac{(Total\ area\ DDE\ and\ DDD)}{(Total\ area\ DDT + DDE + DDD)} \times 100\ \%$$

7.1.6 Benzidine criteria: The tailing factor for benzidine at 10% peak height shall not exceed 3. Benzidine shall be present at normal response. Note: in the event that only acid extractible compounds are to be analyzed, the benzidine criteria is not required.

7.1.7 Pentachlorophenol criteria: The tailing factor for pentachlorophenol at 10 % peak height shall not exceed 5. Pentachlorophenol shall be present at normal response. Note: in the event that only base-neutral compounds are to be analyzed, the pentachlorophenol criteria is not required.

7.2 Initial Calibration (ICAL: Initial Calibrations are performed as needed, when CCAL criteria cannot be met, or following major maintenance such as source cleaning or column replacement

7.2.1 Instrument Performance Check: The GC/MS system must meet the criteria for tuning and inertness detailed in Section 7.1. Analysis must not begin until these criteria are satisfied.

7.2.2 Internal Standards: The internal standard calibration procedure is used for quantitation. To use this approach, internal standards are selected that are similar in analytical behavior to the compounds of interest. The internal standards described in Section 6.4.7 permit most of the components of interest in a chromatogram to have retention times of 0.80 - 1.20 relative to one of the internal standards. The base peak ion from the specific internal standard is used as the primary ion for quantitation. If interferences are noted, the next most intense ion is used as the quantitation ion. The analyst must demonstrate that the measurement of the internal standards is not affected by method or matrix interferences. See Appendix A for the m/z's used for quantification .

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7.2.3 Data acquisition: Parameters for Data Acquisition are listed in Appendix F. This shows the Top-level parameters and Acquisition parameters. One (1) µL of each calibration standard is analyzed.

7.2.4 Calibration Range: Method 8270 requires a minimum of five (5) initial calibration standards. ALSI typically runs eight (8). The calibration range of the analysis is determined by the concentrations of the lowest and highest calibration standards included in the calibration. Sample results (on column) may not be reported at concentrations that are lower than the low standard or higher than the highest standard.

7.2.5 Response Factors: The software tabulates the area response of the primary characteristic ion against the concentration for each compound. A response factor (RF) for each compound relative to one of the internal standards is calculated as follows:

$$RF = (A_x\ C_{is}) / (A_{is}\ C_x)$$

where:

A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L)
C_x = Concentration of the compound being measured (µg/L)

7.2.6 System Performance Check Compounds (SPCC): The average RF is calculated and recorded for each compound using the RF values calculated for each compound from the initial calibration curve. Four compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average relative response factor. These compounds are n-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. These SPCCs typically have very low RFs (0.1 - 0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first compounds to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. Note: for DoD projects, the minimum acceptable average RF for all compounds of interest is 0.050.

7.2.7 Calibration Check Compounds (CCC): %RSD for each compound is calculated as follows:

$$\%RSD = SD/RF \times 100$$

where:

RSD = relative standard deviation.

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RF = mean of initial RFs for a compound.
SD = standard deviation of average RFs for a compound

The percent relative standard deviation (%RSD) for each individual Calibration Check Compound (CCC) must be less than 30%. (Note: It is acceptable to drop calibration levels at the ends of the calibration curves to meet these criteria. See Section 7.2.13 for guidelines and limitations.) If the %RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner, glass wool, gold seal, and/or capillary column, then repeat the calibration procedure beginning with Section 7.1. The Calibration Check Compounds are as follows:

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

7.2.8 Relative retention times (RRT): The relative retention times of each compound in each calibration run shall agree within 0.06 relative retention time units. Late eluting compounds usually have much better agreement.

7.2.9 Curve Types

7.2.9.1 Average Response Factor: If the %RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation. If an average response factor is used, it must contain at least 5 points.

7.2.9.2 Linear and quadratic fits: If the %RSD of any compound, including the CCC compounds, is greater than 15%, construct calibration curves of area ratio (A/A_{is}) versus concentration using first or higher order regression fit of the calibration points. The analyst shall select the regression order that introduces the least calibration error into the quantitation. The use of calibration curves is a recommended alternative to average response factor calibration, and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

7.2.9.2.1 Linear Fit: If a linear fit is used, it must contain at least 5 points. Its R^2 must be greater or equal to 0.99, which is

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equivalent to an R-value greater than or equal to 0.995)

7.2.9.2.2 Quadratic fit: If the quadratic fit is used, it must include at least 6 points. Its R^2 must be greater than or equal to 0.99.

7.2.9.2.3 Other fits: Our current version of Target software does not support higher than second order fits. Power fits and weighted average fits, which are available in the Target software, are not used.

7.2.10 Curve Origin: Target Software has 3 options for curve origins for linear and non-linear curve fits: "none", "include", and "force". Revision 3 of Method 8000 permits the options of "none" and "force", but does not allow "include" for curve fits. Curve fits will still need to meet the criteria given in Sections 7.2.9.2.1 and 7.2.9.2.2.

7.2.11 Reanalysis of ICAL Standards: If there is a problem with one of the calibration solutions it may be re-analyzed, or re-prepared and re-analyzed once. The re-analysis must take place within the same tune period and before any samples are analyzed. The results of the first analysis must be completely discarded.

7.2.12 Dropping levels in the middle of the ICAL: In some exceptional situations, it is acceptable to drop mid-level calibration levels from the initial calibration. There must be compelling technical justification for dropping the level. Some examples would be: a bad injection of a standard, a standard is made up incorrectly, omission of internal standards or secondary stock in a standard, a standard becomes concentrated or evaporates due to a cracked vial or a loose cap. If an average response factor or a linear fit is used, the calibration must contain at least 5 points. If the quadratic fit is used, it must include at least 6 points. If a calibration level in the middle of the initial calibration is dropped, the entire run must be dropped, and the reasons for the deletion must be noted on the run log and on the raw data. Note: it is always preferable to re-analyze a calibration level (Section 7.2.11) rather than to delete the level. It may also be preferable to re-calibrate and re-analyze samples rather than use an ICAL with a dropped level.

7.2.13 Dropping levels at the ends of the ICAL: Calibration levels may be dropped off the ends. In this case, an entire level can be dropped, or individual compounds can have high or low levels dropped, but the remaining levels must be a contiguous subset of the original. If an average response factor or a linear fit is used, the calibration must contain at least 5 points. If the quadratic fit is used, it must include at least 6 points. Dropping a low standard(s) for the initial calibration may raise the reporting limit for that compound. Dropping a high standard(s) will lower

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the calibration range for that compound. (i.e., if the 120 standard is dropped a sample that has a response of 100 would need to be rerun at a dilution because the highest standard would be 80). The calibration range of the analysis for each compound is determined by the concentrations of the lowest and highest calibration standards included in the calibration.

7.2.14 All analytes of interest in the initial calibration must either have acceptable average response factors or acceptable curve fits. If any compound does not, this initial calibration may not be used to report the compound(s) that fail the acceptance criteria. To report these compounds, the system must be recalibrated. Note it may be necessary to perform maintenance before re-calibration, which may include injection port maintenance, source cleaning, replacing the column.

7.2.15 Exceptions: If the CCCs are not analytes of interest they are not required to be included in the initial calibration quantitation Thus a calibration for only PAH compounds only needs to pass the 30% RSD for the CCCs contained in that list. If only acid compounds are analytes of interest, acid CCCs must have less than 30% RSD, etc. If there are no CCCs in the initial calibration, all analytes of interest must have less than 30% RSD. Sample analysis can only contain compounds quantitated in the calibration. ALL SPCCs must be quantitated in the calibration to verify inertness of the system, or all analytes of interest must meet the SPCC criteria. Note: for DoD projects, the minimum acceptable average RF for all compounds of interest is 0.050.

7.2.16 Second Source: The working calibration curve or RF is verified each time an ICAL is run prior to running any samples by the analysis of one or more "second source standards" There must be a second source standard for all the target analytes. An aliquot of each of the Second Source Standards (Section 6.8) is injected. Acceptance criteria are given in Appendix G. 90% of target analytes shall meet these criteria. Note: for DoD projects, acceptance criteria for all compounds of interest is 75-125% and all compounds must meet that criteria.

7.2.16.1 Corrective action: If criteria are not met, the following actions will be taken:

Review the initial calibration data to make sure there are no errors, prepare and analyze a fresh second source standard, re-prepare the initial calibration standards if necessary and re-calibrate.

7.2.16.2 Exceptions: If a failing analyte is not an analyte of interest for the associated samples, then the data is acceptable. If the failing analyte is biased high and is not detected in the associated samples, then the

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data is acceptable.

7.3 Daily GC/MS calibration: The instrument performance and calibration must be verified at the beginning of each analytical shift and every 12 hours during analysis.

7.3.1 Instrument Performance Check (DFTPP): The GC/MS system must meet the criteria for tuning and inertness in a DFTPP analysis as detailed in Section 7.1. prior to sample analysis and every 12 hours during analysis. If criteria are not met the system must be evaluated, and corrective action must be taken before analysis proceeds. See Section 7.3.4 for possible causes for failure and corrective action. Once corrective action has been completed, the instrument performance check must be repeated.

7.3.2 Calibration Check (CCAL): A mid-point calibration standard (CCAL) must be analyzed prior to sample analysis and every 12 hours during analysis. Compare the instrument response factor from the standards every 12 hours with the SPCC (Section 7.2.6) and CCC (Section 7.2.7) criteria.

7.3.2.1 Internal standards: The response (extracted ion chromatogram area) and retention time for each internal standard in the calibration check standard must be evaluated. The response for the internal standards must be within a factor of two (-50% to +100%) from the responses acquired in the last initial calibration, and the retention times for the internal standards must be within 30 seconds from the last initial calibration. If these criteria are not met the system must be evaluated, and corrective action must be taken before analysis proceeds. See Section 7.3.4 for possible causes for failure and corrective action. Once corrective action has been completed, the instrument performance check (DFTPP) and Calibration Check (CCAL) must be repeated.

7.3.2.2 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12-hour shift before analysis of samples. For each SPCC compound in the daily calibration, a minimum response factor of 0.050 must be obtained. This is the same check that is applied during the initial calibration. This check must be met before analysis begins. All SPCCs must be in the CCAL to verify inertness of the system or all calibrated compounds must meet the SPCC criteria. Note: for DoD projects, the minimum acceptable average RF for all compounds of interest is 0.050. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible causes for failure are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system

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7.3.2.3 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Section 7.2.7 are used to check the validity of the initial calibration. The %D is calculated using:

$$\%D = \frac{RF_x - RRF}{RRF} \times 100$$

where:
RF_x = Response factor for x in the 12-hour check standard.
RRF = Relative response factor in the initial 5-point calibration.

If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If these criteria are not met the system must be evaluated, and corrective action must be taken before analysis proceeds. See Section 7.3.4 for possible causes for failure and corrective action. Once corrective action has been completed, the instrument performance check (DFTPP) and Calibration Check (CCAL) must be repeated. If the source of the problem cannot be determined after corrective action has been taken, a new initial calibration must be generated. This criterion must be met before sample analysis begins.

7.3.3 Exceptions: If the SPCCs or CCCs are not analytes of concern, sample analysis can proceed if the analytes of interest have a percent difference or drift of less than 20% and meet the minimum response factor requirement. For example, if only acid compounds are analytes of interest, only the acid CCCs must have less than 20%D and must have response factors above 0.050 If only base/neutral compounds are analytes of interest, only the base/neutral CCCs must have less than 20%D and must have response factors above 0.050. If only PAH compounds are analytes of interest, only the PAH compounds must have less than 20%D and must have response factors above 0.050. Analysis may proceed if all analytes of interest meet the 20% criteria in the CCAL and have response factors above 0.050. Also if a parameter fails with a high bias, but that compound is not detected in the samples, then the sample may be reported with no qualifying comment.

7.3.4 Causes for failure and corrective actions: If the DFTPP or CCAL does not meet criteria, it may be repeated. If the repeat analysis fails, then the problem(s) within the GC/MS system must be located and corrected. Some possible problems are drift in relative abundances of high and/or low masses, standard mixture degradation, injection port inlet contamination, contamination of the front end of the analytical column, and active sites in the column or chromatographic system. Corrective actions would include adjusting tune parameters, injecting a fresh aliquot of standard, injection port maintenance: swabbing the interior of the injection port, replacing the liner and glass wool, gold seal, o-ring, septum; clipping the column. After corrective action has been taken, the DFTPP standard and CCAL must be re-injected . If the DFTPP and CCAL analyses meet criteria,

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then samples can be analyzed. If DFTPP passes but the CCAL fails, a new initial calibration is necessary.

7.3.5 Recalibration: If recalibration is indicated, it may be necessary to perform more heroic maintenance procedures to obtain an acceptable ICAL. For example, replacing the column, cleaning the source, replacing the electron multiplier.

8 Quality Control

8.1 QA Plan: All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Analysts: The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard shall be evaluated to determine if the chromatographic system is operating properly. Questions that shall be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector leaking, the injector septum needs replacing, etc. Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.3 Initial Calibration: There must be an acceptable initial instrument performance check and initial calibration of the GC/MS system as described in Sections 7.1 and 7.2.

8.4 Daily Calibration: The GC/MS system must meet the instrument performance specifications and the GC/MS system must meet the CCAL SPCC criteria and the CCC criteria each 12 hours as determined by analyzing a mid-point continuing calibration check sample (CCAL) as described in Section 7.3.

8.5 Initial Demonstration of Capability: To establish the ability to generate acceptable accuracy and precision, each analyst must perform the following operations before analyzing samples. Ongoing proficiency must also be established annually as specified in the QA Plan, Technical Training. This procedure is referred to as a precision and accuracy study (PNA) and shall be repeated when new staff members are trained or when significant changes in instrumentation or procedure are made, or when analytes are added. See Method 8000C, Section 9.4.10 for examples of changes that would require a PNA study.

8.5.1 Prepare and analyze four PNA samples: Spike four 1-L aliquots of reagent water with the Spike Mixes described in Section 6.9 to achieve a concentration of 100 µg/L by adding 1.0 mL of each spike mix to each aliquot of water. The four PNA samples are extracted and analyzed in exactly the same manner as actual samples as described in Section 10.

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8.5.2 Calculate the average recovery (x) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each analyte of interest using results from the four PNA samples.

8.5.3 For each analyte compare s and x with the laboratory's current acceptance criteria for precision and accuracy, respectively. (For new analytes, limits shall be chosen based on published limits, limits specified for PT samples, or limits for similar compounds. In the absence of these acceptance criteria, the laboratory shall use 70-130% as guidance.) If s and x for all analytes meet the acceptance criteria, the system performance is acceptable and analysis of actual sample can begin. If any individual s exceeds the precision limit or any individual x falls outside the range for accuracy, then the system performance is unacceptable for that analyte.

8.5.4 The large number of analytes presents a substantial probability that one or more will fail at least one of the acceptance criteria when all analytes of a given method are analyzed. When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed as follows. Locate and correct the source of the problem and, beginning with Section 8.5.1, repeat the test only for those analytes that failed to meet criteria. 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 for all compounds of interest beginning with Section 8.5.1.

8.6. Batch QC: Blanks, Laboratory Control Samples, Matrix Spikes, Matrix Spike Duplicates and Duplicates:

Each analytical batch (up to a maximum of 20 samples/batch) that is prepared will include a blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD) or sample duplicate (DUP). If more than twenty (20) samples are processed at one time, additional QC samples shall be processed with one (1) for every twenty samples. The method blank shall be carried through all stages of sample preparation and measurement. Blanks are used to demonstrate that interferences from the analytical system, glassware, and reagents are under control .LCS, MS, and MSD samples are used to assess accuracy. MS/MSD and sample/DUP pairs are used to assess precision. For soil and waste samples where detectable amounts of organics are present, duplicate samples may be appropriate in place of matrix spike duplicate samples. Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.6.1 Blanks: Before processing any samples, the analyst shall demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control.

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8.6.1.1 Frequency: A method blank must be prepared and analyzed for each analytical batch (up to a maximum of 20 samples/batch) to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Each time a set of twenty (20) samples or less samples is extracted or there is a change in reagents, a method blank shall be processed as a safeguard against laboratory contamination

8.6.1.2 Detections in Blanks: Results of all analytes of interest must be less than the reporting limit. For DoD samples, analytes must be less than 1/2 the reporting limit. If the method blank concentration is greater than or equal to the reporting limit (or ½ the reporting limit for DoD projects) and is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. (Note: If an analyte of interest is detected in the blank but is not detected in a sample, then the sample is not affected by the problem and reanalysis of that sample is not required.) If reanalysis is not possible or if the sample holding time has passed, contact the Customer Service Representative so that the client can be contacted in order to decide whether to re-extract (if there is enough remaining sample), raise reporting limits, recollect the sample, cancel the analysis or report with a qualifying comment. (Note: there are some situations where a blank detection may be of no concern to the client, for example, if the regulatory limit or action level is much higher than the levels detected in the blank and sample.)

8.6.1.3 Surrogate Recoveries in Blanks: If surrogate recoveries in the method blank fall outside calculated control limits, corrective action is required. If surrogate recoveries fall outside of acceptable limits in the blank but samples in the batch have acceptable recoveries for surrogates, no re-extractions for those samples are necessary. If any samples have surrogate recoveries outside of acceptable limits, those samples shall be re-extracted if possible, or reported with a qualifying comment. If the sample is to be re-extracted, report the sample results of the original analysis with a comment informing the client that the surrogate recoveries in the method blank and sample were unacceptable, and results are preliminary pending a confirmatory re-extraction and re-analysis. If the sample is not re-extracted, report the results of the surrogate recoveries in the method blank in a comment on the affected samples, and qualify the results as estimated.

Note: if surrogate recoveries are biased high, and there are no detections in the sample, data quality is not affected and reporting results with a comment is acceptable.

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8.6.2 Accuracy Assessment: The laboratory's ability to obtain accurate data is determined by the analysis of Laboratory Control Samples (LCS) and Matrix Spike (MS) and Matrix Spike Duplicates (MSD).

8.6.2.1 Matrix Spike and Matrix Spike Duplicates. Extract one sample aliquot to determine the background concentration, C_u, of each parameter. Spike a second sample aliquot with 1.0 mL of the Spike Mix and analyze it to determine the concentration after spiking, C_s of each parameter. The spiking concentration is C_n. This spiked sample aliquot is referred to as a matrix spike or MS and the spiked sample duplicate is referred to as a matrix spike duplicate or MSD. Calculate each percent recovery (%R) as:

$$\%R = 100 \times (C_s - C_u) / C_n \%$$

8.6.2.2 Laboratory Control Samples. Prepare the LCS by adding 1.0 mL of Spike Mix concentrate to an aliquot of a clean (control) matrix.. Extract and analyze the LCS to determine the concentration measured, C_m, of each parameter. The spiking concentration is C_n. Calculate each percent recovery (%R) as:

$$\%R = 100 \times C_m / C_n \%$$

8.6.2.3 Compare the percent recovery (%R) for each parameter with calculated control limits based on historical data as described in Section 8.8. If the MS or MSD recoveries fall outside of acceptable limits, the LCS shall be examined for those analytes that failed.

Note: If the compounds that fail are not analytes of interest for a sample, no further action is necessary for that sample.

8.6.2.4 If a compound fails recovery criteria in the MS but is acceptable in the LCS the failure may be due to sample matrix effects or may be due to a bad analysis or extraction. The sample, the extraction, and the analysis shall be evaluated to identify possible matrix effects that could cause the recovery issues. Examples would include: the presence of ash or charcoal in the sample; problems during the extraction procedure (emulsions, extracts which cannot be concentrated to 1.0 mL, etc); high levels of target or non-target analytes in the analysis or confirmed internal standard failures. If the LCS is acceptable and the specific matrix interference is identified, report with a qualifying statement. An example of such a comment would be: "One or more of the matrix spike compounds for the semi-volatile GC/MS analysis was recovered outside of the quality control limits due to sample matrix interferences. The LCS sample associated with this sample was within control limits." If no matrix effect can be

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identified, then the extracts shall be re-run to determine if the failure is due to a bad analysis. If re-analysis confirms the failure in the original extract, then the sample shall be re-extracted and re-analyzed. If the re-extract confirms the recovery failures, then the results from the original extract shall be reported with appropriate comments.

8.6.2.5 If an MS and MSD were prepared and a compound fails recovery criteria in both the MS and MSD but is acceptable in the LCS, then the failure is considered to be due to matrix effects. In this case the MSD can confirm the MS failure. The sample shall be reported with a qualifying comment. An example of such a comment would be: "One or more of the matrix spike compounds for the semi-volatile GC/MS analysis was recovered outside of the quality control limits due to sample matrix interferences. The LCS sample associated with this sample was within control limits."

8.6.2.6 If a compound fails recovery criteria in the MS or MSD and in the LCS, then the failure is considered to be due to method bias. The laboratory performance for that parameter is judged to be out of control, and the problem must be investigated and corrected.

8.6.2.7 If the MS or MSD and LCS fail with a high bias and there are samples in the batch that have no detections for that compound, those samples can be reported with no qualifying comment.

8.6.2.8 If the MS or MSD and LCS fail with a high bias and the samples have detections for that compound, the analytical results for that parameter in the associated samples are suspect and may not be useful for regulatory compliance purposes. Those samples shall be re-analyzed, or re-extracted and re-analyzed, or reported with a qualifying comment. An example of such a comment would be: "Naphthalene recoveries were above the laboratory established control limits in the GC/MS semi-volatile LCS and MS associated with this sample. The quantitation of naphthalene may be biased high in this analytical batch."

8.6.2.9 If the MS or MSD and LCS fail with a low bias then the analytical result for that parameter in the associated samples is suspect and may not be useful for regulatory compliance purposes. The samples shall be re-analyzed, or re-extracted and re-analyzed, or the samples shall be reported with a qualifying comment. An example of such a comment would be "Naphthalene recoveries were below the laboratory established control limits in the GC/MS semi-volatile LCS and MS associated with this sample. The quantitation of naphthalene may be biased low in this analytical batch.

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8.6.3 Precision Assessment Precision is assessed from the relative percent difference (RPD) of the concentrations measured for MS/MSD pairs or sample/DUP pairs. The formula for RPD is:

$$\%RPD = 100 *([R1 - R2]) / ((R1 + R2)/2)$$

Where:
R1 = Measured concentration of first sample aliquot or spike and
R2 = Measured concentration of second sample aliquot or spike duplicate

If an analyte of interest has a RPD exceeding the control limit, the sample shall reported with a qualifying comment, if required by the project. An example of such a comment would be: "One or more of the values for relative percent difference did not meet the quality control limits in the GC/MS semi-volatile matrix spike duplicate associated with this sample".

Note: If the compounds that fail are not requested analytes for a sample, no further action is necessary for that sample.

8.7 Sample QC: Surrogates: As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solution and calculate the percent recovery of each surrogate compound. Criteria for surrogate recovery are updated annually from control chart data as described in Section 8.8. Calculate each percent recovery (%R) as:

$$\%R = 100 (A) / T\%$$

where:

T = the known true value of the spike.

8.7.1 If a surrogate fails recovery criteria in a sample but is acceptable in the blank, then the failure may be due to matrix effects or may be due to a bad analysis or extraction. The sample, the extraction, and the analysis shall be evaluated to identify possible matrix effects that could cause the recovery issues. Examples of matrix effects would include: the presence of ash or charcoal in the sample; problems during the extraction procedure (emulsions, extracts which cannot be concentrated to 1.0 mL, etc); high levels of target or non-target analytes in the analysis or confirmed internal standard failures. If a matrix effect can be identified, the sample shall be reported with a qualifying comment. An example of such a comment would be: "Matrix interferences were present in this sample which caused one or more surrogates to be recovered outside of the laboratory established control limits for the GC/MS semi-volatile analysis." If no matrix effect can be identified, then the sample shall be re-analyzed to determine if the failure is due to a bad analysis. If re-analysis confirms the failure in the original extract, then the sample shall be re-extracted and re-analyzed. If the re-extract

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confirms the recovery failures, then the results from the original extract shall be reported with appropriate comments. If the re-extract has acceptable recoveries and was re-extracted during its hold time, report the results from the re-extract. If the sample cannot be re-extracted, the client shall be contacted to determine if the sample will be re-sampled, cancelled, reported with a comment, etc.

8.7.2 If there is a MS or duplicate for the sample that exhibits similar surrogate recoveries, or if there is historical data of surrogate failures, then those can be used to confirm surrogate failures.

8.7.3 If a surrogate fails recovery criteria in a sample and in the blank, then the failure is considered to be due to method bias. The laboratory performance for that analysis is judged to be out of control, and the problem must be investigated and corrected. The analytical result for that sample is suspect and may not be useful for regulatory compliance purposes.

8.7.4 If a surrogate fails in a sample and in the blank with a high bias and the sample has no detections for analytes of interest, the sample can be reported with no qualifying comment.

8.7.5 If a surrogate fails in a sample and in the blank with a high bias and the sample has detections for analytes of interest, the affected sample and blank shall be re-analyzed to determine if the failures are due to a bad analysis. If re-analysis confirms the failures in the original extract, then the sample shall be re-extracted and re-analyzed. If the sample cannot be re-extracted, the client shall be contacted to determine if the sample will be re-sampled, cancelled, reported with a comment, etc. An example of such a comment would be: "Nitrobenzene-d5 recoveries were above the laboratory established control limits in this sample and in the GC/MS semi-volatile blank associated with this sample. The quantitation of this surrogate may be biased high in this analytical batch."

8.7.6 If a surrogate in a sample and the blank fail with a low bias, the affected sample and blank shall be re-analyzed to determine if the failures are due to a bad analysis. If re-analysis confirms the failures in the original extract, then the sample shall be re-extracted and re-analyzed. If the sample cannot be re-extracted, the client shall be contacted to determine if the sample will be re-sampled, cancelled, reported with a comment. An example of such a comment would be: "Nitrobenzene-d5 recoveries were below the laboratory established control limits in this sample and in the GC/MS semi-volatile blank associated with this sample. The quantitation of this surrogate may be biased low in this analytical batch."

8.8 Control Charts and Control Limits. As part of the QC program, method accuracy and precision for environmental samples must be assessed and records must be maintained. Control charts are maintained by the QC department from data uploaded to the LIMS: see the QC plan and SOP on QC limits for more information. Control limits for LCS and

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MS/MSD samples are developed from LCS data. Control limits for surrogate recoveries are developed from sample data. Control limits for relative percent difference are developed from MS/MSD sample data. Control limits are updated annually from control chart data. At least 20 measurements are required to obtain valid control limits. In the event that there are not sufficient data points available, control limits will be set by referring to available information in the applicable methods and will be documented in a corrective action form.

8.8.1 Criteria for recovery are updated annually from control chart data, using recoveries for at least 20 samples. Average percent recoveries (P) and standard deviations of the percent recovery (Sp) are calculated. Upper control limits (UCL) and lower control limits (LCL) are calculated:

UCL = P + 3 (Sp)
LCL = P – 3 (Sp)

8.8.2 Criteria for relative percent difference are updated annually from control chart data using relative percent differences for at least 20 MS/MSD pairs. Average relative percent differences (ARPD) and standard deviations of the relative percent differences (Sp) are calculated; control limits (CL) are calculated:

CL = ARPD + 3 (Sp)

8.8.3 For DoD projects the control limits will be compared to limits in the DoD QSM. In-house control limits will be used unless the in-house limits exceed the DoD limits.

8.9 Internal Standards in sample analyses

8.9.1 Extracts from environmental samples frequently have high levels of organic acids and other contaminants, resulting in erratic responses for internal standards. Internal standard areas will be compared to the daily calibration check standard or the 50 mg/L Level 5 standard if an initial calibration was analyzed. If internal standard areas observed in a sample analysis are in the range of -50 to + 100 % of that observed in the standard, sample results may be reported.

8.9.2 If internal standard areas observed in a sample analysis are not in the range -50 to + 100 % of that observed in the standard, the sample will be re-run undiluted and/or at a dilution. If internal standard areas are still outside the -50 to +100 % range, then the samples will be reported with a comment. An example of such a comment would be: "One or more of the GCMS semi-volatile internal standards were recovered at <50%. The sample was reanalyzed with similar results indicating a sample matrix interference."

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8.9.3 Internal standard retention times shall be within 0.5 min of the retention times observed in the daily calibration check.

8.9.4 If a particular sample has a limited number of analytes of interest, and a failing internal standard is not used for quantitation of the analytes of interest, then sample results may be reported.

8.10 MDL Studies. MDL studies must be performed according to SOP 99-MDL .

9 Sample Collection, Preservation and Handling

9.1 Sample Collection

9.1.1 Containers used to collect samples must be specialty cleaned 1-L glass bottles for water and specialty cleaned 4 or 8 oz glass jars for soil samples. The sample containers shall have screw caps with Teflon lined caps. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. For T0-13, air samples are collected on polyurethane foam cartridges or sorbent tubes. See the Prep SOPs for more information.

9.1.2 Conventional sampling practices shall be followed, except that the bottle must not be pre-washed with sample before collection. Composite samples shall be collected in refrigerated glass containers. Automatic sampling equipment must be as free as possible of Tygon and other potential sources of contamination.

9.1.3 Sample containers shall be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples shall not be collected or stored in the presence of exhaust fumes.

9.1.4 A minimum of 1000 mL is recommended for aqueous samples. A minimum of 30 g is recommended for 3550 soil samples and 15.0 g for 3545 soil extractions. Extractions performed with reduced sample amounts may result in elevated PQLs. The impact on PQLs and other data quality objectives shall be evaluated by the section leader when amounts other than those recommended are used.

9.2 Sample Preservation

9.2.1 The samples must be iced or refrigerated at 4 ± 2°C from the time of collection until extraction.

9.2.2 If residual chlorine is present in water samples, add 80 mg of sodium thiosulfate per each liter of water.

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9.3 Sample Handling

9.3.1 All water samples must be extracted within 7 days and completely analyzed within 40 days of extraction. All soil samples must be extracted within 14 days and completely analyzed within 40 days of extraction All samples not analyzed within this time frame must be discarded and resampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.

10 Procedure

10.1 Sample preparation: Samples must be prepared by one of the following methods prior to GC/MS analysis. See the Prep department SOPs.

Matrix	Methods
Water	3510, 3520
Soil/sediment	3540, 3545, 3550
Waste	3540, 3550, 3580
Air	TO-13A (modified)

10.2 Calibration: There must be an acceptable initial instrument performance check and initial calibration (See Sections 7.1 and 7.2). Daily instrument performance check criteria (Sections 7.1, and 7.3.1, and Table 1) and GC/MS calibration verification criteria (See Section 7.3.2) must be met before analyzing samples.

10.3 GC/MS analysis:

10.3.1 Transfer 200 µL of extract to a vial with a low volume insert and add 4 µL internal standard solution.

10.3.2 Analyze the extract using the method listed in Appendix F. A 1 µL injection is used.

10.3.3 If the response for any compound exceeds the calibration range of the GC/MS system, dilute the extract and reanalyze. Internal standard must be added to the diluted extract at 40 mg/L of each internal standard in the diluted extract.

10.3.4 Store the remainder of the extracts at 4 ± 2°C, protected from light in crimp-top amber vials equipped with un-pierced Teflon-lined septa.

10.3.5 Sample extracts must inject within 12 hours after the injection of the DFTPP.

10.3.6 Injection logs are printed from the Chemstation after the sequence has completed. See Appendix H for an example.

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10.4 Qualitative Identification

10.4.1 Obtain EICPs for the primary m/z (Appendix A) and one or two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification.

10.4.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

10.4.1.2 The relative retention time (RRT) of the sample component must fall within ± 0.06 of the relative retention time of the authentic standard component.

RRT = RT of analyte/RT of associated internal standard

10.4.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum.

10.4.2 Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4.3 Library search analysis: When requested, library search analysis will be used to identify peaks not identified as compounds in the calibration (non-target compounds).

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10.4.3.1 The selection and quantitation of non-target peaks is performed automatically by the Target software. Note: The sample shall be processed for client specific compounds only. Otherwise, the Target software will not pick any other target compounds as library peaks. If a sublist containing client specific analytes is not used, the analyst must add, to the library search form, any compounds that were detected in the analysis that the client does not want reported. Also, if any changes have been made to the sample such as deleting or integrating peaks, the library search must be redone by selecting “process unknowns” in Target.

10.4.3.2 If the software selects any internal standard, surrogate, or target compounds, delete them in Target. The solvent peak (the first large peak on the chromatogram) shall be deleted also. Also, early eluters with mass 40 can be deleted.

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10.4.3.3 The Target software will perform a library search on and estimate the concentration of the 20 largest non-target peaks.

10.4.3.4 Only after visual comparison of sample spectra with the nearest library searches shall a tentative identification be assigned to the peak. The analyst will need to search each non-target peak on the Target system to view the library searches. Consider the following sets of guidelines before making a tentative identification.

10.4.3.5 Guidelines for making tentative identification of non-target compounds. In other words, making a specific identification of non-target compounds, i.e., limonene, pentacosane. These guidelines are from a contract laboratory program statement of work.

10.4.3.5.1 Major ions (ions greater than 10% of the most abundant ion) in the reference spectrum shall be present in the sample spectrum.

10.4.3.5.2 The relative intensities of the major ions shall agree within plus or minus 20%. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

10.4.3.5.3 Molecular ions present in the reference spectrum shall be present in the sample spectrum.

10.4.3.5.4 Ions present in the reference spectrum but not in the sample spectrum shall be reviewed for possible subtraction from the sample spectrum because of background contamination or co eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.3.5.5 If in the analyst's technical judgment, no valid identification is possible, the compound shall be reported as unknown. If possible give an additional classification to the compound (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound, etc.). If the probable molecular weights can be determined, include them.

10.4.3.6 Guidelines for making tentative ID based on match quality.

10.4.3.6.1 If a non-target compound is present in the calibration mixes it can be identified as that compound no matter how good or bad the match quality is as long as the criteria for identifying the spectra of target compounds are met for those compounds, i.e., hexane, benzyl chloride, 1,2,4-trimethylbenzene, etc.

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10.4.3.6.2 If a tentatively identified compound has a match of greater than 90% and the next closest match is greater than 30 % less, tentatively identify the peak as that compound.

10.4.3.6.3 If the match quality of 2 or more isomers are very close together and greater than 70% with no other unrelated compounds within 10 %, identify that peak as _____ isomer. Be as specific as possible.

10.4.3.6.4 Identify classes of compounds if all the compounds above 50 % match belong to the same class, or if the 2 or 3 closest matches belong to one chemical class and the next matches have significantly different match quality.

10.4.3.6.5 Use the analyst's experience when possible. Also, be consistent throughout a group of samples, referring back to retention times as a guide.

10.5 Quantitation

10.5.1 Integration: Integration parameters shall be optimized for each compound. SOP 99-Integration shall be followed for any manual integrations.

10.5.2 Quantitation of target compounds: Concentrations for target compounds are quantitated by Target using the internal standard method. The nearest internal standard free of interferences shall be used. Extracted ion chromatogram area counts are used for both the target compound and the internal standard. Response factors or calibration curves from the most recent initial calibration are used to calculate ng/injection. Values for volume or weight of sample extracted, final volume of extract, and dilution factor are entered into the Target software for each sample. Sample concentrations are calculated by the Target software.

10.5.3 Calibration range: If the response for any compound exceeds the calibration range of the GC/MS system, dilute the extract and reanalyze. Internal standard must be added to the diluted extract at 40 mg/L of each internal standard in the diluted extract. The results for both analyses shall be reported: Compounds that did not exceed the calibration range will be reported from the undiluted extract. Note: If the over range analytes are not analytes of interest for the sample, then dilution is not required.

10.5.4 Data Quality: QC results will be compared to the control limits generated for the extraction procedure to determine if the data is of acceptable quality for reporting. See Section 8.

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10.5.5 Quantitation of library search compounds: Estimated concentrations for library search compounds are quantitated by Target using the internal standard method. The nearest internal standard free of interferences shall be used. Total area counts from the total ion chromatogram are used for both the non-target compound and the internal standard to calculate ng/injection. A RRF of 1 is assumed. Values for volume or weight of sample extracted, final volume of extract, and dilution factor are entered into the Target software for each sample. Estimated sample concentrations are calculated by the Target software.

11 Calculations

11.1 All calculations are performed by the ChemServer Target4 software.

11.2 When a parameter has been identified, the quantitation of that parameter will be based on the integrated abundance from the EICP of the primary characteristic m/z given in Appendix A. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

12 Reporting Results

12.1 All raw data from Target used for reporting results must be printed, dated and initialed by the qualified laboratory personnel performing first and second review. Any errors must be marked through with a single line with the analyst's initial, the date, and the correction.

12.2 Target results are uploaded to Horizon. Results from soil samples are uploaded without the moisture correction. The moisture correction is performed by Horizon. All results are uploaded including values below the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable): Horizon will automatically report the appropriate detection limit.

12.3 Target results are uploaded to Horizon with 6 significant figures. Horizon LIMS will automatically perform rounding appropriate to the method. Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.4 If a sample must be diluted due to matrix interferences, the detection limit of the sample will be raised to reflect the dilution performed and a comment must be added in Horizon to explain the raised detection limit to the client.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

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14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

15.2 “Target compounds” are the compounds that are in the calibration mix and the Target method.

15.3 “Analytes of interest” are the compounds in the calibration mix and Target method that have been requested for a particular sample or group of samples.

15.4 “Non-target compounds” are peaks in the analytical results that are not identified as calibrated compounds. Non-target compounds include tentatively identified compounds, which have been identified by library search, and unknowns, which cannot be identified by library search.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this method.

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TABLE 1

DFTPP KEY M/Z ABUNDANCE CRITERIA

MASS	M/Z ABUNDANCE CRITERIA
51	30 TO 60% OF MASS 198
68	<2% OF MASS 69
70	<2% OF MASS 69
127	40 TO 60% OF MASS 198
197	<1% OF MASS 198
198	BASE PEAK, 100% RELATIVE ABUNDANCE
199	5 TO 9% OF MASS 198

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275	10 TO 30% OF MASS 198
365	>1% OF MASS 198
441	PRESENT BUT LESS THAN MASS 443
442	>40% OF MASS 198
443	17 TO 23% OF MASS 442

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APPENDIX A
List of Compounds

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COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m
Quant Method : ISTD Target Version : 4.12
Last Update : 07-Mar-2006 16:11 Number of Cpnds : 112
Data Type : MS DATA

Global Integrator : HP RTE

Chromat Events	Values
Initial:Thresh Units	1.000000
Initial:Area Thresh	2.000000
Initial:Max Peaks	150.000000
Initial:Bunching	1.000000
Initial:Smoothing	0.000000
Initial:Start Thresh	0.112000
Initial:Stop Thresh	0.000000
Initial:Baseline Reset	5.000000
Initial:Set Valley	100.000000

	Compound	RT	RT Window	RF	Mass
	1 1,4-Dioxane	4.063	1.096-7.030	7.51e-001	88.00
		4.063	1.096-7.030		58.00
	2 Pyridine	5.250	0.572-9.927	8.01e-001	79.00
		5.260	0.582-9.938		52.00
		5.250	0.572-9.927		51.00
	3 N-Nitrosodimethylamine	5.291	2.324-8.258	9.35e-001	74.00
		5.291	2.324-8.258		42.00
		5.291	2.324-8.258		43.00
$	4 2-Fluorophenol	9.116	6.149-12.083	1.49e+000	112.00
		9.116	6.149-12.083		64.00
	5 N-Nitrosodiethylamine	9.607	6.640-12.574	7.37e-001	102.00
		9.607	6.640-12.574		42.00
		9.607	6.640-12.574		56.00
	6 Benzaldehyde	10.906	7.939-13.873	4.53e-001	77.00
		10.906	7.939-13.873		105.00
		10.906	7.939-13.873		106.00
$	7 Phenol-d5	11.244	8.277-14.211	1.28e+000	99.00
		11.244	8.277-14.211		71.00
		11.244	8.277-14.211		42.00
	8 Phenol	11.285	8.318-14.252	1.33e+000	94.00
		11.285	8.318-14.252		65.00
		11.285	8.318-14.252		66.00
	9 Aniline	11.244	8.277-14.211	1.45e+000	93.00
		11.244	8.277-14.211		66.00
		11.244	8.277-14.211		65.00
	10 Bis(2-chloroethyl)ether	11.387	8.420-14.354	1.05e+000	93.00
		11.387	8.420-14.354		63.00
		11.387	8.420-14.354		95.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

	Compound	RT	RT Window	RF	Mass
	11 2-Chlorophenol	11.500	8.533-14.467	1.27e+000	128.00
		11.500	8.533-14.467		130.00
		11.489	8.522-14.456		64.00
	12 n-Decane	11.592	8.625-14.559	1.00e+000	57.00
		11.592	8.625-14.559		43.00
		11.592	8.625-14.559		71.00
	13 1,3-Dichlorobenzene	11.786	8.819-14.753	1.57e+000	146.00
		11.786	8.819-14.753		148.00
		11.786	8.819-14.753		111.00
*	14 1,4-Dichlorobenzene-d4	11.868	8.901-14.835		152.00
		11.868	8.901-14.835		150.00
		11.868	8.901-14.835		115.00
	15 1,4-Dichlorobenzene	11.909	8.942-14.876	1.54e+000	146.00
		11.909	8.942-14.876		148.00
		11.909	8.942-14.876		111.00
	16 Benzyl Alcohol	12.257	9.290-15.224	6.80e-001	108.00
		12.257	9.290-15.224		79.00
		12.257	9.290-15.224		77.00
	17 1,2-Dichlorobenzene	12.318	9.351-15.285	1.43e+000	146.00
		12.318	9.351-15.285		148.00
		12.318	9.351-15.285		111.00
	18 2-Methylphenol	12.574	9.607-15.541	9.01e-001	107.00
		12.574	9.607-15.541		108.00
		12.574	9.607-15.541		90.00
	19 bis(2-Chloroisopropyl)e	12.594	9.627-15.561	1.22e+000	45.00
		12.594	9.627-15.561		121.00
		12.594	9.627-15.561		41.00
	20 Acetophenone	12.840	9.873-15.807	1.76e+000	105.00
		12.840	9.873-15.807		77.00
		12.840	9.873-15.807		51.00
	21 3 & 4-Methylphenols	12.901	9.934-15.868	1.24e+000	107.00
		12.901	9.934-15.868		108.00
		12.901	9.934-15.868		77.00
	22 N-Nitrosodi-n-propylami	12.911	9.944-15.878	8.77e-001	70.00
		12.911	9.944-15.878		42.00
		12.911	9.944-15.878		101.00
	23 N-Nitrosopyrrolidine	12.840	9.873-15.807	5.62e-001	100.00
		12.840	9.873-15.807		68.00
		12.840	9.873-15.807		41.00
	24 Hexachloroethane	13.003	10.036-15.970	7.52e-001	117.00
		13.003	10.036-15.970		200.60
		13.003	10.036-15.970		198.60
$	25 Nitrobenzene-d5	13.167	9.486-16.847	4.19e-001	82.00
		13.167	9.486-16.847		54.00
		13.167	9.486-16.847		128.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
26 Nitrobenzene	13.208	9.527-16.888	4.00e-001	77.00
	13.208	9.527-16.888		123.00
	13.208	9.527-16.888		65.00
27 Isophorone	13.740	10.059-17.420	6.34e-001	82.00
	13.740	10.059-17.420		95.00
	13.750	10.069-17.430		138.00
28 2-Nitrophenol	13.924	10.243-17.604	2.31e-001	139.00
	13.924	10.243-17.604		65.00
	13.924	10.243-17.604		109.00
29 2,4-Dimethylphenol	14.057	10.376-17.737	3.10e-001	122.00
	14.057	10.376-17.737		107.00
	14.057	10.376-17.737		121.00
30 Bis(2-chloroethoxy)meth	14.251	10.571-17.932	3.63e-001	93.00
	14.251	10.571-17.932		95.00
	14.251	10.571-17.932		123.00
31 Benzoic Acid	14.384	10.704-18.065	1.66e-001	105.00
	14.384	10.704-18.065		122.00
	14.384	10.704-18.065		77.00
32 2,4-Dichlorophenol	14.466	10.785-18.146	3.51e-001	162.00
	14.466	10.785-18.146		164.00
	14.466	10.785-18.146		98.00
33 1,2,4-Trichlorobenzene	14.640	10.959-18.320	4.22e-001	180.00
	14.640	10.959-18.320		182.00
	14.640	10.959-18.320		145.00
* 34 Naphthalene-d8	14.722	11.041-18.402		136.00
	14.722	11.041-18.402		68.00
35 a-Terpineol	14.793	11.113-18.474	2.76e-001	59.00
	14.793	11.113-18.474		93.00
	14.793	11.113-18.474		121.00
36 Naphthalene	14.773	11.092-18.453	9.88e-001	128.00
	14.773	11.092-18.453		129.00
	14.773	11.092-18.453		127.00
37 4-Chloroaniline	14.957	11.276-18.637	4.28e-001	127.00
	14.957	11.276-18.637		65.00
	14.957	11.276-18.637		129.00
38 2,6-Dichlorophenol	14.978	11.297-18.658	3.40e-001	162.00
	14.978	11.297-18.658		164.00
	14.978	11.297-18.658		98.00
39 Hexachlorobutadiene	15.192	11.512-18.873	2.80e-001	224.60
	15.192	11.512-18.873		222.60
	15.192	11.512-18.873		226.60
40 N-Nitrosodi-n-butylamin	15.775	12.095-19.456	2.26e-001	84.00
	15.775	12.095-19.456		57.00
	15.775	12.095-19.456		116.00
41 Resorcinol	15.939	12.258-19.619	3.75e-001	110.00
	15.939	12.258-19.619		81.00
	15.939	12.258-19.619		82.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
42 Caprolactam	15.765	12.085-19.446	1.06e-001	113.00
	15.775	12.095-19.456		55.00
	15.775	12.095-19.456		56.00
43 4-Chloro-3-Methylphenol	16.092	12.412-19.773	3.63e-001	107.00
	16.103	12.422-19.783		142.00
	16.092	12.412-19.773		77.00
44 2-Methylnaphthalene	16.328	12.647-20.008	6.56e-001	142.00
	16.328	12.647-20.008		141.00
	16.328	12.647-20.008		115.00
45 1-Methylnaphthalene	16.563	12.882-20.243	6.28e-001	142.00
	16.563	12.882-20.243		141.00
	16.563	12.882-20.243		115.00
46 1,2,4,5-Tetrachlorobenz	16.788	12.110-21.466	6.82e-001	215.60
	16.788	12.110-21.466		213.60
	16.788	12.110-21.466		74.00
47 Hexachlorocyclopentadie	16.839	12.161-21.517	2.14e+000	236.60
	16.839	12.161-21.517		234.60
48 2,4,6-Trichlorophenol	17.044	12.366-21.721	4.76e-001	196.00
	17.044	12.366-21.721		198.00
	17.044	12.366-21.721		97.00
49 2,3-Dichloroaniline	17.044	12.366-21.721	7.16e-001	161.00
	17.044	12.366-21.721		163.00
	17.044	12.366-21.721		90.00
50 2,4,5-Trichlorophenol	17.136	12.458-21.813	4.93e-001	196.00
	17.136	12.458-21.813		198.00
	17.136	12.458-21.813		97.00
$ 51 2-Fluorobiphenyl	17.207	12.530-21.885	1.47e+000	172.00
	17.207	12.530-21.885		171.00
52 1,1'-Biphenyl	17.402	12.724-22.079	1.46e+000	154.00
	17.402	12.724-22.079		153.00
	17.402	12.724-22.079		76.00
53 2-Chloronaphthalene	17.412	12.734-22.090	1.23e+000	162.00
	17.412	12.734-22.090		127.00
	17.412	12.734-22.090		164.00
54 2-Nitroaniline	17.729	13.051-22.407	4.51e-001	65.00
	17.729	13.051-22.407		92.00
	17.729	13.051-22.407		138.00
55 1,4-Dinitrobenzene	17.985	13.307-22.663	2.75e-001	168.00
	17.985	13.307-22.663		75.00
	17.985	13.307-22.663		76.00
56 Dimethylphthalate	18.230	13.552-22.908	1.63e+000	163.00
	18.230	13.552-22.908		194.00
	18.230	13.552-22.908		164.00
57 1,3-Dinitrobenzene	18.251	13.573-22.928	2.97e-001	168.00
	18.241	13.563-22.918		75.00
	18.230	13.552-22.908		76.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
58 2,6-Dinitrotoluene	18.363	13.685-23.041	3.94e-001	165.00
	18.363	13.685-23.041		63.00
	18.363	13.685-23.041		89.00
59 Acenaphthylene	18.363	13.685-23.041	1.87e+000	152.00
	18.363	13.685-23.041		151.00
	18.363	13.685-23.041		153.00
60 1,2-Dinitrobenzene	18.517	13.839-23.194	2.05e-001	168.00
	18.517	13.839-23.194		75.00
	18.517	13.839-23.194		76.00
61 3-Nitroaniline	18.680	14.002-23.358	3.74e-001	138.00
	18.680	14.002-23.358		65.00
	18.680	14.002-23.358		92.00
* 62 Acenaphthene-d10	18.711	14.033-23.389		164.00
	18.711	14.033-23.389		162.00
	18.711	14.033-23.389		160.00
63 Acenaphthene	18.783	14.105-23.460	1.06e+000	154.00
	18.783	14.105-23.460		153.00
	18.783	14.105-23.460		152.00
64 2,4-Dinitrophenol	18.905	14.228-23.583	3.33e+000	184.00
	18.905	14.228-23.583		63.00
65 4-Nitrophenol	19.130	14.452-23.808	3.61e-001	65.00
	19.130	14.452-23.808		109.00
	19.130	14.452-23.808		139.00
66 Dibenzofuran	19.141	14.463-23.818	1.81e+000	168.00
	19.130	14.452-23.808		139.00
67 Pentachlorobenzene	19.202	14.524-23.880	6.25e-001	249.60
	19.202	14.524-23.880		251.60
	19.202	14.524-23.880		108.00
68 2,4-Dinitrotoluene	19.253	14.575-23.931	5.51e-001	165.00
	19.253	14.575-23.931		63.00
	19.253	14.575-23.931		89.00
69 2,3,5,6-Tetrachlorophen	19.427	14.749-24.105	3.87e-001	232.00
	19.427	14.749-24.105		230.00
	19.427	14.749-24.105		131.00
70 2-Napthylamine	19.509	14.831-24.187	8.47e-001	143.00
	19.509	14.831-24.187		115.00
	19.509	14.831-24.187		116.00
71 2,3,4,6-Tetrachlorophen	19.540	14.862-24.217	3.99e-001	232.00
	19.540	14.862-24.217		230.00
	19.540	14.862-24.217		131.00
72 Diethylphthalate	19.826	15.148-24.504	1.74e+000	149.00
	19.826	15.148-24.504		177.00
	19.826	15.148-24.504		150.00
73 4-Chlorophenyl-phenylet	19.928	15.250-24.606	7.15e-001	204.00
	19.928	15.250-24.606		206.00
	19.928	15.250-24.606		141.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
74 Fluorene	19.928	15.250-24.606	1.41e+000	166.00
	19.928	15.250-24.606		165.00
	19.918	15.240-24.596		167.00
75 4-Nitroaniline	20.082	15.404-24.759	3.62e-001	138.00
	20.082	15.404-24.759		65.00
	20.082	15.404-24.759		108.00
76 2-Methyl-4,6-Dinitrophe	20.174	14.667-25.680	4.32e+000	198.00
	20.174	14.667-25.680		51.00
	20.174	14.667-25.680		105.00
77 N-Nitrosodiphenylamine	20.225	14.718-25.731	5.83e-001	169.00
	20.225	14.718-25.731		168.00
	20.225	14.718-25.731		167.00
78 Diphenylamine	20.225	14.718-25.731	6.83e-001	169.00
	20.225	14.718-25.731		168.00
	20.225	14.718-25.731		167.00
79 1,2-Diphenylhydrazine	20.286	14.780-25.792	8.30e-001	77.00
	20.286	14.780-25.792		105.00
	20.286	14.780-25.792		182.00
80 Azobenzene	20.286	14.780-25.792	8.38e-001	77.00
	20.286	14.780-25.792		105.00
	20.286	14.780-25.792		182.00
$ 81 2,4,6-Tribromophenol	20.511	15.005-26.017	1.69e-001	329.60
	20.511	15.005-26.017		331.60
	20.511	15.005-26.017		141.00
82 n-Heptadecane	20.818	15.312-26.324	3.65e-001	57.00
	20.818	15.312-26.324		71.00
	20.818	15.312-26.324		85.00
83 4-Bromophenyl-phenyleth	21.023	15.516-26.529	2.47e-001	248.00
	21.023	15.516-26.529		250.00
	21.023	15.516-26.529		141.00
84 Hexachlorobenzene	21.350	15.843-26.856	3.18e-001	283.65
	21.350	15.843-26.856		248.60
	21.350	15.843-26.856		142.00
85 Dimethoate	21.391	15.885-26.897	2.33e-001	87.00
	21.391	15.885-26.897		93.00
	21.391	15.885-26.897		125.00
86 Atrazine	21.514	16.007-27.020	2.13e-001	200.00
	21.514	16.007-27.020		173.00
	21.514	16.007-27.020		215.00
87 n-Octadecane	21.831	16.324-27.337	3.79e-001	57.00
	21.831	16.324-27.337		43.00
	21.831	16.324-27.337		71.00
88 Pentachlorophenol	21.770	16.263-27.276	5.23e+000	265.60
	21.770	16.263-27.276		263.60
	21.770	16.263-27.276		267.60

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
* 89 Phenanthrene-d10	22.025	16.519-27.532		188.00
	22.025	16.519-27.532		94.00
	22.025	16.519-27.532		80.00
90 Phenanthrene	22.076	16.570-27.583	1.20e+000	178.00
	22.076	16.570-27.583		179.00
	22.076	16.570-27.583		176.00
91 Anthracene	22.179	16.672-27.685	1.23e+000	178.00
	22.179	16.672-27.685		179.00
	22.179	16.672-27.685		176.00
92 Carbazole	22.567	17.061-28.073	1.05e+000	167.00
	22.557	17.050-28.063		166.00
	22.557	17.050-28.063		139.00
93 Di-n-Butylphthalate	23.508	18.002-29.015	1.55e+000	149.00
	23.508	18.002-29.015		150.00
	23.508	18.002-29.015		104.00
94 Fluoranthene	24.797	19.291-30.303	1.22e+000	202.00
	24.797	19.291-30.303		101.00
	24.797	19.291-30.303		203.00
95 Benzidine	25.145	17.958-32.331	1.84e+000	184.00
	25.145	17.958-32.331		92.00
	25.145	17.958-32.331		185.00
96 Pyrene	25.350	18.163-32.536	1.40e+000	202.00
	25.350	18.163-32.536		200.00
	25.350	18.163-32.536		203.00
$ 97 Terphenyl-d14	25.810	18.623-32.996	9.07e-001	244.00
	25.800	18.613-32.986		122.00
	25.810	18.623-32.996		212.00
98 Butylbenzylphthalate	27.283	20.096-34.469	7.93e-001	149.00
	27.273	20.086-34.459		91.00
	27.283	20.096-34.469		206.00
99 bis (2-ethylhexyl)adipa	27.559	20.373-34.745	5.29e-001	129.00
	27.559	20.373-34.745		112.00
	27.559	20.373-34.745		147.00
100 Benzo(a)Anthracene	28.694	21.508-35.881	1.40e+000	228.00
	28.694	21.508-35.881		229.00
	28.694	21.508-35.881		226.00
101 3,3'-Dichlorobenzidine	28.715	21.528-35.901	5.23e-001	252.00
	28.715	21.528-35.901		254.00
	28.705	21.518-35.891		126.00
* 102 Chrysene-d12	28.746	21.559-35.932		240.00
	28.746	21.559-35.932		120.00
	28.746	21.559-35.932		236.00
103 Chrysene	28.827	21.641-36.013	1.30e+000	228.00
	28.827	21.641-36.013		226.00
	28.827	21.641-36.013		229.00

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Analytical Laboratory Services, Inc.

COMPOUND LISTING

Method file : \\Gc01\TargetData\chem2\ms08.i\8060307.b\8-8270-8-0226.m

Compound	RT	RT Window	RF	Mass
104 bis(2-ethylhexyl)Phthal	29.145	21.958-36.331	1.06e+000	149.00
	29.145	21.958-36.331		167.00
	29.145	21.958-36.331		279.00
105 Di-n-octylphthalate	31.057	22.789-39.325	5.92e-001	149.00
	31.057	22.789-39.325		167.00
	31.057	22.789-39.325		43.00
106 Benzo(b)fluoranthene	31.968	23.699-40.235	1.31e+000	252.00
	31.978	23.710-40.246		253.00
	31.968	23.699-40.235		125.00
107 Benzo(k)fluoranthene	32.050	23.781-40.318	1.35e+000	252.00
	32.050	23.781-40.318		253.00
	32.050	23.781-40.318		125.00
108 Benzo(a)pyrene	32.909	24.641-41.177	1.24e+000	252.00
	32.909	24.641-41.177		253.00
	32.909	24.641-41.177		125.00
* 109 Perylene-d12	33.072	24.804-41.341		264.00
	33.072	24.804-41.341		260.00
	33.072	24.804-41.341		265.00
110 Indeno(1,2,3-cd)pyrene	36.039	27.770-44.307	6.62e-001	276.00
	36.039	27.770-44.307		138.00
111 Dibenzo(a,h)anthracene	36.100	27.832-44.368	1.30e+000	278.00
	36.100	27.832-44.368		139.00
	36.100	27.832-44.368		279.00
112 Benzo(g,h,i)perylene	36.724	28.456-44.992	1.37e+000	276.00
	36.714	28.446-44.982		138.00
	36.724	28.456-44.992		277.00

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APPENDIX B

Certificates of Analysis for Primary Stock Solutions

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Certificate of Analysis

DHF

4 ampoules

Semi-Volatiles GC/MS Tuning Standard

Product Number: GCM-150 **Expiration Date:** Apr-2006

Lot Number: W-0211 04 10 08 001 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
decafluorotriphenylphosphine	005074-71-5	90004227	1003 ± 5 µg/mL
benzidine	000092-87-5	RAO18	1003 ± 5 µg/mL
pentachlorophenol	000087-86-5	07119HO	1003 ± 5 µg/mL
4,4'-DDT	000050-29-3	MD-101797	1003 ± 5 µg/mL

Matrix: methylene chloride (dichloromethane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

ISO-9001:2000
Registered by
PRO INCORPORATED
Reg. No. 00-R1192rev.1

ISO 17025
Certified by
Cert. No. 0851-01

John E. Russo, Chem. Eng.
Quality Control Manager

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CERTIFICATE OF ANALYSIS

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31850 **Lot No.:** A035788

Description: 8270 MegaMix

Expiration Date[1]: September 2006 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Pyridine	110-86-1	99%	1000 ug/mL	+/- 0.2
2	N-Nitrosodimethylamine	62-75-9	99%	1000 ug/mL	+/- 0.2
3	Aniline	62-53-3	99%	1000 ug/mL	+/- 0.2
4	Phenol	108-95-2	99%	1000 ug/mL	+/- 0.2
5	Bis(2-chloroethyl)ether	111-44-4	99%	1000 ug/mL	+/- 0.2
6	2-Chlorophenol	95-57-8	99%	1000 ug/mL	+/- 0.2
7	1,3-Dichlorobenzene	541-73-1	99%	1000 ug/mL	+/- 0.2
8	1,4-Dichlorobenzene	106-46-7	99%	1000 ug/mL	+/- 0.2
9	1,2-Dichlorobenzene	95-50-1	99%	1000 ug/mL	+/- 0.2
10	Benzyl alcohol	100-51-6	99%	1000 ug/mL	+/- 0.2
11	Bis(2-chloroisopropyl)ether	108-60-1	99%	1000 ug/mL	+/- 0.2
12	2-Methylphenol (o-cresol)	95-48-7	99%	1000 ug/mL	+/- 0.2
13	Hexachloroethane	67-72-1	99%	1000 ug/mL	+/- 0.2
14	N-Nitroso-di-n-propylamine	621-64-7	99%	1000 ug/mL	+/- 0.2
15	4-Methylphenol (p-cresol)	106-44-5	99%	500 ug/mL	+/- 0.2
16	3-Methylphenol (m-cresol)	108-39-4	99%	500 ug/mL	+/- 0.2
17	Nitrobenzene	98-95-3	99%	1000 ug/mL	+/- 0.2
18	Isophorone	78-59-1	99%	1000 ug/mL	+/- 0.2
19	2-Nitrophenol	88-75-5	99%	1000 ug/mL	+/- 0.2
20	2,4-Dimethylphenol	105-67-9	99%	1000 ug/mL	+/- 0.2
21	Bis(2-chloroethoxy)methane	111-91-1	99%	1000 ug/mL	+/- 0.2
22	2,4-Dichlorophenol	120-83-2	99%	1000 ug/mL	+/- 0.2
23	1,2,4-Trichlorobenzene	120-82-1	99%	1000 ug/mL	+/- 0.2
24	Naphthalene	91-20-3	99%	1000 ug/mL	+/- 0.2
25	4-Chloroaniline	106-47-8	99%	1000 ug/mL	+/- 0.2
26	Hexachloro-1,3-butadiene (Hexachlorobutadiene)	87-68-3	99%	1000 ug/mL	+/- 0.2
27	4-Chloro-3-methylphenol	59-50-7	99%	1000 ug/mL	+/- 0.2
28	2-Methylnaphthalene	91-57-6	99%	1000 ug/mL	+/- 0.2
29	1-Methylnaphthalene	90-12-0	99%	1000 ug/mL	+/- 0.2
30	Hexachlorocyclopentadiene	77-47-4	98%	1000 ug/mL	+/- 0.2
31	2,4,6-Trichlorophenol	88-06-2	99%	1000 ug/mL	+/- 0.2
32	2,4,5-Trichlorophenol	95-95-4	98%	1000 ug/mL	+/- 0.2
33	2-Chloronaphthalene	91-58-7	99%	1000 ug/mL	+/- 0.2
34	2-Nitroaniline	88-74-4	99%	1000 ug/mL	+/- 0.2
35	1,4-Dinitrobenzene	100-25-4	99%	1000 ug/mL	+/- 0.2
36	Acenaphthylene	208-96-8	99%	1000 ug/mL	+/- 0.2
37	1,3-Dinitrobenzene	99-65-0	99%	1000 ug/mL	+/- 0.2
38	Dimethylphthalate	131-11-3	99%	1000 ug/mL	+/- 0.2
39	2,6-Dinitrotoluene	606-20-2	99%	1000 ug/mL	+/- 0.2
40	1,2-Dinitrobenzene	528-29-0	99%	1000 ug/mL	+/- 0.2
41	Acenaphthene	83-32-9	99%	1000 ug/mL	+/- 0.2
42	3-Nitroaniline	99-09-2	99%	1000 ug/mL	+/- 0.2
43	2,4-Dinitrophenol	51-28-5	97%	1000 ug/mL	+/- 0.2
44	Dibenzofuran	132-64-9	99%	1000 ug/mL	+/- 0.2
45	2,4-Dinitrotoluene	121-14-2	98%	1000 ug/mL	+/- 0.2
46	4-Nitrophenol	100-02-7	99%	1000 ug/mL	+/- 0.2
47	2,3,4,6-Tetrachlorophenol	58-90-2	99%	1000 ug/mL	+/- 0.2
48	2,3,5,6-Tetrachlorophenol	935-95-5	99%	1000 ug/mL	+/- 0.2

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49	Fluorene	86-73-7	99%	1000 ug/mL	+/- 0.2
50	4-Chlorophenyl phenyl ether	7005-72-3	99%	1000 ug/mL	+/- 0.2
51	Diethylphthalate	84-66-2	99%	1000 ug/mL	+/- 0.2
52	4-Nitroaniline	100-01-6	99%	1000 ug/mL	+/- 0.2
53	4,6-Dinitro-2-methylphenol (Dinitro-o-cresol)	534-52-1	99%	1000 ug/mL	+/- 0.2
54	Diphenylamine	122-39-4	99%	1000 ug/mL	+/- 0.2
55	Azobenzene	103-33-3	98%	1000 ug/mL	+/- 0.2
56	4-Bromophenyl phenyl ether	101-55-3	99%	1000 ug/mL	+/- 0.2
57	Hexachlorobenzene	118-74-1	99%	1000 ug/mL	+/- 0.2
58	Pentachlorophenol	87-86-5	97%	1000 ug/mL	+/- 0.2
59	Phenanthrene	85-01-8	99%	1000 ug/mL	+/- 0.2
60	Anthracene	120-12-7	99%	1000 ug/mL	+/- 0.2
61	Carbazole	86-74-8	98%	1000 ug/mL	+/- 0.2
62	Di-n-butylphthalate	84-74-2	99%	1000 ug/mL	+/- 0.2
63	Fluoranthene	206-44-0	98%	1000 ug/mL	+/- 0.2
64	Pyrene	129-00-0	99%	1000 ug/mL	+/- 0.2
65	Benzyl butyl phthalate	85-68-7	99%	1000 ug/mL	+/- 0.2
66	Bis(2-ethylhexyl)adipate	103-23-1	99%	1000 ug/mL	+/- 0.2
67	Benz(a)anthracene	56-55-3	99%	1000 ug/mL	+/- 0.2
68	Chrysene	218-01-9	98%	1000 ug/mL	+/- 0.2
69	Bis(2-ethylhexyl)phthalate	117-81-7	99%	1000 ug/mL	+/- 0.2
70	Di-n-octyl phthalate	117-84-0	99%	1000 ug/mL	+/- 0.2
71	Benzo(b)fluoranthene	205-99-2	99%	1000 ug/mL	+/- 0.2
72	Benzo(k)fluoranthene	207-08-9	99%	1000 ug/mL	+/- 0.2
73	Benzo(a)pyrene	50-32-8	98%	1000 ug/mL	+/- 0.2
74	Indeno(1,2,3-cd)pyrene	193-39-5	99%	1000 ug/mL	+/- 0.2
75	Dibenz(a,h)anthracene	53-70-3	99%	1000 ug/mL	+/- 0.2
76	Benzo(g,h,i)perylene	191-24-2	99%	1000 ug/mL	+/- 0.2
	Solvent: Methylene Chloride (MEOH FREE)	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

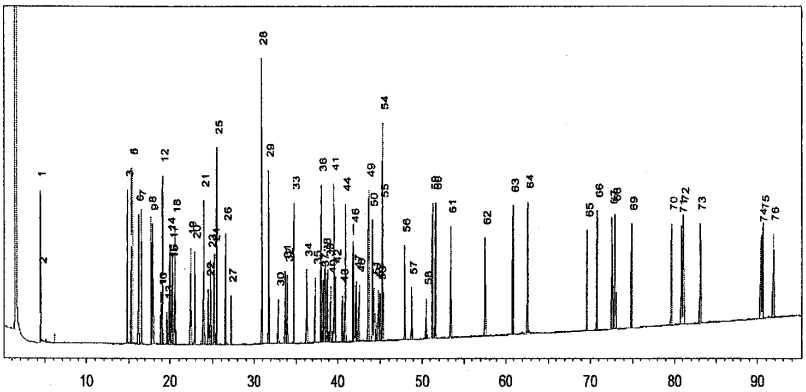
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
35°C (hold 3 min.) to 330°C
@ 3°C/min

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: FJT

John Lidgett - Q.A.Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reflactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

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Tech Tip:

N-Nitrosodiphenylamine is prone to breakdown in the injection port and will be converted to diphenylamine. N-Nitrosodiphenylamine is also a reactive species that can initiate premature decomposition of other compounds in the mix. For these reasons diphenylamine is used in the preparation of this mixture. When comparing the response of this compound to mixtures manufactured using N-nitrosodiphenylamine, a difference in response will be observed.

Note:

Product Notice 8270 MegaMix™, cat. #31850 The presence of benzene or other high boiling solvents can lead to low response,poor peak shape, or lack of resolution by gas chromatography of certain early eluting semivolatile organic compounds at low concentrations. These compounds include pyridine, N-nitrosodimethylamine, 1,4-dioxane, 2-picoline, and N-nitrosomethylethylamine. Therefore, we have discontinued the use of methylene chloride:benzene (75:25) as the solvent for this analytical reference material andreplaced it with 100% methylene chloride to ensure superior performance. If you have any questions, please do not hesitate to contact our technical servicedepartment at 800-356-1688 or 814-353-1300 ext.4. Restek Corporation 110 Benner Circle, Bellefonte, PA16823 phone: (800) 356-1688 or (814) 353-1300 fax: (814) 353-1309; www.restekcorp.com

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31494 **Lot No.:** A039458

Description: CLP 04.1 Phenols Calibration Mix

Expiration Date[1]: October 2013 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Phenol	108-95-2	99%	2000 ug/mL	+/- 0.1
2	2-Chlorophenol	95-57-8	99%	2000 ug/mL	+/- 0.1
3	2-Methylphenol (o-cresol)	95-48-7	99%	2000 ug/mL	+/- 0.1
4	4-Methylphenol (p-cresol)	106-44-5	99%	2000 ug/mL	+/- 0.1
5	2-Nitrophenol	88-75-5	99%	2000 ug/mL	+/- 0.1
6	2,4-Dimethylphenol	105-67-9	99%	2000 ug/mL	+/- 0.1
7	2,4-Dichlorophenol	120-83-2	99%	2000 ug/mL	+/- 0.1
8	4-Chloro-3-methylphenol	59-50-7	99%	2000 ug/mL	+/- 0.1
9	2,4,6-Trichlorophenol	88-06-2	99%	2000 ug/mL	+/- 0.1
10	2,4,5-Trichlorophenol	95-95-4	98%	2000 ug/mL	+/- 0.1
11	2,4-Dinitrophenol	51-28-5	98%	2000 ug/mL	+/- 0.1
12	4-Nitrophenol	100-02-7	99%	2000 ug/mL	+/- 0.1
13	4,6-Dinitro-2-methylphenol (Dinitro-o-cresol)	534-52-1	99%	2000 ug/mL	+/- 0.1
14	Pentachlorophenol	87-86-5	97%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

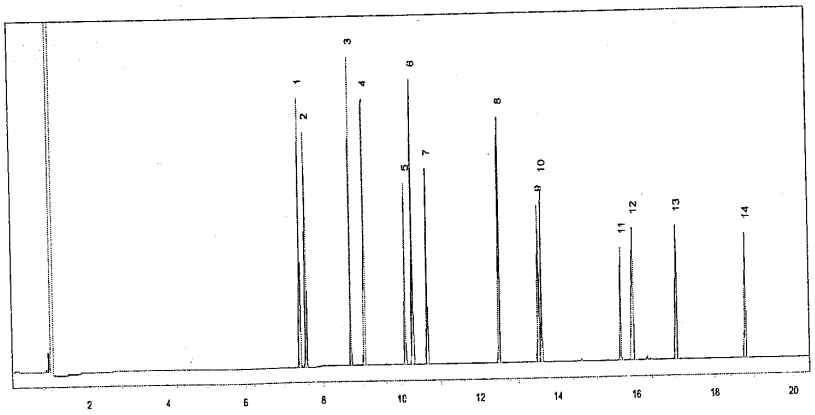
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 10°C/min. (hold 5 min.)

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: n/a

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

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rptCertificate

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31834 **Lot No.:** A039446

Description: Benzidine Mix

Expiration Date[1]: September 2008 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Benzidine	92-87-5	99%	2000 ug/mL	+/- 0.1
2	3,3'-Dichlorobenzidine	91-94-1	99%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

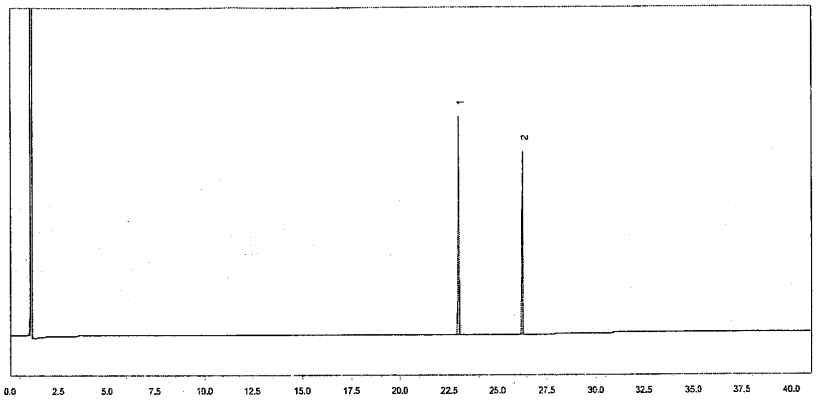
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
40°C (hold 2 min.) to 330°C
@ 10°C/min. (hold 10 min.)

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: GD

John Lidgett - Q.A.Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 559802 **Lot No.:** A042833

Description: Custom Acids Supplemental Standard

Expiration Date[1]: February 2009 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	alpha-Terpineol (+)	7785-53-7	99%	500 ug/mL	+/- 0.1
2	2,6-Dichlorophenol	87-65-0	99%	2000 ug/mL	+/- 0.1
3	Benzoic acid	65-85-0	99%	2000 ug/mL	+/- 0.1
4	2,3,4,6-Tetrachlorophenol	58-90-2	99%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .25um
Rtx-5 (cat.#10223-124)

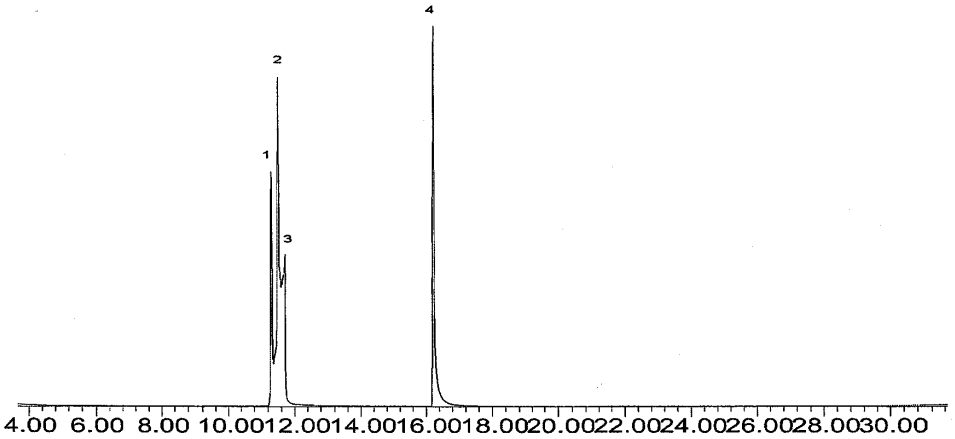
Carrier Gas:
helium @ 1 ml/min.

Temp. Program:
40°C (hold 2 min.) to 330°C
@10°C/min. (hold 10 min.)

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
MSD



Manufactured By: FJT

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

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110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 559801 **Lot No.:** A042834

Description: Custom B/N Supplemental Standard

Expiration Date[1]: August 2007 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Dioxane	123-91-1	99%	2000 ug/mL	+/- 0.1
2	N-Nitrosodiethylamine	55-18-5	99%	2000 ug/mL	+/- 0.1
3	Benzaldehyde	100-52-7	99%	2000 ug/mL	+/- 0.1
4	n-Decane (C10)	124-18-5	99%	2000 ug/mL	+/- 0.1
5	Acetophenone	98-86-2	99%	2000 ug/mL	+/- 0.1
6	N-Nitrosopyrrolidine	930-55-2	99%	2000 ug/mL	+/- 0.1
7	N-Nitrosodi-n-butylamine	924-16-3	99%	2000 ug/mL	+/- 0.1
8	epsilon-Caprolactam	105-60-2	99%	2000 ug/mL	+/- 0.1
9	1,2,4,5-Tetrachlorobenzene	95-94-3	99%	2000 ug/mL	+/- 0.1
10	2,3-Dichloroaniline	608-27-5	98%	2000 ug/mL	+/- 0.1
11	Biphenyl	92-52-4	99%	2000 ug/mL	+/- 0.1
12	Pentachlorobenzene	608-93-5	99%	2000 ug/mL	+/- 0.1
13	2-Naphthylamine (2-aminonaphthalene)	91-59-8	99%	2000 ug/mL	+/- 0.1
14	n-Heptadecane (C17)	629-78-7	99%	2000 ug/mL	+/- 0.1
15	Dimethoate	60-51-5	99%	2000 ug/mL	+/- 0.1
16	Atrazine	1912-24-9	99%	2000 ug/mL	+/- 0.1
17	n-Octadecane (C18)	593-45-3	99%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .25um
Rtx-5 (cat.#10223-124)

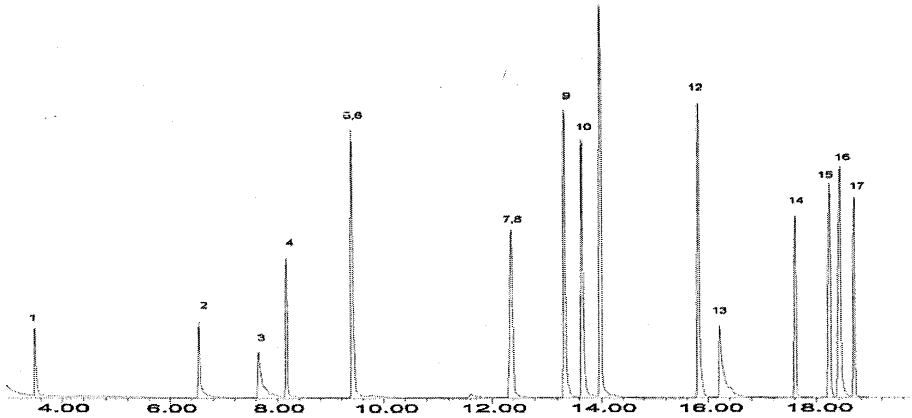
Carrier Gas:
helium @ 1 ml/min.

Temp. Program:
40°C (hold 2 min.) to 240°C @10°C/min.

Inj. Temp:
200°C

Det. Temp:
300°C

Det. Type:
MSD



Manufactured By: FJT

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

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RESTEK

CERTIFICATE OF COMPOSITION

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 559803 **Lot No.:** A042850

Description: Custom Resorcinol Standard

Expiration Date[1]: February 2009 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Resorcinol	108-46-3	99%	100 mg/mL	+/- 0.1
	Solvent: MTBE	1634-04-4	99%		

Column:
30m x .25mm x .25um
Rtx-5 (cat.#10223-124)

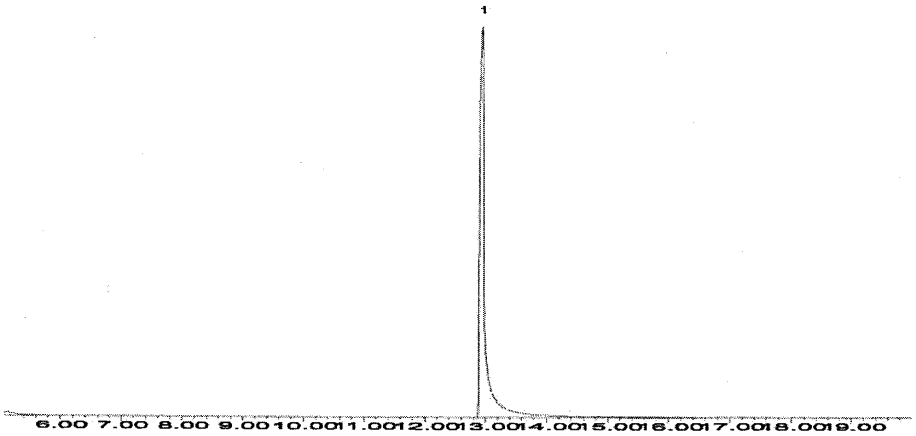
Carrier Gas:
helium @ 1 ml/min.

Temp. Program:
40°C (hold 2 min.) to 220°C
@10°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
MSD



Manufactured By: MC

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

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Certificate of Analysis

Number : 050629014
Expires : 04/01/08
Open Date: 1/24/6
Opened By: DHR

Opened 1/24/6

DESCRIPTION: 8270 Surrogate Standard

CATALOG NO.: 47960-U MFG DATE: Apr-2005

LOT NO.: LB29380 EXPIRATION DATE: Apr-2008

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
NITROBENZENE-D5	4165-60-0	99.9	4000	4119	+/-	76.9	LB24286
P-TERPHENYL-D14	1718-51-0	99.9	4000	4017	+/-	33.4	LB24806
PHENOL-D6	13127-88-3	99.9	4000	4139	+/-	87.1	LB23082
2-FLUOROBIPHENYL	321-60-8	99.9	4000	4096	+/-	54.7	LB12824
2-FLUOROPHENOL	367-12-4	99.7	4000	4166	+/-	99.5	LB08773
2,4,6-TRIBROMOPHENOL	118-79-6	99.9	4000	4058	+/-	19.2	LB21400

(1) Listed in alphabetical order.
(2) Determined by capillary GC-FID, unless otherwise noted.
(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.
(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

Supelco warrants that its products conform to the information contained in this publication. Purchaser must determine the suitability of the product for its particular use. Please see the latest catalog or order invoice and packing slip for additional terms and conditions of sale.

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA

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RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31206 **Lot No.:** A037805

Description: SV Internal Standard Mix 2mg/ml 06 0106003

Expiration Date[1]: August 2008 **Storage:** Refrigerate

Handling: Warm & Sonicate prior to use

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Dichlorobenzene-d4	3855-82-1	99%	2000 ug/mL	+/- 0.03
2	Naphthalene-d8	1146-65-2	97%	2000 ug/mL	+/- 0.03
3	Acenaphthene-d10	15067-26-2	99%	2000 ug/mL	+/- 0.03
4	Phenanthrene-d10	1517-22-2	97%	2000 ug/mL	+/- 0.03
5	Chrysene-d12	1719-03-5	98%	2000 ug/mL	+/- 0.03
6	Perylene-d12	1520-96-3	99%	2000 ug/mL	+/- 0.03
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

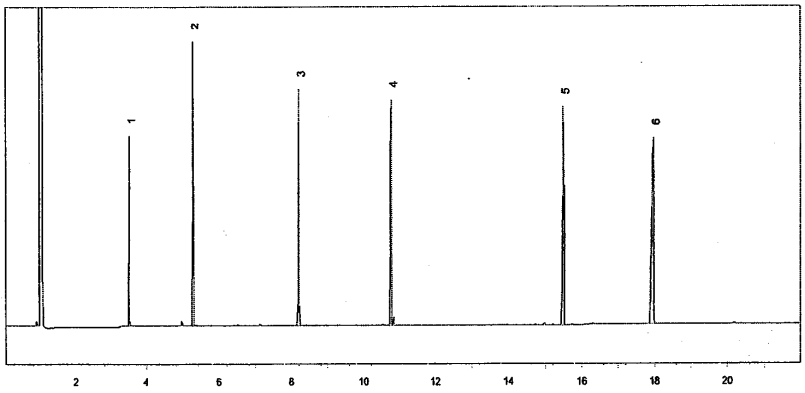
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
75°C to 330°C
@ 15°C/min. (hold 5 min.)

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: SLB

John Lidgett - Q.A.Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

15 vials

Mixture Name: Custom BNA Mix
Catalog Number: Q-4570
Lot Number: 012706-01

Number : 060201003
Expires : 02/01/2011
Open Date: _____
Opened By: _____

Preparation Date: 01/27/06
Solvent: Methylene chloride
(FLAMMABLE, IRRITANT)

COMPOUND NUMBER	ANALYTE	PURITY (%)	GRAVIMETRIC CONCENTRATION (ug/mL)	DILUTION	FINAL CONCENTRATION (ug/mL)
W-01-04	Acenaphthene	99.9	3200	3.125:100	100
W-75-17	Acenaphthylene	99.5	3200	3.125:100	100
W-76-05	Anthracene	99.5	3200	3.125:100	100
W-70-03	Benzo(a)anthracene	99.7	3200	3.125:100	100
W-71-13	Benzo(a)pyrene	99.6	3200	3.125:100	100
W-72-13	Benzo(b)fluoranthene	99.2	3200	3.125:100	100
W-73-12	Benzo(k)fluoranthene	99.0	3200	3.125:100	100
W-77-11	Benzo(g,h,i)perylene	99.8	3200	3.125:100	100
W-74-10	Chrysene	99.8	3200	3.125:100	100
W-231-15	Dibenz(a,h)anthracene	99.3	3200	3.125:100	100
W-37-04	Fluoranthene	99.8	3200	3.125:100	100
W-78-04	Fluorene	98.2	3200	3.125:100	100
W-81-17	Indeno(1,2,3-c,d)pyrene	99.8	3200	3.125:100	100
W-53-04	Naphthalene	99.0	3200	3.125:100	100
W-79-09	Phenanthrene	99.4	3200	3.125:100	100
W-82-07	Pyrene	98.4	3200	3.125:100	100
W-39-04	4-Bromophenyl phenyl ether	98.9	3200	3.125:100	100
W-65-03	Butyl benzyl phthalate	96.3	3200	3.125:100	100
W-41-08	bis(2-Chloroethoxy)methane	99.4	3200	3.125:100	100
W-16-05	bis(2-Chloroethyl)ether	99.9	3200	3.125:100	100
W-40-19	bis(2-Chloroisopropyl)ether	99.6	3200	3.125:100	100
W-38-10	4-Chlorophenyl phenyl ether	99.8	3200	3.125:100	100
W-68-05	Diethyl phthalate	98.5	3200	3.125:100	100
W-69-04	Dimethyl phthalate	99.6	3200	3.125:100	100
W-66-04	Di-n-butyl phthalate	98.9	3200	3.125:100	100
W-67-08	Di-n-octyl phthalate	99.7	3201	3.125:100	100
W-64-05	bis(2-Ethylhexyl)phthalate	99.0	3200	3.125:100	100
W-59-05	N-Nitrosodimethylamine	99.8	3200	3.125:100	100
W-61-09	N-Nitrosodi-n-propylamine	99.8	3201	3.125:100	100
W-60-05	N-Nitrosodiphenylamine	96.9	3200	3.125:100	100
W-542-03	Aniline	99.9	3200	3.125:100	100
W-1250-01	Azobenzene	98.5	3200	3.125:100	100
W-422-05	Carbazole	98.6	3200	3.125:100	100
W-305-04	4-Chloroaniline	99.9	3200	3.125:100	100
W-324-03	o-Nitroaniline	99.9	3200	3.125:100	100
W-325-03	m-Nitroaniline	99.7	3200	3.125:100	100
W-342-02	p-Nitroaniline	99.9	3200	3.125:100	100
W-326-07	Benzyl alcohol	99.9	3200	3.125:100	100
W-18-05	2-Chloronaphthalene	99.9	3200	3.125:100	100

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

COMPOUND NUMBER	ANALYTE	PURITY (%)	GRAVIMETRIC CONCENTRATION (ug/mL)	DILUTION	FINAL CONCENTRATION (ug/mL)
W-261-04	Dibenzofuran	99.9	3200	3.125:100	100
W-23-03	1,2-Dichlorobenzene	99.1	3235	3.125:100	101
W-214-05	1,3-Dichlorobenzene	99.7	3211	3.125:100	100
W-25-07	1,4-Dichlorobenzene	99.9	3200	3.125:100	100
W-33-05	2,4-Dinitrotoluene	98.7	3200	3.125:100	100
W-34-06	2,6-Dinitrotoluene	99.8	3200	3.125:100	100
W-08-04	Hexachlorobenzene	99.9	3200	3.125:100	100
W-50-05	Hexachlorobutadiene	98.7	3200	3.125:100	100
W-51-08	Hexachlorocyclopentadiene	98.8	3200	3.125:100	100
W-11-04	Hexachloroethane	99.8	3200	3.125:100	100
W-52-04	Isophorone	97.9	3200	3.125:100	100
W-562-03	2-Methylnaphthalene	99.0	3200	3.125:100	100
W-54-04	Nitrobenzene	99.9	3200	3.125:100	100
W-07-02	1,2,4-Trichlorobenzene	99.9	3200	3.125:100	100
W-541-04	Benzoic acid	99.8	5001	2:100	100
W-20-06	4-Chloro-3-methylphenol	99.8	3209	3.125:100	100
W-22-08	2-Chlorophenol	99.3	3200	3.125:100	100
W-250-03	o-Cresol	98.8	3206	3.125:100	100
W-252-03	p-Cresol	99.9	3214	3.125:100	100
W-29-07	2,4-Dichlorophenol	98.5	3204	3.125:100	100
W-224-07	2,4-Dimethylphenol	99.7	3207	3.125:100	100
W-58-04	4,6-Dinitro-2-methylphenol	87.0	3202	3.125:100	100
W-57-07	2,4-Dinitrophenol	86.0	3207	3.125:100	100
W-55-04	2-Nitrophenol	99.9	3208	3.125:100	100
W-56-05	4-Nitrophenol	99.8	3203	3.125:100	100
W-62-10	Pentachlorophenol	99.8	3206	3.125:100	100
W-63-05	Phenol	99.8	3203	3.125:100	100
W-179-07	2,4,5-Trichlorophenol	99.8	3201	3.125:100	100
W-19-08	2,4,6-Trichlorophenol	99.9	3221	3.125:100	101
W-271-05	Pyridine	99.6	5000	2:100	100
W-411-06	Acetophenone	99.8	2000	5:100	100
W-236-03	n-Decane	99.8	2004	5:100	100
W-1621-03	2,3-Dichloroaniline	99.8	2005	5:100	100
W-1288-01	1-Methylnaphthalene	99.1	2002	5:100	100
W-243-02	n-Octadecane	99.8	2000	5:100	100
W-1635-01	a-Terpineol	94.0	500	5:100	25
W-1483-01	1,2-Dinitrobenzene	99.0	1999	5:100	100
W-527-03	1,3-Dinitrobenzene	95.1	1989	5:100	99
W-1484-02	1,4-Dinitrobenzene	99.8	1999	5:100	100
W-498-08	2,3,4,6-Tetrachlorophenol	95.2	2020	5:100	101
W-652-03	2,3,5,6-Tetrachlorophenol	99.0	1990	5:100	100
W-1211-03	bis(2-Ethylhexyl)adipate	99.6	1992	5:100	100
W-345-13	Dimethoate	98.0	2002	5:100	100
W-1365-01	2-Napthylamine	95.0	2110	5:100	106
W-700-02	Resorcinol	99.0	2036	5:100	102
W-1473-01	Caprolactam	99.0	2017	5:100	101
W-298-03	n-Nitrospyrrolidine	99.9	2005	5:100	100
W-334-04	n-Nitrosodiethylamine	99.9	2005	5:100	100
W-404-05	n-Nitrosodi-n-butylamine	99.8	2011	5:100	101
W-177-03	1,2,4,5-Tetrachlorobenzene	99.5	2015	5:100	101

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

W-260-04	Pentachlorobenzene	99.9	2013	5:100	101
W-219-02	Mirex	99.3	2033	5:100	102
W-302-04	2,6-Dichlorophenol	99.9	2023	5:100	101
W-317-02	1,1-Biphenyl	99.0	1998	5:100	100
W-1090-11	Atrazine	98.0	2002	5:100	100
W-407-03	Benzaldehyde	99.0	1996	5:100	100
W-242-02	n-Heptadecane	99.4	1995	5:100	100

PREPARATION: Reported concentration value has been corrected for purity using purity values obtained by NSI analysts. Concentration is calculated from wt/vol or vol/vol measurements using microbalances calibrated with NIST traceable weights to 0.0001 g and or ASTM Class A volumetric glassware or calibrated equivalent labware.

STORAGE & HANDLING: Store at ≤ 5° C. Allow equilibration to room temperature before use.

SHAKE WELL BEFORE USING!

Kristi Bright, Organic Chemist

15 vials
opened
2/3/16 DHT

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APPENDIX C
Preparation of DFTTP Standard

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SV Tuning Solution at 50 mg/l

Add reagents to 40 ml VOC vial:

Volume	Reagent	Conc.	Maker	Lot No	Log-in No
1.4 ml (syringe)	Semi-volatiles GC/MS Tuning Standard	1000 ug/ml	Ultra		
26.6 ml (grad cyl)	Methylene chloride				
28 ml	Final	50 mg/l			

Stored in foil-wrapped vial in refrigerator 8 in organic prep lab.
Transfer to GC vials for use.

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APPENDIX D

Preparation of Calibration Standards

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BN275

Calibration Standards for 8270C and 625

Primary and Secondary Stocks.

Prepare Secondary Stock by adding volumes indicated below to a 5 ml amber vial.
Use Secondary Stock to prepare Working Standards.
Save leftover Secondary Stock to make fresh CCAL weekly.

Primary Stock	Volume Primary Standard, uL	Supplier	Catalog No.	Lot #	ALSI Log #	Primary Stock, ug/ml	Secondary Stock, ug/ml	Exp. Date	Comment
MegaMix	700	Restek	31686-510			1000	200		
Benzidine Mix	350	Restek	31834			2000	200		
CLP 04.1 Phenols	350	Restek	31494	A039458		2000	200		
Custom Acids Sup	350	Restek	559802	A042833		2000	200		0.25x terp
Custom BN Sup	350	Restek	559801	A042834		2000	200		
Custom Resorcinol	7	Restek	559803	A042850		100000	200		in MTBE
8270 Surrogates	175	Supelco	47960-U			4000	200		
Methylene Chloride	1218								
Final Volume	3500								

		Supplier	Catalog No.	Lot #	ALSI Login #	Stock, ug/ml	Vial opened:	Exp. Date	Comment
Internal Standard		Restek	31206			2000			

Phenols Mix/10:	900 ul Methylene Chloride + 100 ul Phenols Mix.	Prep: _____		

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BN275

Working Standards.

Preparation of Standards
Mix volumes indicated below in 2 ml amber crimp top vials.
Make 3 vials of SSTD050, labelled BNA-50A, BNA-50B, BNA-50C
Save leftover secondary stock (about 1.5 ml) to make fresh CCAL weekly.
Store at 4C.

Label vials with:	Example:
Level:	BNA050
Std ID:	BN275
Preparer:	ttr
Date Prep	O21406

Preparation of ICAL Standards

Level	Volume MeCl, uL	Volume Secondary Stock, uL	Volume Phenols Mix/10, uL	Volume ISTD, uL	Total Volume, uL	Nominal Conc., ug/ml	*Phenols Nominal Conc., ug/ml*	*alpha-Terpinol Nominal Conc., ug/ml*	*N-NDPhA Nominal Conc., ug/ml*
BNA-1.5	987.5	7.5	5	20	1020	1.5	*4*	*0.375*	*1.7415*
BNA-3.0	975	15	10	20	1020	3	*8*	*0.75*	*3.483*
BNA-8.0	960	40	0	20	1020	8	*16*	*2*	*9.288*
BNA-16	920	80	0	20	1020	16	*32*	*4*	*18.576*
BNA-50 (CCAL)	750	250	0	20	1020	50	*100*	*12.5*	*58.05*
BNA-70	650	350	0	20	1020	70	*140*	*17.5*	*81.27*
BNA-90	550	450	0	20	1020	90	*180*	*22.5*	*104.49*
BNA-120	400	600	0	20	1020	120	*240*	*30*	*139.32*
Prep By:			Prep Date:				Exp. Date:		

Preparation of Second sources:

Primary Stock	Volume Primary Standard, uL	Supplier	Catalog No.	Lot #	ALSI Log #	Exp. Date	Volume MeCl, uL	Volume ISTD, uL	Primary Stock, ug/ml	Final Conc., ug/ml
BNA 2nd	1000	NSI	Q-4570				0	20	100	100
Mega 2nd	100	Restek	31686				900	20	1000	100
Benzidine spike*	500	Prep							100	50
Surrogate spike*	500	Prep					0	20	100	50
Prep By:			Prep Date:				Exp. Date:			

*Benzidine and Surrogate spike are combined in one vial

Weekly Prep of CCAL (labelled, for example, BNA-050D, BNA-050E, etc)

Standard ID	D	E	F	G	H	I
Prep Date:						
Exp. Date:						
Prepared by:						

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APPENDIX E

Certificate of Analysis of BNA Spike Mix

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

Mixture Name: Custom BNA Spike Mix
Catalog Number: Q-4494
Lot Number: 021406-05

Preparation Date: 02/14/06
Solvent: Methanol
(FLAMMABLE, IRRITANT)

COMPOUND NUMBER	ANALYTE	PURITY (%)	GRAVIMETRIC CONCENTRATION (ug/mL)	DILUTION	FINAL CONCENTRATION (ug/mL)
W-01-04	Acenaphthene	99.9	3200	3.125:100	100
W-75-17	Acenaphthylene	99.5	3200	3.125:100	100
W-76-05	Anthracene	99.5	3200	3.125:100	100
W-70-03	Benzo(a)anthracene	99.7	3200	3.125:100	100
W-71-13	Benzo(a)pyrene	99.6	3200	3.125:100	100
W-72-13	Benzo(b)fluoranthene	99.2	3200	3.125:100	100
W-73-12	Benzo(k)fluoranthene	99.0	3200	3.125:100	100
W-77-11	Benzo(g,h,i)perylene	99.8	3200	3.125:100	100
W-74-10	Chrysene	99.8	3200	3.125:100	100
W-231-15	Dibenz(a,h)anthracene	99.3	3200	3.125:100	100
W-37-04	Fluoranthene	99.8	3200	3.125:100	100
W-78-04	Fluorene	98.2	3200	3.125:100	100
W-81-17	Indeno(1,2,3-c,d)pyrene	99.8	3200	3.125:100	100
W-53-04	Naphthalene	99.0	3200	3.125:100	100
W-79-09	Phenanthrene	99.4	3200	3.125:100	100
W-82-07	Pyrene	98.4	3200	3.125:100	100
W-39-04	4-Bromophenyl phenyl ether	98.9	3200	3.125:100	100
W-65-03	Butyl benzyl phthalate	96.3	3200	3.125:100	100
W-41-08	bis(2-Chloroethoxy)methane	99.4	3200	3.125:100	100
W-16-05	bis(2-Chloroethyl)ether	99.9	3200	3.125:100	100
W-40-19	bis(2-Chloroisopropyl)ether	99.6	3200	3.125:100	100
W-38-10	4-Chlorophenyl phenyl ether	99.8	3200	3.125:100	100
W-68-05	Diethyl phthalate	98.5	3200	3.125:100	100
W-69-04	Dimethyl phthalate	99.6	3200	3.125:100	100
W-66-04	Di-n-butyl phthalate	98.9	3200	3.125:100	100
W-67-08	Di-n-octyl phthalate	99.7	3201	3.125:100	100
W-64-05	bis(2-Ethylhexyl)phthalate	99.0	3200	3.125:100	100
W-59-05	N-Nitrosodimethylamine	99.8	3200	3.125:100	100
W-61-09	N-Nitrosodi-n-propylamine	99.8	3201	3.125:100	100
W-60-05	N-Nitrosodiphenylamine	96.9	3200	3.125:100	100
W-542-03	Aniline	99.9	3200	3.125:100	100
W-1250-01	Azobenzene	98.5	3200	3.125:100	100
W-422-05	Carbazole	98.6	3200	3.125:100	100
W-305-04	4-Chloroaniline	99.9	3200	3.125:100	100
W-324-03	o-Nitroaniline	99.9	3200	3.125:100	100
W-325-03	m-Nitroaniline	99.7	3200	3.125:100	100
W-342-02	p-Nitroaniline	99.9	3200	3.125:100	100
W-326-07	Benzyl alcohol	99.9	3200	3.125:100	100
W-18-05	2-Chloronaphthalene	99.9	3200	3.125:100	100

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

COMPOUND NUMBER	ANALYTE	PURITY (%)	GRAVIMETRIC CONCENTRATION (ug/mL)	DILUTION	FINAL CONCENTRATION (ug/mL)
W-261-04	Dibenzofuran	99.9	3200	3.125:100	100
W-23-03	1,2-Dichlorobenzene	99.1	3235	3.125:100	101
W-214-05	1,3-Dichlorobenzene	99.7	3211	3.125:100	100
W-25-07	1,4-Dichlorobenzene	99.9	3200	3.125:100	100
W-33-05	2,4-Dinitrotoluene	98.7	3200	3.125:100	100
W-34-06	2,6-Dinitrotoluene	99.8	3200	3.125:100	100
W-08-04	Hexachlorobenzene	99.9	3200	3.125:100	100
W-50-05	Hexachlorobutadiene	98.7	3200	3.125:100	100
W-51-08	Hexachlorocyclopentadiene	98.8	3200	3.125:100	100
W-11-04	Hexachloroethane	99.8	3200	3.125:100	100
W-52-04	Isophorone	97.9	3200	3.125:100	100
W-562-03	2-Methylnaphthalene	99.0	3200	3.125:100	100
W-54-04	Nitrobenzene	99.9	3200	3.125:100	100
W-07-02	1,2,4-Trichlorobenzene	99.9	3200	3.125:100	100
W-541-04	Benzoic acid	99.8	5001	2:100	100
W-20-06	4-Chloro-3-methylphenol	99.8	3209	3.125:100	100
W-22-08	2-Chlorophenol	99.3	3200	3.125:100	100
W-250-03	o-Cresol	98.8	3206	3.125:100	100
W-252-03	p-Cresol	99.9	3214	3.125:100	100
W-29-07	2,4-Dichlorophenol	98.5	3204	3.125:100	100
W-224-07	2,4-Dimethylphenol	99.7	3207	3.125:100	100
W-58-04	4,6-Dinitro-2-methylphenol	87.0	3202	3.125:100	100
W-57-07	2,4-Dinitrophenol	86.0	3207	3.125:100	100
W-55-04	2-Nitrophenol	99.9	3208	3.125:100	100
W-56-05	4-Nitrophenol	99.8	3203	3.125:100	100
W-62-10	Pentachlorophenol	99.8	3206	3.125:100	100
W-63-05	Phenol	99.8	3203	3.125:100	100
W-179-07	2,4,5-Trichlorophenol	99.8	3201	3.125:100	100
W-19-08	2,4,6-Trichlorophenol	99.9	3221	3.125:100	101
W-271-05	Pyridine	99.6	5000	2:100	100
W-411-06	Acetophenone	99.8	2000	5:100	100
W-236-03	n-Decane	99.8	2004	5:100	100
W-1621-03	2,3-Dichloroaniline	99.8	2005	5:100	100
W-1288-01	1-Methylnaphthalene	99.1	2002	5:100	100
W-243-02	n-Octadecane	99.8	2000	5:100	100
W-1635-01	a-Terpineol	94.0	500	5:100	25
W-1483-01	1,2-Dinitrobenzene	99.0	1999	5:100	100
W-527-03	1,3-Dinitrobenzene	95.1	1989	5:100	99
W-1484-02	1,4-Dinitrobenzene	99.8	1999	5:100	100
W-498-08	2,3,4,6-Tetrachlorophenol	95.2	2020	5:100	101
W-652-03	2,3,5,6-Tetrachlorophenol	99.0	1990	5:100	100
W-1211-03	bis(2-Ethylhexyl)adipate	99.6	1992	5:100	100
W-345-13	Dimethoate	98.0	2002	5:100	100
W-1365-01	2-Napthylamine	95.0	2110	5:100	106
W-700-02	Resorcinol	99.0	2036	5:100	102
W-1473-01	Caprolactam	99.0	2017	5:100	101
W-298-03	n-Nitrospyrrolidine	99.9	2005	5:100	100
W-334-04	n-Nitrosodiethylamine	99.9	2005	5:100	100
W-404-05	n-Nitrosodi-n-butylamine	99.8	2011	5:100	101
W-177-03	1,2,4,5-Tetrachlorobenzene	99.5	2015	5:100	101

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Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

W-260-04	Pentachlorobenzene	99.9	2013	5:100	101
W-219-02	Mirex	99.3	2033	5:100	102
W-302-04	2,6-Dichlorophenol	99.9	2023	5:100	101
W-317-02	1,1-Biphenyl	99.0	1998	5:100	100
W-1090-11	Atrazine	98.0	2002	5:100	100
W-407-03	Benzaldehyde	99.0	1996	5:100	100
W-242-02	n-Heptadecane	99.4	1995	5:100	100

PREPARATION: Reported concentration value has been corrected for purity using purity values obtained by NSI analysts. Concentration is calculated from wt/vol or vol/vol measurements using microbalances calibrated with NIST traceable weights to 0.0001 g and or ASTM Class A volumetric glassware or calibrated equivalent labware.

STORAGE & HANDLING: Store at $\leq 5^{\circ}$ C. Allow equilibration to room temperature before use.

SHAKE WELL BEFORE USING!

Kristi Bright

Kristi Bright, Organic Chemist

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APPENDIX F
Data Acquisition

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```
                              TOPLEVEL PARAMETERS
                              -------------------

Method Information For: C:\HPCHEM\1\METHODS\8-8270.M

Method Sections To Run:

  ( ) Save Copy of Method With Data
  ( ) Pre-Run Cmd/Macro  = macro "autolan.mac"  autolan "c:\chemlan\8270.n",
  (X) Data Acquisition
  ( ) Data Analysis
  ( ) Post-Run Cmd/Macro = macro "msacq2.mac", go

Method Comments:
  8270 METHOD

                              END OF TOPLEVEL PARAMETERS
                              --------------------------



                              INSTRUMENT CONTROL PARAMETERS
                              -----------------------------

Sample Inlet:        GC
Injection Source:    GC ALS
Mass Spectrometer:   Enabled

                              HP GC Injector

     Front Injector:
No parameters specified

     Back Injector:
        Sample Washes                 1
        Sample Pumps                  3
        Injection Volume            1.0 microliters
        Syringe Size               10.0 microliters
        On Column                   Off
        Nanoliter Adapter           Off
        PostInj Solvent A Washes      4
        PostInj Solvent B Washes      4
        Viscosity Delay               1 seconds
        Plunger Speed              Fast

                         HP5890 Temperature Parameters

Zone Temperatures:  State     Setpoint
     Inlet A:        Off        50 C
     Inlet B:         On       250 C
     Detector A:     Off        50 C
     Detector B:      On       280 C

Method: 8-8270.M            Wed Mar 08 13:17:34 2006            Page: 1
```

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```
     Auxiliary:      Off        50 C

Oven Parameters:
     Oven Equib Time:         0.50 minutes
     Oven Max:                 350 C
     Oven State:                On
     Cryo State:               Off
     Cryo Blast:               Off
     Ambient:                   35 C

Oven Program:
     Initial Temperature:       45 C
     Initial Time:            5.00 minutes

                 Rate            Final            Final
     Level    (C/minute)    Temperature (C)   Time (minutes)
      1          10.6             245              0.00
      2(A)        6.0             320              1.50
      3(B)        0.0               0              0.00
     Next Run Time:          37.87 minutes


                        HP5890 Inlet Pressure Programs

GC Pressure Units:  psi

Inlet A:
     Constant Flow:              Off
     Constant Flow Pressure:     0.0 psi
     Constant Flow Temperature:   50 C
     Initial Pressure:           0.0 psi
     Initial Time:            650.00 minutes

                 Rate            Final            Final
     Level    (psi/minute)   Pressure (psi)   Time (minutes)
      1          0.00             0.0              0.00
      2(A)       0.00             0.0              0.00
      3(B)       0.00             0.0              0.00
     Total Program Time:      650.00 minutes

     Column Length:            30.00 m
     Column Diameter:          0.530 mm
     Gas:                         He
     Vacuum Compensation:        Off

Inlet B:
     Constant Flow:              Off
     Constant Flow Pressure:    14.0 psi
     Constant Flow Temperature:  150 C
     Initial Pressure:           7.7 psi
     Initial Time:              0.00 minutes

                 Rate            Final            Final
     Level    (psi/minute)   Pressure (psi)   Time (minutes)
      1         99.00            25.0              0.70
      2(A)      99.00             7.7              1.00
      3(B)       0.97            39.6              2.40
     Total Program Time:       37.34 minutes

Method: 8-8270.M            Wed Mar 08 13:17:34 2006            Page: 2
```

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Column Length:	30.00 m
Column Diameter:	0.250 mm
Gas:	He
Vacuum Compensation:	On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.69	0.00	Yes
B	Off	0.69	0.00	Yes

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:

Valve 1: Off	Valve 2: Off	Valve 3: Off	Valve 4: Off

19405 Valves:

Valve 5: Off	Valve 6: Off	Valve 7: Off	Valve 8: Off

19405 Relays:

Relay 1: Off	Relay 2: Off	Relay 3: Off	Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Comp1	0.053	5.000	0.00	1.00
2	Comp1	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETERS

General Information
--- ---

Tune File	: dftpp.u
Acquistion Mode	: Scan

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```
MS Information
-- -----------

Solvent Delay              : 3.50 min

EM Absolute                : False
EM Offset                  : 0
Resulting EM Voltage       : 1600.0

[Scan Parameters]

Low Mass                   : 35
High Mass                  : 500
Threshold                  : 100
Sample #                   : 2       A/D Samples    4


                             END OF MS ACQUISITION PARAMETERS

                            END OF INSTRUMENT CONTROL PARAMETERS
                            ------------------------------------




                            DATA ANALYSIS PARAMETERS
                            ------------------------

Method Name: C:\HPCHEM\1\METHODS\8-8270.M


Percent Report Settings
-----------------------

Sort By: Signal

Output Destination
    Screen:  No
    Printer: Yes
    File:    No

Integration Events: AutoIntegrate

Generate Report During Run Method:  No

Signal Correlation Window: 0.020


Qualitative Report Settings
---------------------------
```


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```
Peak Location of Unknown: Apex

Library to Search       Minimum Quality
DEMO.L                   0

Integration Events: AutoIntegrate

Report Type: Summary

Output Destination
    Screen:  No
    Printer: Yes
    File:    No

Generate Report During Run Method:  No


Quantitative Report Settings
----------------------------

Report Type: Summary

Output Destination
    Screen:  Yes
    Printer: No
    File:    No

Generate Report During Run Method:  Yes



Calibration Last Updated:


Reference Window: 10.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information
--------------------

------------------------------------------------------------------------------
*** Empty Quantitation Database ***


                    END OF DATA ANALYSIS PARAMETERS
                    -------------------------------
```


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APPENDIX G

Criteria for Second Source Standards

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SVMS 2005
2nd Source Control Limits

Analysis Method: 8270, 625

Units: % Recovery

90% of target analytes should meet these criteria

Analyte	Low % Recovery	High % Recovery
N-Nitrosodimethylamine	60	140
Pyridine	60	150
Phenol	60	140
Aniline	4	140
Bis(2-Chloroethyl)Ether	60	140
2-Chlorophenol	60	140
1,3-Dichlorobenzene	60	140
1,4-Dichlorobenzene	60	140
Benzyl Alcohol	60	190
1,2-Dichlorobenzene	60	140
2-Methylphenol	60	140
bis(2-Chloroisopropyl)ether	60	140
3 & 4-Methylphenols	60	140
N-Nitrosodinpropylamine	60	140
Hexachloroethane	60	140
Nitrobenzene	60	140
Isophorone	60	140
2-Nitrophenol	60	150
2,4-Dimethylphenol	60	150
Benzoic Acid	50	190
Bis(2-Chloroethoxy)Methane	60	160
2,4-Dichlorophenol	60	150
1,2,4-Trichlorobenzene	60	140
Naphthalene	60	150
4-Chloroaniline	50	140
Hexachlorobutadiene	60	170
4-Chloro-3-Methylphenol	60	140
2-Methylnaphthalene	60	140
Hexachlorocyclopentadiene	60	190
2,4,6-Trichlorophenol	60	140
2,4,5-Trichlorphenol	60	140
2-Chloronaphthalene	60	140
2-Nitroaniline	60	140
Dimethylphthalate	60	140
2,6-Dinitrotoluene	60	140
Acenaphthylene	60	140
3-Nitroaniline	50	140
Acenaphthene	60	140
2,4-Dinitrophenol	60	180
4-Nitrophenol	60	140

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SVMS 2005
2nd Source Control Limits

Analysis Method: 8270, 625

Units: % Recovery

90% of target analytes should meet these criteria

Analyte	Low % Recovery	High % Recovery
2,4-Dinitrotoluene	60	140
Dibenzofuran	60	140
Diethylphthalate	60	140
Fluorene	60	140
4-Chlorophenyl-phenylether	60	140
4-Nitroaniline	50	140
2-Methyl-4,6-dinitrophenol	60	160
N-Nitrosodiphenylamine	60	140
Diphenylamine	60	140
1,2-Diphenylhydrazine	60	140
4-Bromophenyl-Phenylether	60	140
Hexachlorobenzene	60	150
Pentachlorophenol	60	140
Phenanthrene	60	140
Anthracene	60	140
Carbazole	30	130
Di-n-Butylphthalate	60	140
Fluoranthene	60	150
Benzidine	50	250
Pyrene	60	140
Butylbenzylphthalate	60	140
3,3'-Dichlorobenzidine	50	140
Benzo(a)Anthracene	60	140
Chrysene	60	140
bis(2-ethylhexyl)Phthalate	60	160
Di-n-Octylphthalate	60	150
Benzo(b)Fluoranthene	60	140
Benzo(k)Fluoranthene	60	150
Benzo(a)Pyrene	60	150
Indeno(1,2,3-cd)Pyrene	60	150
Dibenzo(a,h)anthracene	60	160
Benzo(g,h,i)Perylene	60	140

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APPENDIX H
Injection Log

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Injection Log

Directory: c:\hpchem\1\data\8060308

Line	Vial	FileName	Multiplier	SampleName	Misc Info	Injected
1	100	8030800.d	1.	DFTPP;1;;;DFTPP;;1000;;1000;;13FEB06		8 Mar 06 18:54
2	1	8030801.d	1.	BNA-50C;1;;5;;;1000;;1000;;BN275	07MAR06	8 Mar 06 19:17
3	2	8030802.d	1.	BNA-1.5;1;1;1;;;1000;;1000;;BN275	07MAR06	8 Mar 06 20:12
4	3	8030803.d	1.	BNA-3.0;1;1;2;;;1000;;1000;;BN275	07MAR06	8 Mar 06 21:00
5	4	8030804.d	1.	BNA-8.0;1;1;3;;;1000;;1000;;BN275	07MAR06	8 Mar 06 21:47
6	5	8030805.d	1.	BNA-16;1;1;4;;;1000;;1000;;BN275	07MAR06	8 Mar 06 22:34
7	1	8030801r.d	1.	BNA-50C;1;2;5;;;1000;;1000;;BN275	07MAR06	8 Mar 06 23:21
8	6	8030806.d	1.	BNA-70;1;1;6;;;1000;;1000;;BN275	07MAR06	9 Mar 06 00:08
9	7	8030807.d	1.	BNA-90;1;1;7;;;1000;;1000;;BN275	07MAR06	9 Mar 06 00:55
10	8	8030808.d	1.	BNA-120;1;1;8;;;1000;;1000;;BN275	07MAR06	9 Mar 06 01:42
11	9	8030809.d	1.	BNA-2ND;1;3;;LCS;;1000;;1000;;BN275	07MAR06	9 Mar 06 02:29
12	10	8030810.d	1.	BNZ+SUR-2ND;1;3;;LCS;;1000;;1000;;BN275	07MAR06	9 Mar 06 03:16
13	11	8030811.d	1.	MEGA-2ND;1;3;;LCS;;1000;;1000;;BN275	07MAR06	9 Mar 06 04:03
14	100	8030800a.d	1.	DFTPP;1;;;DFTPP;;1000;;1000;;13FEB06		9 Mar 06 04:44
15	1	8030801a.d	1.	BNA-50C;1;2;5;;;1000;;1000;;BN275	07MAR06	9 Mar 06 05:07
16	12	8030812a.d	1.	255050;1;3;;BLANK;;1000;;1000;;255050	EXT 7902/7903 AKA 255054	9 Mar 06 05:54
17	13	8030813a.d	1.	255051;1;3;;LCS;;1000;;1000;;255050	EXT 7902/7903 AKA 255055	9 Mar 06 06:41
18	14	8030814a.d	1.	9640873001;1;;;;;50;;1000;;255050	EXT 7902 JLG DUE 3/13	9 Mar 06 07:28
19	15	8030815a.d	1.	255052;1;3;;MS;;50;;1000;;255050	EXT 7902	9 Mar 06 08:15
20	16	8030816a.d	1.	255053;1;3;;MSD;;50;;1000;;255050	EXT 7902	9 Mar 06 09:02
21	17	8030817a.d	1.	254266;1;3;;BLANK;;50;;1000;;255050	EXT 7902	9 Mar 06 09:49
22	18	8030818a.d	1.	9641275001;1;;;;;200;;1000;;255050	EXT 7902 ARM/DSWA DUE3/16	9 Mar 06 10:36
23	19	8030819a.d	1.	9641275003;1;;;;;200;;1000;;255050	EXT 7902 ARM/DSWA DUE3/16	9 Mar 06 11:23
24	20	8030820a.d	1.	9641275005;1;;;;;200;;1000;;255050	EXT 7902 ARM/DSWA DUE3/16	9 Mar 06 12:10
25	21	8030821a.d	1.	9641275002;1;;;;;1050;;1000;;255050	EXT 7902 ARM/DSWA DUE3/16	9 Mar 06 12:56
26	22	8030822a.d	1.	9641275004;1;;;;;1050;;1000;;255050	EXT 7902 ARM/DSWA DUE3/16	9 Mar 06 13:43
27	23	8030823a.d	1.	9641313003;1;;;;;1000;;1000;;255084	EXT 7904	9 Mar 06 14:30
28	24	8030824a.d	1.	9641313002;1;;;;;1040;;1000;5;255084	EXT 7904	9 Mar 06 15:17
29	25	8030825a.d	1.	255086;1;3;;DUP;;1040;;1000;5;255084	EXT 7904	9 Mar 06 16:04
30	26	8030826a.d	1.	9641135002;1;;;;;1060;;1000;;255054	EXT 7903 WSI-CUMB DUE3/10	9 Mar 06 16:52
31	27	8030827a.d	1.	9641294001;1;;;;;1060;;1000;;255054	EXT 7903 MODERN L DUE3/13	9 Mar 06 17:38
32	28	8030828a.d	1.	9641309002;1;;;;;1050;;2000;;255054	EXT 7903 READING DUE3/13	9 Mar 06 18:25
33	29	8030829a.d	1.	9641071004;1;;;;;1040;;1000;;255054	EXT 7903 C. SYN TEC DUE3/15	9 Mar 06 19:13
34	30	8030830a.d	1.	9641071006;1;;;;;1040;;1000;;255054	EXT 7903 C. SYN TEC DUE3/15	9 Mar 06 19:59
35	31	8030831t.d	1.	.997NP;1;3;;LCS;;1040;;1000;5;08MAR06		9 Mar 06 20:47
36	32	8030832t.d	1.	4.985NP;1;3;;LCS;;1040;;1000;5;08MAR06		9 Mar 06 21:34
37	33	8030833t.d	1.	9.97NP;1;3;;LCS;;1040;;1000;5;08MAR06		9 Mar 06 22:20
38	34	8030834t.d	1.	EX30706-1;1;3;;LCS;;1040;;1000;5;08MAR0		9 Mar 06 23:07
39	35	8030835t.d	1.	EX30706-2;2;3;;LCS;;1040;;1000;5;08MAR0		9 Mar 06 23:54

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SOP Change History Summary

Section No.	Section	Reason for Change
Revision 5: 10/18/2005		
1.2	Scope and Application	Method update
5.0	Apparatus and Materials	NELAC requirement
6.0	Reagents	Method update
7.0	Instrument Calibration	Method update
12.2	Reporting Results	Method update
	Appendix E	Added for method update
	Appendix F	Added for method update
	Appendix G	Added for method update
	Appendix H	Added for method update
	Appendix I	Added for method update
	Appendix J	Added for method update
	Appendix K	Added for method update
	Appendix L	Added for method update
	Appendix M	Added for method update
	Appendix N	Added for method update
Revision 6: 03/14/2006		
1.1, 1.2, 1.3	Scope and Application	Updated method from 8270C to 8270D
1.2	Scope and Application	Added Heading to Compounds List, added footnote 1
1.4	Scope and Application	Replaced “special treatment” with “special considerations”
1.4.4	Scope and Application	Revision in wording for clarification

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SOP Change History Summary (continued)

Section No.	Section	Reason for Change
1.4.5	Scope and Application	Revision in wording for clarification; added verbiage to address 1.2-Diphenylhydrazine decomposition
1.4.7	Scope and Application	Added verbiage about late eluting losses
1.4.8-1.4.11	Scope and Application	Added sections
1.6	Scope and Application	Added verbiage about project requirements
2.1	Summary of Method	Included a range of 2-3 characteristic masses instead of 3
3.4	Interferences	Included reference to GPC cleanup
3.5	Interferences	Deleted section on base neutral extraction; Added verbiage about inlet liners and analytical sequences
4.2	Safety	Added availability of MSDS
5.1.2, 5.1.3, 5.6, 5.11	Apparatus and Materials	Updated list to include appropriate materials & vendor information
6.1	Reagents	Removed reference to “inorganic” chemicals
6.4	Reagents	Added location of certificates of analysis; revised holding temps
6.4.1-6.9.3	Reagents	Updated list to include appropriate reagents & vendor information
7.1	Instrument Calibration	Replaced “Instrument Tuning” with “Instrument Performance Check”
7.1.2.2	Instrument Calibration	Added verbiage to clarify background spectrum
7.2	Instrument Calibration	Added clarification to ICALs; added descriptive headers
7.2.2	Instrument Calibration	Deleted number of internal standards and reference to Appendix I
7.2.3	Instrument Calibration	Revised Appendix reference; added volume of calibration standards
7.2.4	Instrument Calibration	Revised # of runs from 7-8; added verbiage about calibration standard concentrations

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SOP Change History Summary (continued)

Section No.	Section	Reason for Change
7.2.5	Instrument Calibration	Deleted calibration volume reference
7.2.6	Instrument Calibration	Added DoD minimums
7.2.7	Instrument Calibration	Added note about dropping calibration levels
7.2.9.2	Instrument Calibration	Added reference to CCC compounds; added R-value reference; deleted verbiage from quadratic fit concerning intercept values closer to zero than linear fit; deleted reference to lack of guidelines in SW846
7.2.10	Instrument Calibration	Revised verbiage about linear and non-linear curve fits and added criteria guidance
7.2.11-7.2.13	Instrument Calibration	Major revisions
7.2.14, 8.9.4, 10.4.1.2	Instrument Calibration	Revised wording from “target compounds” to “analytes of interest”; expanded maintenance description
7.2.15-7.3.5	Instrument Calibration	Multiple revisions, including DoD requirements
8.2	Quality Control	Added verbiage for ongoing proficiency
8.3-8.4	Quality Control	Verbiage revised for clarification of calibration
8.5	Quality Control	Revised sections on initial demonstration of capability parameters
8.6	Quality Control	Multiple revisions to sections on Batch QC
8.7	Quality Control	Multiple revisions to sections on Sample QC
8.8.3	Quality Control	Added DoD control limits verbiage
8.10	Quality Control	Included MDL studies reference
9.1.1	Sample Collection…	Added verbiage for TO-13
9.2.1	Sample Collection…	Revised sample holding temperatures

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SOP Change History Summary (continued)

Section No.	Section	Reason for Change
9.2.2	Sample Collection…	Added sample type (water)
10.2	Procedure	Added requirement of acceptable initial instrument performance check and initial calibration
10.3.2	Procedure	Revised Appendix reference
10.3.3	Procedure	Replaced “m/z” with “compound”; “working” with “calibration”
10.3.5, 10.3.6	Procedure	Added sections
10.4.1	Procedure	Added a range of 1 or 2 secondary masses in lieu of 2
10.4.3	Procedure	Added Library Search analysis
10.5	Procedure	Added entire section on Quantitation
12	Reporting Results	Major revisions and additions to section
15.2-15.4	Definitions	Added 3 definitions
16	Troubleshooting	Added section
A	Appendix	Replaced with current list
B	Appendix	Revised to be certificates of analysis for primary stock solutions
C	Appendix	Revised to be preparation of DFTTP Standards
D	Appendix	Revised to be preparation of calibration standards
E	Appendix	Revised to be certificate of analysis of BNA spike
F	Appendix	Revised to be data acquisition
G	Appendix	Revised to be criteria for second source
H	Appendix	Revised to be injection log, deleted appendices I through N

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Analysis of Total Metals by Inductively Coupled Plasma Using the TJA Trace ICP

Document Control Number: _____

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1 Scope and Application

1.1 This method is adapted from the U.S. EPA SW846 Method 6010B Revision 2, December 1996, "Inductively Coupled Plasma - Atomic Emission Spectroscopy." This method is applicable to a wide variety of matrices including ground water, aqueous samples, TCLP, SPLP, ASTM extracts, industrial and organic wastes, soils, sludges, sediments, industrial hygiene paints, wipes, airs, and other solid wastes.

1.2 The specific elements which ALSI analyzes by this method are listed in Table 1. This table lists the wavelengths used for the appropriate elements.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of an ICP. Each analyst must also be skilled in the interpretation of raw data, including quality control data.

1.4 All samples are digested using appropriate sample preparation techniques.

1.5 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.6 Method Detection Limits can be found on the ALSI network in the Metals folder and are maintained and updated by the QA department. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

1.7 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 This method measures element-emitted light by optical spectrometry. The ICP instrument contains a torch through which flows argon gas. A spark is used to initiate a plasma of ionized argon, which is then maintained by a radio-frequency field. Samples are pulled into the system by a peristaltic pump and nebulized. The resulting aerosol is transported into the plasma torch. Element specific atomic- and ionic-line emission spectra are produced by the excited atoms or ions on their return to the ground state. The emission spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. The average of two intensity exposures, along with inter-element corrections yields the final result.

*(**Note:** For all sequence runs that involve samples from the Dept. of Defense (**DoD**), an average of three separate exposures is used to calculate the data.)*

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2.2 Background correction is used for all element determinations. Background intensity is measured adjacent to the analytical lines from the samples during analysis in an area free from spectral interferences. It's then subtracted from the intensity measured at these lines.

2.3 The possibility for additional interferences such as spectral, chemical, and physical interferences, does exist. Appropriate corrections for these interferences must be made.

3 Interferences

3.1 Spectral interferences are caused by (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background from continuum or recombination phenomena; and (4) stray light from the line emission of high concentration elements.

3.1.1 Background contribution and stray light are normally compensated for by the background correction.

3.1.2 Unresolved overlap requires the selection of an alternate wavelength or an alternate method of analysis such as graphite furnace or flame AA.

3.1.3 Spectral overlap is compensated for in the Trace instrument by automatic correction of the raw data after monitoring and measuring the interfering elements. A linear relationship between the interferant levels and the false interferences they cause can be assumed. Inter-element correction factors are checked daily by analysis of an interference check solution and updated every six months, or whenever indicated by failure of the interference check solution.

3.2 Physical interferences are effects associated with the sample introduction and flow through the instrument. These interferences are brought about due to differences in viscosity and surface tension. Physical interferences are most commonly seen in samples containing high dissolved solids or high acid concentrations. To reduce physical interferences, ALSI uses a peristaltic pump for sample introduction, dilutions of problem samples, and matrix matching of standards to samples. Internal standard addition and the method of standard additions may also be used to compensate for physical interferences.

3.2.1 Another problem that can occur while analyzing samples with high dissolved solids is salt build-up at the tip of the nebulizer, which affects aerosol flow rate and causes instrument drift. This problem is controlled by regular maintenance and by wetting the argon prior to nebulization using a humidifier.

3.3 Chemical interferences include molecular compound formation, ionization effects, and

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solute vaporization effects. Normally, these are not significant with the ICP. If they are encountered, they can be minimized by carefully selecting the operating conditions (RF power, torch position, and nebulizer flow rate), buffering the sample, matrix matching, and performing standard addition procedures. Chemical interferences are highly dependant on matrix type and the specific analyte of interest.

3.4 Memory Interferences result when analytes from a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. This problem can be minimized by flushing the system with rinse blank between samples. If a memory interference is suspected, the sample must be reanalyzed after a rinse period.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully defined; however, each chemical compound shall be treated as a potential health hazard.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available to all staff and are located in the lower level conference room in hard copy and electronically on the ALSI public file server: F:\MSDS - Material Safety Data Sheets.

4.3 Precautions shall be taken when handling samples and/or chemicals in the lab. The use of gloves, safety glasses, and lab coats is required when working with samples.

5 Apparatus and Materials

5.1 Thermo Jarrell Ash 61E Trace Simultaneous ICP Emission Spectrophotometer with the following options:

5.1.1 IBM compatible 386 computer
ThermoSPEC/AE v5.06 software
Axial Torch
Computer controlled emission with background correction.
Computer inter-element correction ability
Radio frequency generator coupled to a water-cooled induction coil
Water cooler
Vacuum Pump
Polychromatic optic system under vacuum
Adjustable variable speed peristaltic pump
Mass flow controllers for argon flow rate
HP LaserJet Printer
TJA 300 automatic liquid sampler
Argon humidifier

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5.1.2 Sample pump tubing - CPI, catalog #4062-535 or equivalent.

5.1.3 Internal Standard pump tubing - CPI, catalog #4062-5015 or equivalent.

5.1.4 Rinse pump tubing - CPI, catalog #4062-545 or equivalent.

5.1.5 13 x 100 polystyrene tubes - Perfector Scientific, catalog #2110 or equivalent.

5.1.6 Internal Standard Mixing Kit - CPI, catalog #4062-910 or equivalent.

5.2 Various Class A volumetric dispensing pipettes. – VWR, catalog #'s 53515-020, 53515-044, 53515-050 or equivalent.

5.3 Disposable Pasteur pipettes - VWR, catalog #14670-103 or equivalent.

5.4 Class A volumetric flasks (100 mL) – VWR, catalog # 29620-142 or equivalent.

6 Reagents

6.1 Concentrated Nitric Acid (HNO_3) - Baker Instra-analyzed Reagent Grade, VWR, catalog #JT9598-34 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date. If the manufacturer does not supply an expiration date, the reagent must be labeled as expiring one year from when opened. The analyst must write this date directly on the outside of the bottle using a permanent marker.

6.2 Concentrated Hydrochloric Acid (HCl) - Baker Analyzed Reagent Grade, VWR catalog #JT9535-33 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date. If the manufacturer does not supply an expiration date, the reagent must be labeled as expiring one year from when opened. The analyst must write this date directly on the outside of the bottle using a permanent marker.

6.3 Reagent water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification system, which provides analyte free, greater than 18.0 megohm-cm deionized water on demand. This water is used for preparation of all reagents and standards.

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6.4 Liquid Argon Supply - High purity grade, purchased from AIRGAS, or an equivalent supplier.

6.5 Stock Standard Solutions are purchased as commercially prepared NIST traceable certified solutions. When received in the lab each is assigned a unique log number and is recorded in the Standard Preparation Logbook along with the manufacturer, date of receipt, expiration date, and analyst's initials. These standards are stored at room temperature and disposed of on or before the manufacturer's expiration date.

6.5.1 SM-1339-002 (STD MIX #2) Stock Solution in 5% HNO_3/Trace HF. High Purity, or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Antimony (Sb)	20 mg/L
Bismuth (Bi)	100 mg/L
Boron (B)	100 mg/L
Cobalt (Co)	100 mg/L
Cooper (Cu)	100 mg/L
Iron (Fe)	100 mg/L
Manganese (Mn)	10 mg/L
Molybdenum (Mo)	100 mg/L
Nickel (Ni)	100 mg/L
Silver (Ag)	10 mg/L
Thallium (Tl)	10 mg/L
Tin (Sn)	10 mg/L
Titanium (Ti)	10 mg/L
Zinc (Zn)	50 mg/L

6.5.2 SM-1339-001 (STD MIX #1-R) Stock Solution in 5% HNO_3. High Purity Express, or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Aluminum (Al)	100 mg/L
Arsenic (As)	10 mg/L
Barium (Ba)	100 mg/L
Beryllium (Be)	20 mg/L
Calcium (Ca)	100 mg/L
Cadmium (Cd)	10 mg/L
Chromium (Cr)	10 mg/L
Lead (Pb)	10 mg/L
Magnesium (Mg)	100 mg/L
Potassium (K)	2000 mg/L
Selenium (Se)	100 mg/L
Sodium (Na)	2000 mg/L
Strontium (Sr)	10 mg/L
Vanadium (V)	5 mg/L

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6.5.3 QC26 Stock Solution in 5% HNO_3. QCD, catalog #QCS26K or equivalent. This standard contains the following elements:

ELEMENT	CONCENTRATION
Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn, Al, Ba, B, Si, Ag, Na, K	all at 100 mg/L

6.5.4 Strontium Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-382 or equivalent NIST certified standard.

6.5.5 Tin Stock Standard (1000 ppm) in 20% HCl. SCP, catalog #140-052-502 or equivalent NIST certified standard.

6.5.6 Bismuth Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-832 or equivalent NIST certified standard.

6.5.7 Scandium Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog #140-051-215 or equivalent NIST certified standard.

6.5.8 INTER18 (Solution A)in 5% HNO_3. CPI, Cat. #4400-INTR18-100 - Solution A, or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
K	20000 mg/L
Se	500 mg/L
As, Pb, Tl	1000 mg/L
Ba, Cd, Cr	300 mg/L
Co, Cu, Ni	
V, Zn	
Mn	200 mg/L
Be	100 mg/L
Hg	50 mg/L

6.5.9 INTR18 (Solution B) in 5% HNO_3. CPI, Cat. #4400-INTR18-100 – Solution B, or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Ag	300 mg/L

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6.5.10 INTER5 in 2% HNO_3. CPI, Cat. #4400-INTR5-500 or equivalent NIST certified standard. This standard contains the following elements:

ELEMENT	CONCENTRATION
Al	1200 mg/L
Ca	6000 mg/L
Fe	5000 mg/L
Mg	3000 mg/L
Na	1000 mg/L

6.5.11 Arsenic Stock Standard (1000 ppm) in 4% HNO_3. SCP, catalog # 7697-37-2 or equivalent NIST certified standard.

6.6 Working Standard Solutions. Prepare in an acid matrix similar to the samples being analyzed. This is most often a 10% HNO_3 matrix, but is dependent on the type of digestion performed on the samples, and shall be adjusted to match the samples being analyzed. After preparation, each standard is assigned a unique log number and is recorded in the standard preparation logbook along with the stock solution used, the concentration of that stock, the volume used, the final volume, the matrix, the date prepared, the date it will expire, and the preparer. Prior to analysis of **DoD** QSM samples, the following calibration standards (Sections 6.6.2 through 6.6.8) are to be prepared fresh prior to analysis. *(**Note:** The associated working standards are prepared daily, and held at room temperature. The maximum storage life is no longer than one day.)*

6.6.1 High Calibration Standard. To a 100 mL volumetric flask containing 10 mL HNO_3 (or that which matches the sample) in reagent water, add 5 mL SM-1339-002 (STD MIX #2)(Section 6.5.1) Stock Solution and 5 mL SM-1339-001 (STD MIX # 1-R)(Section 6.5.2) Stock Solution. Bring up to volume using reagent water. The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.2 Mid Calibration Standard. Add 4 mL of High Calibration Standard to 16 mL of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing cap on vessel and inverting several times. The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.3 Low Calibration Standard. Add 2 mL of High Calibration Standard to 18 mL of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing a cap on the vessel and inverting several times. The formulation of this standard

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must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.4 Calibration Blank (<detection limit (DL)). To 1000 mL volumetric flask, add 100 mL HNO_3 (or that which matches the samples) and bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. The calibration blank standard is stable for 3 months.

***(Note**: For all sequence runs that involve samples from the Dept. of Defense (**DoD**), the calibration reagent blank must have a concentration less than 2x the method detection limit for each analyte.*

Calibration Standard Concentrations (mg/L)

Element	High Cal. Std.	Mid Cal. Std.	Low Cal. Std.
Ag	0.5	0.10	0.05
Al	5.0	1.0	0.5
As	0.5	0.10	0.05
Ba	5.0	1.0	0.5
Be	1.0	0.20	0.10
Ca	5.0	1.0	0.5
Cd	0.5	0.10	0.05
Co	5.0	1.0	0.5
Cr	0.5	0.10	0.05
Cu	5.0	1.0	0.5
Fe	5.0	1.0	0.5
Mg	5.0	1.0	0.5
Mn	0.5	0.10	0.05
Mo	5.0	1.0	0.5
Ni	5.0	1.0	0.5
Pb	0.5	0.10	0.05
Se	5.0	1.0	0.5
Sn	0.5	0.10	0.05
Sr	0.5	0.10	0.05
Ti	0.5	0.10	0.05
Tl	0.5	0.10	0.05
V	0.25	0.05	0.025
Sb	1.0	0.20	0.10
Bi	5.0	1.0	0.5
B	5.0	1.0	0.5
Zn	2.5	0.50	0.25

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6.6.5 Working Profile Solution (5.0 mg/L). To a 1000 mL volumetric flask containing 100 mL HNO_3 (it is not critical for this standard to be matrix matched to the samples being analyzed) in reagent water, add 5 mL Arsenic Stock Standard (Section 6.13). Bring up to volume using reagent water. This profile solution must be documented in the metals Standard Reagent Logbook and transferred to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

6.6.6 Initial Calibration Verification Standard QC26 (1.0 mg/L). To a 100 mL volumetric flask containing 10 mL HNO_3 (or that which matches the sample) in reagent water, add 1 mL QC26 Stock Solution, 0.1 mL Strontium Stock Standard (Section 6.5.4), 0.1 mL Tin Stock Standard (Section 6.5.5), and 0.1 mL Bismuth Stock Standard (Section 6.5.6). Bring up to volume using reagent water. . The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.7 Working Interference Check Solution. To a 1000 mL volumetric flask containing 100 mL HNO_3 (or that which matches the sample) in reagent water, add 2.5 mL INTER18 Stock Solution A, 2.5 mL INTER18 Stock Solution B, and 25 mL INTER5 Stock Solution. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

ELEMENT	CONCENTRATION
Ag	0.75 mg/L
Al	30 mg/L
As	2.5 mg/L
Ba	0.75 mg/L
Be	0.25 mg/L
Ca	150 mg/L
Cd	0.75 mg/L
Co	0.75 mg/L
Cr	0.75 mg/L
Cu	0.75 mg/L
Fe	125 mg/L
Mg	75 mg/L
Mn	0.25 mg/L
Ni	0.75 mg/L
Tl	2.5 mg/L
V	0.75 mg/L
Zn	0.75 mg/L
Pb	2.5 mg/L
Se	1.25 mg/L
Na	25 mg/L
K	25 mg/L

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6.6.8 RPL Standard (CRI). To a 25 mL polyethylene analysis tube add 0.1 mL of RPL Stock Standard (Section 6.10). Using the 10 mL Finn pipette, add 19.9 mL 10% HNO_3 reagent blank (or that which matches the samples). This standard shall be prepared daily, and documented in the sample analysis logbook. The RPL / CRI concentrations are listed in Table 2.

6.7 Rinse Solution. To the rinse reservoir containing reagent water, add 300 mL HNO_3 (or that which matches the samples). Bring up to volume (3 L) using reagent water. Prepare as needed.

6.8 Working Internal Standard Solution (10.0 mg/L Sc). To a 2000 mL volumetric flask containing 40 mL HNO3 in reagent water, (or that which matches the samples), add 10 mL of Scandium Stock Solution (Section 6.5.7), and 50 grams of solid cesium chloride (Section 6.34). Bring up to volume using reagent water. Transfer to a labeled polyethylene jug. Working standard solutions are stable for 3 months when stored at room temperature.

6.9 Instrument Performance Check Solution (IPC). To a 25 mL polyethylene analysis tube add 10 mL of 10% HNO_3 reagent blank (or that which matches the sample), and 10 mL of High Calibration Standard (Section 6.6.1). This standard will need to be prepared throughout the analysis run depending upon how many samples are being analyzed. This Working Calibration standard solution needs to be prepared daily with every analysis calibration. This standard contains the following elements:

ELEMENT	CONCENTRATION
Ag	0.25 mg/L
Al	2.5 mg/L
As	0.25 mg/L
Ba	2.5 mg/L
Be	0.50 mg/L
Ca	2.5 mg/L
Cd	0.25 mg/L
Co	2.5 mg/L
Cr	0.25 mg/L
Cu	2.5 mg/L
Fe	2.5 mg/L
Mg	2.5 mg/L
Mn	0.25 mg/L
Mo	2.5 mg/L
Ni	2.5 mg/L
Pb	0.25 mg/L
Se	2.5 mg/L
Sn	0.25 mg/L

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ELEMENT	CONCENTRATION
Sr	0.25 mg/L
Ti	0.25 mg/L
Tl	0.25 mg/L
V	0.125 mg/L
Zn	1.25 mg/L
Sb	0.50 mg/L
Bi	2.5 mg/L
B	2.5 mg/L

6.10 Report Limit Stock Standard (RPL / CRI) is prepared by adding the following amounts of single element standard to a 200 mL volumetric flask containing 5 mL conc. HNO3. This working standard is stable for three months when stored at room temperature.

ELEMENT	Volume of 1000 ppm Single Element Solution in 200 mL.	Final Concentration (mg/L)
Aluminum	4.00 mL	20
Antimony	1.00 mL	5.0
Arsenic	0.40 mL	2.0
Barium	0.40 mL	2.0
Beryllium	0.16 mL	0.8
Bismuth	2.00 mL	10.0
Boron	4.00 mL	20.0
Cadmium	0.08 mL	0.40
Calcium	4.00 mL	20.0
Chromium	0.20 mL	1.0
Cobalt	0.20 mL	1.0
Copper	0.40mL	2.0
Iron	2.40mL	12.0
Lead	0.24 mL	1.2
Magnesium	4.00 mL	20.0
Manganese	0.20 mL	1.0
Molybdenum	0.80 mL	4.0
Selenium	0.80 mL	4.0
Silver	.16 mL	0.8
Nickel	0.80 mL	4.0
Tin	0.80 mL	4.0
Strontium	0.20 mL	1.0
Titanium	0.80 mL	4.0
Thallium	0.80 mL	4.0
Vanadium	0.20 mL	1.0
Zinc	0.80 mL	4.0

6.11 Silver Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-472 or

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equivalent NIST certified standard.

6.12 Aluminum Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-132 or equivalent NIST certified standard.

6.13 Arsenic Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-332 or equivalent NIST certified standard.

6.14 Barium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-562 or equivalent NIST certified standard.

6.15 Beryllium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-042 or equivalent NIST certified standard.

6.16 Calcium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-202 or equivalent NIST certified standard.

6.17 Cadmium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-482 or equivalent NIST certified standard.

6.18 Cobalt Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-272 or equivalent NIST certified standard.

6.19 Chromium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-242 or equivalent NIST certified standard.

6.20 Copper Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-292 or equivalent NIST certified standard.

6.21 Iron Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-262 or equivalent NIST certified standard.

6.22 Magnesium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-122 or equivalent NIST certified standard.

6.23 Manganese Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-252 or equivalent NIST certified standard.

6.24 Molybdenum Stock Standard (1000 ppm) in H_2O. SCP catalog #140-050-422 or equivalent NIST certified standard.

6.25 Nickel Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-282 or equivalent NIST certified standard.

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6.26 Lead Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-822 or equivalent NIST certified standard.

6.27 Selenium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-342 or equivalent NIST certified standard.

6.28 Titanium Stock Standard (1000 ppm) in H_2O/Trace HF. SCP catalog #140-050-222 or equivalent NIST certified standard.

6.29 Thallium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-812 or equivalent NIST certified standard.

6.30 Vanadium Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-232 or equivalent NIST certified standard.

6.31 Zinc Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-302 or equivalent NIST certified standard.

6.32 Antimony Stock Standard (1000 ppm) in 4% HNO_3. SCP catalog #140-051-512 or equivalent NIST certified standard.

6.33 Boron Stock Standard (1000 ppm) in H_2O. SCP catalog #140-050-052 or equivalent NIST certified standard.

6.34 Cesium Chloride (100%). VWR catalog #4042-02 or equivalent NIST certified standard.

7 Instrument Calibration

7.1 Immediately preceding calibration, each spectral line must be centered on its exit slit which is positioned in front of each photomultiplier tube. Maintaining this optical alignment during operation is called profiling.

7.1.1 Only the scandium spectral line is profiled. All other lines are preset relative to scandium.

7.1.2 To profile the instrument, aspirate the Working Profile Solution. From the main menu, enter Analysis. Enter the method name to be used and press Enter. Press F5, Profile. The profile line shall read Sc 361.384.

7.1.3 Press F3, Automatic. When the Working Profile solution has reached the plasma, press F1, Run. After scanning is complete, the scandium peak is displayed on the screen, along with the peak position, peak intensity and peak width.

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7.1.4 In order to get the peak position as close to the center as possible, the peak position number shall be as close to 0.00 ± .02 as possible. This is accomplished by choosing F1, calc SS, and pressing enter. The new vernier (Hg profile) position will be displayed on the screen. Adjust the vernier knob on the instrument manually to the value displayed on the screen. Then press F9, Done/Keep.

7.1.5 Go to Section 7.1.3 and repeat the process until the peak position is as close to 0.00 ± 0.02 as possible. Then press F9, Done/Keep and F9, Done. Allow the system to rinse for 5 minutes before starting calibration.

7.1.6 Re-profiling may be performed periodically throughout the analysis to compensate for diurnal changes, which are apparent by drifting QCs. It must only be adjusted, however, after instrument check standards and calibration blank have been analyzed.

7.2 The instrument prepares a standard curve by analyzing three levels of calibration standards and a calibration blank. Starting with the blank and working toward the high standard, the standards are aspirated and emission intensity readings are recorded by the data system.

7.3 All calibration standards are analyzed in duplicate and an average intensity is reported and used by the data system to prepare the calibration curve.
*(Note: All runs involving Dept. of defense (**DoD**) samples require three replicates for calibration standards.)*

7.4 A daily calibration curve is created by plotting the average intensity readings on the y-axis and concentration readings on the x-axis. The software of the data system plots the curve in a linear configuration. The calibration curve occurring most immediately preceding a particular sample is used to calculate the concentration for that sample. The acceptance criteria for a calibration curve for all analytes is a correlation coefficient of 0.995 or greater.

7.5 The calibration curve is validated using instrument check solutions prepared at known concentrations from a different source than that of the calibration standards. Validation occurs immediately following calibration and then at a frequency of 10% throughout the analysis run.

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8 Quality Control

8.1 Initial demonstration of performance

8.1.1 Linear Dynamic Range (LDR) - The upper limit of linearity must be established for each element being analyzed. Analyze successively higher concentrations of the analyte until the percent recovery falls under 90%. The last concentration maintaining greater or equal to 90% recovery is considered the upper limit of linearity. Samples containing analytes greater than 90% of the upper limit of linearity must be diluted and reanalyzed for those analytes. The LDRs are verified every six months or any time a change in operating conditions occurs that may change the LDR.
(***Note**: When analyzing samples from **DoD** (Dept. of Defense), the upper linearities must be verified in every analytical run. A verification standard can be analyzed either before or after the **DoD** samples in your analysis run. If the analyte recoveries are within 10% of their known value, the upper linear ranges can be used. Otherwise, all samples must be diluted to analyte concentrations, which fall within the calibration curve.)*

8.1.2 Method Detection Limits (MDL) - MDLs must be established and verified annually, and any time a change in operating conditions occurs that may change the MDL (reference 99-MDL). When analyzing Dept. of Defense (DoD) samples, MDL studies must be performed annually followed by an MDL Verification Check Standard analyzed at 2X the MDL. If The MDL verification standard is not recovered within at least 3X the instruments noise level, a new set of MDLs at a higher concentration will be analyzed. Once the MDL levels have been established, the Lower Quantitation Limits / Reporting Limits (RPL) can be set. The RPL shall be at least 3X the MDL.

8.1.3 Demonstration of Capability (DOC) – DOCs must be performed yearly by each analyst prior to performing this method and repeated at any time there is a significant change in instrument type (See: QA Plan, technical Training). To perform DOCs, four consecutive Laboratory Control Samples (LCS), with a matrix matching that of the calibration standards are analyzed. The recoveries obtained must be within 80-120% of the known values for each associated metal, and consecutive reads must have an RSD less than 20%. If the DOCs are outside these acceptance limits, a new calibration curve must be established, and the LCS's reanalyzed. This process is repeated until the DOCs are completed successfully.

8.1.4 Interelement correction factors must be verified and updated every six months or at any time a change in instrument operating conditions occurs which may change the interelement correction requirements. The IECs are calculated while performing the bi-yearly linear dynamic ranges (Section 8.1.1). When the upper

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limit for each analyte is established, the recovery effects on the remaining analytes must be updated. Any effect, whether positive or negative the analyte detection limit, must be corrected in the method IEC table. The IEC correction is calculated by dividing the affected analytes recovery by the concentration of the linear range standard being analyzed. The resulting correction must then be manually added or subtracted to the existing IEC. (ex.: An upper limit is established for Fe at 900 ppm. The Pb recovery is -0.006 ppm when Fe is 900 ppm. Since the Pb reporting limit is 0.005 ppm, the IEC is in need of correction. The -0.006 ppm result is divided by 900 yielding a correction factor of -0.000000666.)

8.1.5 Prior to performing analysis on NLLAP samples (IH), analysts will have read through the latest AALA lead requirements, and have demonstrated ability to produce reliable results through accurate analysis of standard reference material (i.e. PAT rounds or ELPAT studies), or in-house quality control samples. Their performance must be documented in their training logs.

8.1.6 Analysts/Technicians involved in IH Lead analysis shall demonstrate their ability to adequately analyze certified reference materials (i.e. PAT rounds or ELPAT studies), on a bi-yearly basis. Their performance must be documented in their training log.

8.1.7 Contamination Control – Lead dust wipe sampling must be performed in all associated areas of the lab on a quarterly basis to determine surface concentrations of lead. Sample preparation and analysis is not to proceed until surface contamination is less that the specified maximum allowable limit of 40 micrograms per square foot.

8.1.8 Instrument Detection Limit (IDL). The IDLs for the Trace will be performed Quarterly. This study is performed following a standard calibration, and includes analyzing ten calibration blanks on three non-consecutive days. Each measurement must be performed as though it were a separate analytical sample. (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples. The IDL responses are then manually entered off the raw data into an excel spreadsheet which then calculates the Standard Deviation. This number shall then be used as a starting point for setting up your MDL standards. (Note: the IDL limits shall be less than the MDL limits. If they are not, the IDL and MDL studies must be reviewed for errors, and redone if necessary.)

8.2 Daily demonstration of instrument performance

8.2.1 Quality Control Sample (QC26) - Initial and periodic verification of calibration standards is necessary to verify instrument performance. To verify the calibration

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standards, the Working Quality Control Sample QC26 must be within ± 10% of the true value immediately following the daily calibration. If outside of this acceptable range for target elements, the problem must be corrected by re-analysis, preparation, recalibration, or instrument maintenance. Samples may not be analyzed until the problem has been corrected and a QCS has been recovered within acceptable range.

8.2.2 A laboratory method blank (MB) is prepared with every batch of samples or one per every 20 samples digested, if the batch contains more than 20 samples. The method blank must be less than 1/10 of the concentration of any sample in the associated batch or less than 1/10 pf the specific regulatory limit (MCL) if the concentration exceeds the concentration of the sample. Otherwise, the source of the contamination must be investigated and measures taken to correct, minimize, or eliminate the problem. Any sample associated with a contaminated method blank shall be reprocessed or the results reported with an appropriate data qualifier.
***(Note**: The method blank result must be equal to or less than ½ the reporting limit for all sequence batches that include samples from the Dept. of Defense (**DoD**))*

8.2.2.1 IH Lead Wipe QC. The wipe media used for the quality control samples (i.e. MB / LCS / LCSD), shall be of the same lot number or manufacturer as the wipes used for sample collection. If the samples are collected by an outside source, the lab is requesting that extra wipe media is provided to perform the required QC. If the wipe media is not provided by the client, then ALSI will use in-house wipe media purchased from Environmental Express.

8.2.3 A laboratory fortified blank (LCS) is processed with every batch of samples, or one every 20 samples if the batch contains more than 20 samples. The LCS must be subjected to all sample preparation steps, such as digestion, if necessary. The percent recovery must be 80-120% of the true spike value. If the recovery falls outside of this range, the source of the problem shall be identified and resolved before continuing analyses. LCS results are documented in the ALSI LIMS system, which can produce Control Charts that will show method recovery trends over time.

8.2.3.1 To prepare a laboratory fortified blank while analyzing samples which do not require digestion, add 1 mL SM-1339-002 (STD MIX #2) (Section 6.5.1) Stock Solution and 1 mL SM-1339-001 (STD MIX # 1-R) (Section 6.5.2) Stock Solution to 100 mL of sample. This will result in a mid-level concentration for the LCS.

8.2.3.2 Calculating LCS Recoveries:

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% Recovery = (C_m/C_n) x 100
where: C_m = measured concentration of LCS
C_n = spiking concentration

8.2.4 Instrument Performance Check Solution (IPC) is analyzed following the calibration, after every 10 samples and at the end of the run. The results of the analytes in the check solution immediately following calibration must be within ± 10% of the true value with <5% RSD between replicates. Subsequent analyses of the IPC solution must be within ± 10% of the true value. If the result falls outside of this range for any element, the IPC may be rerun for that element. If the result still falls outside of this range, the problem must be corrected by re-profiling, preparation of new IPC, recalibration, or instrument maintenance. Samples following the last acceptable IPC requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the IPC. All results of IPCs are documented in the ALSI LIMS system.

8.2.5 The Working Interference Check Solution (ICS) is analyzed to provide an adequate test of the inter-element and background correction factors. It must be analyzed at the beginning and end of an analytical run or twice during every 8-hour shift, whichever is more frequent. The result must be within ± 20% of the true value. If the results fall outside of this range, the interference check solution may be rerun. If it is still outside this range for any element, the problem must be corrected by preparation of new working interference solution, recalibration, or adjustment of inter-element and background correction factors. Any sample requiring an element that fails must be rerun for that element after the problem had been corrected and the interference check successfully meets the criteria.

8.2.6 Calibration reagent blanks are analyzed directly after each IPC. The result for every element being analyzed must be less than 2.2 x the MDL. If the result does not meet this requirement for any element, the calibration blank may be rerun for that element. If the result is still not acceptable, the problem must be corrected by preparation of new blank, recalibration, or instrument maintenance. Samples following the last acceptable calibration reagent blank requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the calibration reagent blank. If reanalysis is not possible, data shall be reported with a qualifying statement.

Note: *For analysis runs involving samples from The Dept. of Defense* ***(DoD****), the calibration blanks must have analyte concentrations less than 2x the MDL. Otherwise, the source of the contamination must be investigated, and measures taken to minimize or eliminate the problem before the samples can be reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.*

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8.3 Daily demonstration of data quality

8.3.1 A matrix spike sample is processed at a frequency of 10% of the routine samples. Per EPA Method 6010 requirements the percent recovery must be 75-125% of the true spike value. Recovery calculations are not required if the concentration of the spike added is greater than ten times the sample background and a comment must be placed on the lab report. If the recovery falls outside of the acceptable range, and the system is found to be in control (Section 8.2), the recovery problem is judged to be matrix related and not system related. To determine if the method of standard additions is necessary, the sample must be post spiked or diluted as described below. Results of all matrix spikes are documented in the ALSI LIMS system.
*(**Note**: When analyzing samples from the Dept. of Defense (**DoD**) the matrix spike must be recovered at 80-120% of the true spiked value.)*

***% Recovery** = MS Conc. – Sample Conc. / Actual Spike Concentration x 100*

8.3.2 Analyte Addition Test. To prepare a post spike, add an amount of high standard that will produce a minimum level of 20x and a maximum level of 100x the MDL and analyze. The percent recovery must be 75-125% of the true value as determined by the following formula. If the result is outside of this range, make successive dilutions of the sample and re-spike until the recovery falls within this range. The reporting limit of the sample must then be raised to reflect the dilution used. If this raises the reporting limit higher than the client needs, the method of standard additions shall be performed. NOTE: If the analyte concentration is greater than ten times the matrix spike concentration, post-spikes are not required, but a comment must be added to the lab report stating "No Spike Calculated".

$$A = \frac{B - \left(\frac{E}{F}\right)(C)}{\left(\frac{E}{F}\right)(D)} \times 100$$

where:
A = Post spike percent recovery
B = Resulting spike concentration
C = Sample concentration
D = Working calibration standard concentration
E = Amount of spike added (mL)
F = Final volume spike solution + sample

*(**Note**: When analyzing samples from the Dept. of Defense (**DoD**) the post digestion spikes must be recovered at 80-120% of the true spiked value.)*

8.3.3 Dilution Test. If (100 x MDL) <20% of the sample concentration, prepare a 1/5 dilution on the sample and reanalyze. The resulting corrected concentration shall be within ± 10% of the original sample concentration. If not, a matrix effect shall

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be suspected, and the sample diluted until the matrix problem has been eliminated. If this raises the reporting limit higher than the client needs, the method of standard additions shall be performed.

8.3.4 A matrix spike duplicate or sample duplicate is processed at a frequency of 5% of the routine samples. The relative percent difference (RPD) must be within 20%. If the RPD falls outside of the acceptable range, and the system is found to be in control, the precision problem is judged to be matrix related and not system related. A comment must be added to the lab report. Results of all duplicate analyses are documented in the ALSI LIMS system.

RPD *= Difference / Average x 100*

8.4 Method of Standard Additions - The method of standard additions is used when sample dilution and spikes fail to produce good recoveries. In the standard addition technique, two identical aliquots (Volume Vx) of the sample solution are taken. To the first, (labeled A), is added a small a volume (Vs) of a standard analyte solution of concentration Cs. To the second (labeled B), is added the same volume Vs of the matrix blank. The intensity counts of A and B are measured and corrected for non-analyte intensity counts. The unknown sample concentration (Cx) is calculated as follows:

$$Cx = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where:

S_A = intensity counts of A corrected for the blank
S_B = intensity counts of B corrected for the blank

Vs and Cs shall be chosen so that S_A is roughly twice S_B on the average. It is best if Vs is much less than Vx, and thus Cs is much greater than Cx, to avoid excess dilution of the sample matrix. If a concentration or separation step is used, the additions are best made first and carried through the entire procedure. For results from this technique to be valid, the following limitations must be taken into consideration:

8.4.1 The analytical curve must be linear.

8.4.2 The chemical form of the analyte added must respond the same as the analyte in the sample.

8.4.3 The interference effect must be constant over the working range of concern.

8.4.4 The signal must be corrected for any additive interference.

8.5 Samples resulting in high negative (|conc.| > reporting limit) concentrations must be post-

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spiked (Section 8.3.2) to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.

8.6 When analyzing using a scandium internal standard, the intensity of scandium in each sample must be within ± 30% of the intensities in the initial calibration blank. If the intensities fall outside of this range, re-analyze the sample at a dilution to eliminate matrix interferences.

8.7 Each sample, QC check, and calibration standard is analyzed in duplicate and the results averaged. The relative standard deviation (RSD) between sample replicates must be less than 20% for all concentrations greater than the reporting limit to be accepted. If the RSD is greater than 20% and the sample concentration is above the reporting limit, the sample must be reanalyzed.
(Note: *All runs involving* ***DoD*** *samples require three replicates for standards, samples, and QC Checks.)*

8.8 It is recommended that whenever a new or unusual sample matrix is encountered, either of the following tests be performed to determine if either positive or negative matrix interferences are present to distort the accuracy of the reported values.

8.8.1 Serial dilution. If the analyte concentration is at least 40x the detection limit, a 1:4 dilution shall agree within ± 10% of the original determination. If not, a chemical or physical interference shall be suspected and must either be diluted out and the detection level raised or the sample may be analyzed by the method of standard addition.

8.8.2 Post digestion spike. A post digestion spike prepared as directed in Section 8.3.2, shall be recovered within 75 to 125% of the true value. If the spike is not recovered and the necessary sample dilution to recover the analyte concerned raises the detection limit of the sample above the limit needed by the client, the method of standard additions must be used.

8.9 ALSI participates regularly in applicable performance evaluation studies conducted by various certifying organizations.

8.10 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.11 Ongoing proficiency must be established annually as specified in the QA Plan.

Quality Control Requirements

(Specific Project Requirements may override these requirements)

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Parameter	Concentration	Frequency	Control Limits	Corrective Action
Calibration Reagent Blank	--	Beginning of run, after every 10 samples, and at the end of the run.	< 2.2 x MDL *< 2X the MDL for runs involving samples from the Dept. of Defense (**DoD**)*	Reanalyze the blank, prepare new blank and analyze, perform maintenance on instrument, recalibrate, reanalyze any samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying comment.
Method Blank (MB)	--	One per batch of no more than 20 samples. Analyze with associated sample batch.	<Reporting Limit *or < ½ reporting limit for **DoD** QSM samples*	Reanalyze the blank. Samples in the batch must be < the reporting limit or ≥ 10x the method blank. If not, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment. For **DoD** QSM samples the method blank must be ½ the reporting limit
High Calibration Standard	See Section 6.6.1	After calibration and before analysis of samples.	90-110%	Reanalyze the High Standard. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
*Laboratory Fortified Blank (LFB or LCS)	Listed in Table 3	One per batch of no more than 20 samples. Analyze with associated sample batch.	80-120%	Reanalyze the LFB. If still outside of acceptable range, samples must be re-digested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.
Quality Control Sample (QCS) Second Source Standard	1.0 mg/L	Immediately after calibration.	90-110%	Reanalyze the QCS. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
Instrument Performance Check Solution (IPC) Same Source	Listed in Table 4	Beginning of run, after every 10 samples, and at the end of the run.	90-110%	Reanalyze the IPC. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration. Reanalyze any samples since the last acceptable IPC. If reanalysis is not possible, report with a qualifying comment.
Reporting Limit Standard (RPL)	Listed in Table 2	Beginning of run, after calibration.	50-150% *Dept. of Defense samples (**DoD**)* *(80-120%)*	Reanalyze the RPL. If the standard is still not acceptable, perform instrument maintenance, prepare a new calibration, and reanalyze.

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Parameter	Concentration	Frequency	Control Limits	Corrective Action
* Matrix Spike (MS)	Listed in Table 3	One every 10 samples with at least one per batch.	75-125% *Dept. of Defense samples (**DoD**)* *80-120%* *50-150% for TCLP leachates*	Post Spike the sample for the failing analyte. If the post spike recovery is outside of the acceptable 75-125% limit, the sample will need to be analyzed at a 1:1 dilution and post spiked at a 1:1 dilution. If the post spike recovery is still outside acceptable limits, the sample will need to be further diluted and post spiked until an acceptable recovery has been reached. Report the results with a qualifying comment. If the matrix interference is identified, and the batch LCS was recovered within acceptable limits, report with a qualifying statement. (example: The sample in question contains % level iron.)
*Duplicate or matrix spike duplicate (MSD)	--	One every 10 samples with at least one per batch.	RPD ≤ 20%	Reanalyze the duplicate. If the RPD is still >20% or if reanalysis is not possible, report the results with a qualifying comment.
Sample replicates	--	Every sample	RSD < 20% for all samples > reporting limit	Reanalyze the sample. If the RSD is still > 20%, check to see if the relative percent difference of the four obtained exposures is less than 20%. If so, average the four exposures, and report the result with a qualifying comment. If the RPD is greater than 20%, reanalyze the sample a third time, and average all six exposures. Report the results with a qualifying comment.
Samples with high negative concentration	--	--	(|conc.| < reporting limit	Post-spike sample to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.
Scandium Internal Standard	--	Every sample	Intensity must be within 30% of the intensity of the initial calibration blank	Reanalyze the sample at the lowest possible dilution to eliminate sample matrix interference. The detection limits then need to be raised accordingly.

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Parameter	Concentration	Frequency	Control Limits	Corrective Action
Interference Check Solution	See Section 6.6.7	Beginning and end of every run, and at least once every 8 hours.	80-120%	Rerun the interference check solution, preparation of new solution, recalibrate, or adjustment of interelement correction factors. Rerun any samples requiring an element that fails, or if re-analysis is not possible, report the results with a qualifying comment.

* Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.
- LCS recoveries and duplicate precision limits stated in the QC Chart are also used for all IH analyses.

9 Sample Collection, Preservation and Handling

9.1 Sample Collection.

9.1.1 Samples must be collected in plastic or glass containers.

9.1.2 Soil/Sediment/Solid samples can be collected in plastic or glass containers.

9.1.3 A minimum of 3.00 g for soil samples, and 150 mL for water samples must be supplied by the sampler in order for the laboratory to perform analysis.

9.2 Sample Preservation

9.2.1 Preserve aqueous samples using HNO_3 to a pH<2. Sample preservation shall be performed immediately upon sample collection. If this is not possible, then samples shall be preserved ASAP when received by the laboratory.

9.3 Sample Handling

9.3.1 All samples must be analyzed within 180 days of collection. All samples not analyzed within this time frame must be discarded and re-sampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.

9.3.2 Soil samples must be preserved above the freezing point of water up to 6° C until analysis.

9.3.3 Water samples and sample digestates shall be stored at room temperature both

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before and after analysis.

9.3.4 All samples and digestates must be held by the laboratory for a minimum of two weeks after the lab reports have been sent to the client.

9.3.5 For samples requiring digestion, refer to the Sample Preparation SOPs for procedures.

10 Procedure

10.1 Initial Set-up and Analysis for the TJA Trace ICP

10.1.1 Perform the daily and as needed maintenance.

10.1.1.1 Refill the rinse solution.

10.1.1.2 Replace the sample and rinse pump tubing.

10.1.1.3. Inspect all other autosampler tubing for clogs and/or visible leaks.

10.1.1.4 Inspect the nebulizer for clogs and position in the spray chamber.

10.1.1.5 Inspect the spray chamber for cleanliness and clean if needed.

10.1.1.6 Set the incoming Argon pressure to 70 psi.

10.1.1.7 Clean the torch and tip if needed.

10.1.1.8 Adjust the tension on the pump tubing in the peristaltic pump.

10.1.1.9 Check the vacuum gauge. (This must read below 30 millitorr. If the pressure rises above 30, the vacuum pump oil must be changed.)

10.1.1.10 Check to be sure the water cooler is on and is filled to the proper level.

10.1.1.11 Check to be sure the hood is operating.

10.1.1.12 Empty the waste containers.

10.1.1.13 Check the reagent water level in the argon humidifier and refill if necessary.

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10.1.2 Turn the computer on and using the arrow keys, move to Plasma Control Panel under Set-up. Press Enter.

10.1.3 Press F1, Start-up and then press F9, continue. After purging for 90 sec. the torch will automatically be ignited. After the torch lights, press F2 from the menu, and scroll down to the pump speed / rate, and type in 130. Press Enter and then F9.

10.1.4 Press Esc. Use the arrow keys to move to Operation. Press Enter. After the Enter Method Name prompt, type in OPTIMIZE. The instrument will load and set the parameters, such as nebulizer pressure and pump speed, specified in this method. Put the probe of the autosampler in the rinse solution.

10.1.5 Allow the instrument to become thermally stable before beginning. This requires at least 30 minutes of running while aspirating rinse solution.

10.1.6 Profile the instrument using the Working Profile Solution as described in Section 7.1. When an acceptable profile is reached, print the screen by pressing Control F2, Print screen.

10.1.7 Leaving the probe in the profile solution, press F1, Analyze and F1, Run. The method will run 10 replicates of the Profile solution and print the %RSD. When an acceptable %RSD of less than 0.5 is achieved, print by pressing Control F2, Print Screen. Press F9, Done/Keep. You will now need to escape to the main menu and go to Analysis under the Operation tab. Next to the method command prompt type in SCICPHSB. This is the existing method set up for 6010B analysis.

*(**Note**: If samples from the Dept. of Defense (**DoD**) are being analyzed, you will need to type **DoD** method name.)*

10.1.8 The following parameters shall also be checked while still in Methods. When everything has been set correctly, save any changes made to the method by pressing F9, Done/Keep. After checking the method settings, hit F3 (Method Info). Scroll down to the Analysis File Date, and change it to the current days date. (NOTE: a zero cannot be entered as the first digit.) If more than analysis run is performed on the same day, the sequence date must be followed by a sequential lettering system. (ex. 11306, 11306A, 11306B……) After entering the analysis file date hit F9 (Keep/Done), then F8 (OPTIONS), then F2 (PrintMTD), then F9 (Done/Print), and finally F9 2x (Done/Keep).

Sample Introduction Device: Normal
Calibration Mode: Concentration

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Number of Repeats: 2 *(**3** Replicates are required for Dept. of Defense (**DoD**))*
Flush Time: 65.0 sec
Auto-Store Analysis Data? Yes
Auto-Store Stdzn Data? Yes
Store Individual Repeats? No
Autoprint Analysis Data? Yes
Autoprint Stdzn Report: None
Condensed Print Format? No

Output Mode: Concentration
Override Print Limits? Yes
Override Sig. Figures? No
Apply Background Correction? Yes
Apply Blank Subtraction? No

Torch gas: High Flow
Auxilary Gas Flow: Low (0.5 L/min)
Nebulizer Pressure: 25

Approximate RF Power: 950

Analysis Pump Rate: 150
Flush Pump Rate: 150
Relaxation Time (sec): 0
Pump Tubing Type: Tygon-Orange

10.1.9 After selecting, saving, and printing the required method, you will need to choose an autosampler table. Select OPERATION / Autosampler Setup from the main menu. Hit F8 (Filer), and select Autosampler Tables as a file type. You will need to scroll through the exiting files to find SC6010B. Place the cursor on SC6010B and hit F8 (COPY). Type in an autosampler name which matches the analysis file date you entered in (Section 10.1.8). Press Enter.

***(NOTE**: If samples from the Dept. of Defense (**DoD**) are being analyzed, you will need to use the DoD10 autosampler table.)*

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10.1.10 Use the arrow keys to move to Operation, Autosampler set-up to create a sequence table for the Autosampler. Press Enter, then F1Edit Set., then press F1, Edit Samples, and ALT F2. The autosampler table will already have default QC's and sample ID's. The default sample ID's will need to be changed to match the sample ID's on your horizon analysis batch. Immediately following the sample ID, the analysts' initials in parentheses need to be entered. (If the sample requires a dilution analysis, this dilution factor must be typed inside the parentheses after the analysts' initials.) Hit Enter 2X after entering the above information so that the cursor is in the third available field from the left. The horizon batch # off your analysis batch needs to be entered in this field. Immediately following this batch #, a T or D must be typed in. The T represents samples requiring a total metals analysis, and the D represents samples requiring a dissolved metals analysis. If this T or D is not entered properly, the automatic download of data in to horizon **will not** function. Build the table and save by pressing F9, Done/Keep three times. The computer will dictate the positions for the calibration blank and standards as well as the QCs that the analyst has put into the sequence table. Rinse and refill the calibration standards, blanks, and QC sample wells in the Autosampler and insert them in the correct positions. (NOTE: The correct autosampler positions can be viewed while in the autosampler edit mode)

10.1.11 Go into Operation off the main menu screen, and select Analysis. Enter the method name. Press Enter. Press F9, Autosampler. Enter the name of the Autosampler table to be used (Section 10.1.10). Press F1, Run. The instrument will, with the use of the Autosampler and peristaltic pump, start to standardize the instrument.

10.2 Analyze by the method of standard addition (Section 8.4) any samples containing matrix interferences which cannot be eliminated by dilution.

10.3 For the inter-element spectral interference correction factors to remain valid, the interferant concentration must not exceed its limit of linearity. Sample dilution is necessary in these cases, and the reporting limit must be raised to reflect the dilution performed.

11 Calculations

11.1 Sample results are reported directly from the readout of the instrument (from the calibration curve), and input into the LIMS. Appropriate prep factors are applied to the result at the time of supervisor approval.

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11.2 Any sample result requiring dilution to bring the sample into the linear range, is multiplied by the dilution factor before being entered into the LIMS using the following equation:

$$A = \frac{Z\,(B)}{C}$$

where A = concentration of element in sample
Z = concentration of element in diluted sample
B = final volume of dilution (mL)
C = volume of sample aliquot used in dilution (mL)

12 Reporting Results

12.1 Report results in the Horizon LIMS system: The TJA Trace 61E ICP is set up to automatically download analysis results in to a system called NuGenesis. The NuGenesis system reads the raw data and compiles it into a form that the Horizon LIMS system will understand. The NuGenesis files can be accessed by logging onto horizon and selecting "Autopost pipe" under OPERATIONS. A box labeled Advanced Find will appear. You must enter the Queue "META", and enter the analysis batch # off your batch sheet. The sample IDs that will then appear in NuGenesis, will match the sample IDs that you typed in the autosampler table (Section 10.1.10) Therefore, if the sample ID was incorrectly entered in the autosampler table, it will be wrong in NuGenesis as well. Scroll down through the samples in NuGenesis, and make sure the results match your raw data. Once you have checked for errors, hit the AUTOPOST tab and select all.

12.2 Checking results in Horizon LIMS system: Once sample results have been autoposted from NuGenesis, you must enter the OPERATIONS tab and select POSTING-by Worklist. Enter the HBN (Horizon Batch Number) from the analysis batch sheet. The analyst must go through this work list and double check to make sure that all results autoposted correctly.

12.3 Choosing sample Condition Codes in Horizon LIMS system: The column to the right of the sample name is CC, which stands for Condition Code. The CC will read OK if Horizon received data from NuGenesis. If any sample in the analysis run required an additional dilution, or some kind of rerun while analyzing, it will appear more than once in your analysis batch. If the results were left as is, the lab report would have duplicate results. Therefore, you must change the condition code on the sample so that you can determine which analytes are taken from which run. There are three CC's that can be used in this case. They are **LR, LX**, and **RP**. The **LR** condition code is to be used only if an analyte could not be completed in the current analysis run. (ex.: Ag did not pass in your calibration QC). The **LX** condition code is to be used if a sample was analyzed more than once in the same run, and all reruns have been completed. (ex.: A sample had a high RSD for Pb, and it was successfully reanalyzed later in the same run.) The **RP** condition code is to be used if the digestion QC associated with the sample did not pass

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method criteria, and needs to be sent back up to the prep Dept. to be redigested. (ex.: The LCS failed high for cadmium, and the sample in question required a cadmium analysis).

12.4 Choosing analyte Condition Codes in Horizon LIMS system: After one of the condition codes listed in Section 12.3 has been entered next to the sample, another condition code needs to be placed after the individual analytes in order for the analyst to be able to determine which analytes are used or not used. The three available condition codes are: **MO**, **NO**, and **RP**. The **MO** condition code is used for analyte results that you want to be taken from the current sample. The **NO** condition code is used for analytes that you do not want to be taken from the current sample. The **RP** condition code is used for analytes that need to be re-digested by the prep dept. (ex.: A sample is analyzed for cadmium, silver, and zinc. The cadmium result was fine, the silver result was no good because the digestion method blank failed for silver, and zinc was over your linear range and requires an additional dilution. In this case cadmium would be MO, silver would be RP, and zinc would be NO.)

12.5 All raw data used for reporting results must dated and initialed by the qualified laboratory personnel performing the first and second reviews. Use the Trace Run Case Narrative sheets located next to the PC to record this information.

12.6 If you need to hand enter results into Horizon due to problems with the automatic download, be sure not to round off results. Horizon will automatically perform rounding appropriate to the method. The actual result needs to be typed into horizon, even if it is less than the reporting limit. Any sample with a result less than the reporting limit will automatically be reported as ND (non-detect) by Horizon.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees.

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ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 The ICP will often have carryover issues with silver that are confusing and difficult to remedy. Due to the chemical properties of silver, the analyte will build up inside the sample lines in the form of silver chloride. When a higher acid matrix is introduced to the system, the silver will sometimes fall back into solution, and show up as false positives. Usually the problem can be mediated by running some 20% HCL solution through the system for several minutes. Other times, the analyst will need to shut down, and clean the entire system. (NOTE: If the 20% HCL solution is run through the system for an extended period of time, the sample lines will develop a white haze. It is best to change out the lines when this occurs.)

16.2 Communication Errors or Power Outage. Often after power outages or periods of time when the computer has not been restarted for awhile, the Trace software will develop communication errors. If this occurs, the instrument and PC will need to be completely shut down and restarted. To shut down: extinguish the torch and exit out of TJA software, turn off the computer, turn off the one inch wide power strip located on the back of the instrument, turn off the red one inch wide switch on the front of the voltage regulator (small floor unit next to the wall.) Wait two to three minutes and then check to see if the vacuum pump is on by pressing the black start button located on the vacuum control system panel. Turn on the PC, voltage regulator, and instrument power switches. On the front of the ICP press the black RESET button located in the controller panel. Press the red RESET button on the back of the ICP. Then once again press the black RESET button on the controller panel. If the problems are still present, repeat this process. If this process is ineffective, technical support may need to be requested from Thermo Elemental.

16.3 Monitoring instrument trends for requesting service. By monitoring the peak intensities obtained from your As Optimization, the Optimization average recoveries, and the Hg profile throughout your analytical runs, you can often tell when service will soon be needed. The instruments RF tuning is checked annually during the PM Service; however, it is not uncommon for the tuning to require further adjustment throughout the year. By recording the information listed above in your analysis logbook, a trend will be more easily monitored. If a definite trend is detected, a service call to Thermo Elemental shall be placed. The reason for the service call must be documented in the maintenance

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logbook. Following the field service, an additional note shall be made that shows that the problem was corrected, and what was done to correct it.

16.4 Recording instrumentation problems in the maintenance logbook. Any daily service or issue involving the instrument must be noted in the maintenance logbook. The logbook is then a good source to refer to if problems develop. Usually the current issue with the instrument has occurred sometime in the past. The analyst can then look to see what if any corrective actions were performed.

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APPENDIX A

Trace ICP Analytical Worksheet

Acid Matrix: ____% HNO3 + ____% HCl

Instrument Method:_____
Sequence #:_____
Date Started:_____
Analyst:_____

*High Std:_____ml each of _____+_____ into _____ml Pipette ID #_____
*Mid Std:_____ml High Std into _____ml ICS:_____
*Low Std:_____ml High Std into _____ml RPL:_____ml_____into_____ml
*CCV/IPC:_____ml each of _____+_____ into _____ml
*Second Source:_____ml of _____+ Pipette ID #_____
_____ml each of _____+_____+_____ into _____ml
*Additional Second Source:_____ml of _____+_____ into _____ml
*Cal Blank:_____ml HNO3 Lot #:_____ HCl Lot #:_____ Pipette ID #_____

* All Standard are prepared daily prior to analysis of samples in acid matrix listed.

PDS: 3.5 ml sample * 3.5 ml High Std

BLANK		
LOW STD		
MID STD		71
HIGH STD		72
SECOND SOURCE	31	73
	32	74
	33	75
	34	76
	35	77
	36	78
	37	79
	38	80
	39	
	40	
		81
		82
	41	83
	42	84
1	43	85
2	44	86
3	45	87
4	46	88
5	47	89
6	48	90
7	49	
8	50	
9		
10		
		91
		92
	51	93
	52	94
11	53	95
12	54	96
13	55	97
14	56	98
15	57	99
16	58	100
17	59	
18	60	
19		
20		
		101
		102
	61	103
	62	104
21	63	105
22	64	106
23	65	107

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TABLE 1
(Element Wavelengths)

Element	Wavelength
Ag	328.068
Al	308.215
As	189.042
B	249.678
Ba	493.409
Be	313.042
Bi	223.061
Ca	317.933
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
Fe	271.441
Mg	202.030
Mo	202.030
Mn	257.610
Ni	231.604
Pb	220.353
Sb	206.838
Se	361.384
Sn	189.989
Sr	421.552
Ti	334.941
Tl	190.864
V	292.402
Zn	213.856

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TABLE 2

(Reporting Limit Concentrations)

Element	Concentration
Ag	0.004
Al	0.10
As	0.010
B	0.10
Ba	0.010
Be	0.004
Bi	0.05
Ca	0.10
Cd	0.002
Co	0.005
Cr	0.005
Cu	0.01
Fe	0.06
Mg	0.10
Mo	0.02
Mn	0.005
Ni	0.02
Pb	0.005
Sb	0.02
Se	0.02
Sn	0.02
Sr	0.005
Ti	0.02
Tl	0.02
V	0.005
Zn	0.02

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TABLE 3
(Laboratory Fortified Blank and Matrix Spike Concentrations)

Element	Concentration (mg/L)
Ag	0.100
Al	1.00
As	0.100
B	1.00
Ba	1.00
Be	0.200
Bi	1.00
Ca	1.00
Cd	0.100
Co	1.00
Cr	0.100
Cu	1.00
Fe	1.00
Mg	0.100
Mo	1.00
Mn	0.100
Ni	1.00
Pb	0.100
Sb	0.20
Se	1.00
Sn	0.10
Sr	0.10
Ti	0.10
Tl	0.10
V	0.050
Zn	0.50

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TABLE 4
(Interference Check Solution Concentrations)

Element	Concentration
Ag	0.75
Al	30.0
As	2.50
Ba	0.75
Be	0.25
Ca	150
Cd	0.75
Co	0.75
Cr	0.75
Cu	0.75
Fe	125
Mg	75.0
Mn	0.50
Ni	0.75
Pb	2.50
Se	1.25
Tl	2.50
V	0.75
Zn	0.75

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TABLE 5
(Matrix Spike / Matrix Spike Duplicate Concentrations for TCLP and SPLP leachates)

Element	Concentration
Ag	0.90
As	0.90
Ba	9.00
Cd	0.90
Cr	4.50
Pb	4.50
Se	4.50

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SOP Change History Sheet

Section No.	Section	Reason for Change
Revision 10: 02/24/2005		
2	Summary of Method	U.S. Army Corp. audit response
6.1	Reagents	SOP Update
6.2	Reagents	SOP Update
6.5.1-6.5.2	Reagents	SOP Update
6.5.10	Reagents	SOP Update
6.6.4	Reagents	U.S. Army Corp. audit response
6.6.5	Reagents	SOP Update
6.6.6	Reagents	SOP Update
6.6.7	Reagents	SOP Update
6.6.8	Reagents	SOP Update
6.8-6.10	Reagents	SOP Update
6.9-6.11	Reagents	SOP Update
7.3	Instrument Calibration	SOP Update
7.4	Instrument Calibration	SOP Update
8.1.1	Quality Control	U.S. Army Corp. audit response
8.1.3	Quality Control	SOP Update
8.1.5	Quality Control	SOP Update
8.1.6	Quality Control	SOP Update
8.1.7	Quality Control	SOP Update
8.2.2	Quality Control	U.S. Army Corp. audit response
8.2.2.1	Quality Control	SOP Update

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
8.3.1	Quality Control	SOP Update
8.3.4	Quality Control	SOP Update
8.7	Quality Control	SOP Update
8.12	Quality Control	Section Removed
9.3.2-9.3.4	Sample Handling	SOP Update
10.1.8	Procedure	U.S. Army Corp. audit response
12.1	Reporting Results	SOP Update
12.2 / 12.3	Reporting Results	Sections Removed
	Appendix A (Analytical Worksheet)	U.S. Army Corp. audit response
	Table 2 (RPL Concentrations)	SOP Update
	Table 3 (LCS / MS Concentrations)	SOP Update
	Table 4 (Interference Check Concentrations)	SOP Update

Revision 11: 01/26/2006

1.1	Scope and Application	Matrices update
1.7	Scope and Application	Project requirements note
2	Summary of Method	DoD audit response
3.1.3	Interferences	Frequency change instituted for correction factors
3.4	Interferences	SOP update from Method 4.2
4.2	Safety	Directive to read MSDS sheets and their availability
5.2 & 5.4	Apparatus and Materials	SOP materials update

SOP Change History Sheet (continued)

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Section No.	Section	Reason for Change
6.1 & 6.2	Reagents	Expiration date and labeling
6.5.8	Reagents	Addition of "Solution A"
6.5.9	Reagents	Addition of "Solution B"
6.6.1	Reagents	Reagent source change, documentation and hold time additions
6.6.2	Reagents	Documentation and hold time additions
6.6.3	Reagents	Documentation and hold time additions
6.6.4	Reagents	DoD audit response
6.6.5	Reagents	Documentation statement
6.6.6	Reagents	Documentation and hold time additions
6.6.7	Reagents	Preparation revision
6.6.8	Reagent	Preparation revision
6.9	Reagents	Preparation revision
6.10	Reagents	Volume & concentration change
6.34	Reagents	Section added
7.1.1	Instrument Calibration	Scandium replaces arsenic
7.1.2	Instrument Calibration	Profile line change
7.1.3	Instrument Calibration	Scandium replaces arsenic
7.1.4	Instrument Calibration	DoD audit response
7.1.5	Instrument Calibration	DoD audit response
7.3	Instrument Calibration	DoD audit response
8.1.1	Quality Control	DoD audit response

SOP Change History Sheet (continued)

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Section No.	Section	Reason for Change
8.1.2	Quality Control	DoD audit response
8.1.3	Quality Control	Reference to QA Plan
8.1.4	Quality Control	Instructions added for IECs
8.1.8	Quality Control	DoD audit response
8.2.3	Quality Control	LCS replaces LFB
8.2.3.1	Quality Control	Change in Stock Solution
8.2.3.2	Quality Control	Section Removed
8.2.2	Quality Control	DoD audit response
8.2.6	Quality Control	DoD audit response
8.3.1	Quality Control	DoD audit response
8.3.2	Quality Control	DoD audit response
Quality Control Requirements (Calibration Blanks)		DoD audit response
Quality Control Requirements (RPL)		DoD Audit Response
Quality Control Requirements (Matrix Spike)		SOP Update
Quality Control Requirements (Sample Replicates)		SOP Update
8.6	Quality Control	Scandium intensity adjusted from ± 20% to ± 30%
10	Procedure (throughout section)	Additional instrument directions
12	Reporting Results	Major revisions throughout
16	Troubleshooting	DoD audit response

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
Revision 12: 02/13/2007		
1.6	Scope and Application	Updated MDL file location/maintenance
4.2	Safety	Updated MSDS file locations
6.4	Reagents	Updated vendor information
6.8	Reagents	Revised Working Internal Standard Solution concentration
6.10	Reagents	Revised volumes of single element solution for some elements
8.11	Quality Control	Deleted terminology “reprofile and/or” from corrective action for High Cal Stnd, QCS, and ICP; reworded RPL corrective action and revised control limits; revised Scandium Internal Standard corrective action for greater detail; revised Internal Check Solution frequency
2	Table	Revised some of the element reporting limit concentrations

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Mercury by Cold-Vapor Atomic Absorption Using an Automated Continuous-Flow Vapor Generator

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Anna Milliken Date
Laboratory Operations Manager

_____ _____
Natalie Hufford Date
Validator

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1.1 This document states the policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This method is adapted from EPA Method 245.1, Revision 3.0, May 1994; SW-846 Method 7470A, Mercury in Liquid Waste, September 1994 Revision 1; and, Method 7471B, Mercury in Solid or Semisolid Waste, January 1998 Revision 2; Method 7000 Atomic Absorption Methods, July 1992, Revision 1

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of cold vapor analysis. Each analyst must also be skilled in the interpretation of raw data, including quality control data.

1.4 This method measures total mercury (organic-inorganic) in drinking, surface, saline, and ground waters, domestic and industrial wastes, and mobility-procedure extracts. It also applies to soils, sediments, bottom deposits, and sludge-type materials.

1.5 In addition to inorganic forms of Mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system.

1.6 All samples must be digested prior to analysis.

1.7 Method Detection Limits can be found in the metals department method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.8 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 The flameless AA procedure is a physical method based on the absorption of radiation

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at 253.7 nm by mercury vapor. The samples/standards and reagents are pumped into the analyzer and mixed. Argon gas is introduced into the solution stream, which flows to a mixing coil where the samples and reagents are thoroughly combined in the mixing coil. The gas and liquid stream is transferred to the gas/liquid separator where the gas and liquid phases are separated. The liquid waste is drained off and the gas is pumped to the absorption cell. The absorption cell is positioned in the light path of the mercury lamp. Absorbance (peak height) is measured as a function of mercury concentration and recorded as ppb of mercury.

3 Interferences

3.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL). During the oxidation step, chlorides are converted to free chlorine, which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

3.4 Interference from certain volatile organic materials which will absorb at this wavelength is also possible. All positive samples must be checked for false increases due to organics by analysis without the addition of stannous chloride.

4 Safety

4.1 Operation of an atomic absorption spectrophotometer involves the use of argon gas and hazardous materials including corrosive fluids. Unskilled, improper, and careless use of equipment can create explosion hazards, fire hazards or other hazards, which can cause death, serious injury to personnel, or severe damage to equipment or property.

4.2 Caution shall be taken when handling all samples, standards, and QC material because of the acidic nature of the prepared samples as well as the possible mercury content in the samples.

4.3 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

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4.4 Proper personal protective equipment must be used, including gloves, safety glasses, and lab coat.

4.5 The fume hood must be turned on during the analysis of mercury to vent the waste vapor.

5 Apparatus and Materials

5.1 PSA Automated Mercury Analyzer

5.1.1 CETAC M-6100 Mercury Analyzer.

5.1.2 PSA Automated Mercury Analyzer.

5.2 Green-Green Santoprene tubing: P S Analytical, cat. #M025T002

5.3 Yellow-Yellow Santoprene tubing: CETAC SP5705A

5.4 Black-Black Santoprene tubing: CETAC SP5705B

5.5 Finnpipette with disposable tips: 2 mL – 10 mL VWR, cat# 53516-178 or equivalent
0.2 mL – 1 mL VWR, cat #53515-876 or equivalent
20 µL – 200 µL VWR, cat#53503-094 or equivalent

5.6 Various Class A volumetric glassware.

5.7 Various calibrated dispensers.

5.8 40 mL VOA vials: Industrial Glassware #2795FL-PC.

5.9 25 mL graduated cylinder: Class A.

5.10 8 mL polystyrene tubes: VWR 60818-849 or equivalent

5.11 14 mL polypropylene tubes: VWR #60818-618 or equivalent.

6 Reagents

6.1 Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System, which provides analyte-free DI water greater than 16.0 megohm on demand. This water is

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used for preparation of all reagents, calibration standards, and as dilution water.

6.2 Liquid Argon: high purity grade, MG Industries or equivalent.

6.3 Stannous Chloride: Prepare by adding 100 g of stannous chloride crystal (VWR, catalog #EM-SX0885-1 or equivalent) to a 1000 mL volumetric flask. Add 14.0 mL conc. H_2SO_4 and stir until dissolved. Bring up to volume with reagent water. Stored at room temperature. Maximum storage life for stannous chloride is approximately 12 hours.

6.4 Sulfuric Acid, conc. Baker Instra-analyzed grade: VWR, cat. #JT9673-33 or equivalent. Store at room temperature up to the manufacturer's expiration date.

6.5 Sodium Chloride (NaCl.) Baker Instra-analyzed grade: VWR, cat. #JT3625-15 or equivalent. Store at room temperature up to the manufacturer's expiration date.

6.6 Hydroxylamine hydrochloride decolorizing reagent: To prepare, dissolve 120 g Hydroxylamine hydrochloride crystals (VWR, cat. #JT2196-1 or equivalent) and 120 g NaCl in reagent water in a 1000 mL volumetric flask. Bring up to volume using reagent water. Stored at room temperature. Maximum storage life is 3 months from date of preparation. The amount used for decolorizing is dependent upon the type of analysis being performed. Water samples require 1.5 mL and solid sample require 6.0 mL.

7 Instrument Calibration

7.1 The instrument plots a standard calibration curve each day of use using five standards and a blank. The calibration standards for EPA Method 245.1 and 7470 are Blank, 0.2 µg/L, 1.0 µg/L, 2.0 µg/L, 4.0 µg/L, and 10.0 µg/L. The calibration standards for EPA Method 7471 are Blank, 0.5 µg/L, 1.0 µg/L, 2.0 µg/L, 4.0 µg/L, and 10.0 µg/L. The calibration standards used for low level requests are 0.050 µg/L, 0.075 µg/L, 0.100 µg/L, 0.200 µg/L, and 0.500 µg/L. Starting with the blank and working toward the high standard, the standards are introduced into the mercury analyzer by the autosampler. Absorbance readings are recorded by the data system.

7.2 A calibration curve is drawn by plotting the absorbance readings on the y-axis and concentration readings on the x-axis. The software of the data system plots the curve. The calibration curve is used to calculate the concentration for the samples. The correlation coefficient must be 0.995 or greater.

7.3 A set of calibration standards is prepared along with every batch of mercury samples digested. It is these standards, which must be used to prepare the calibration curve for that batch of samples.

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7.3.1 This is especially important because Method 245.1, Method 7470 and Method 7471 batches are prepared differently. Therefore these standards shall never be interchanged.

7.4 An Initial Calibration Verification (ICV) must be analyzed after every calibration to verify the instrument performance during analysis. The ICV is prepared from the second source standard. Analysis of the ICV immediately following calibration must verify that the instrument is within ± 5% of calibration for EPA Method 245.1, and ±10% for EPA Methods 7470/7471. Subsequent analysis of this standard is called the continuing calibration verification standard (CCV) and must be within ±10% of calibration. If outside of this range, determine and correct the problem. If necessary, recalibrate. Samples may not be analyzed until an acceptable ICV/CCV is analyzed.

7.5 Laboratory Control Sample (LCS). A digested standard must be analyzed with each batch and after every calibration. It is prepared at 2.0 ppb from the same source as that of the calibration standards. The recovery must be within ± 15% of the true value for the calibration. If outside of this range, determine and correct the problem and re-analyze. If necessary, recalibrate. Samples may not be analyzed until an acceptable LCS is analyzed

7.6 If the calibration blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

7.6.1 If the calibration blank concentration is greater than or equal to 2x the MDL, the source must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement. **(DoD Requirements)**

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 A demonstration of capability shall be performed before any client samples are analyzed. The DOC is determined by the analysis of four different Laboratory Control Samples (LCS). The percent recovery of the four LCSs must be ± 15% and the percent RPD must be ≤ 10%. If demonstration of capability fails, reanalyze. Ongoing proficiency must be established annually as specified in the QA plan, Technical Training.

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Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Acceptance Criteria	Corrective Action
Calibration Blank (ICB/CCB)	NA	Prepared with each batch of samples. Analyzed after every ICV/CCV, at a minimum frequency of 10% and after calibration.	< MDL	Re-analyzed the blank. If still out of range, the problem must be solved by preparing a new blank, recalibration, or instrument maintenance. Samples following the last acceptable blank must be rerun.
Method Blank	NA	One digested with each batch of 20 or less samples. They are analyzed with that batch of samples.	1/2 the Reporting Limit	Re-analyze the blank. The samples in the prep batch must be less than the reporting limit or greater than 10X the reagent blank value for the affected analyte. It not, the affected samples in that batch must be re-digested. If re-digestion is not possible, they will be reported with a qualifying comment.
Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)	Water: 2.0 ug/L Soil: 100 ug/kg Low detection waters: 0.070 ug/L	One digested with each batch of 20 or less samples. They are analyzed with that batch of samples.	85-115% R As required by the Method	Re-analyze the LCS. If the recovery is still outside the given range, the source of the problem must be identified and corrected before continuing analyses. If the problem cannot be identified, the samples in that batch must be re-digested. If re-digestion is not possible, report with a qualifying comment.
Matrix Spike (MS)*	Water: 5.0 ug/L Soil: 250 ug/kg Low detection waters: 0.200 ug/L	Frequency of 10% per matrix per batch	80-120% R As required by the Method	Re-analyze the MS. If still out of range analyze a post digestion spike (85-115%). If still out of range, a qualifying comment on the final lab report.
Parameter	**Concentration**	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Matrix Spike Duplicate (MSD) or Duplicate (Dup)*	Water: 5.0 ug/L Soil: 250 ug/kg Low detection waters: 0.200 ug/L	Frequency of 10% (**DoD samples** - 100% frequency)	<20% RPD As required by the Method	Re-analyze the duplicate. If the sample is outside the range, redigest the sample. If still outside of acceptable limits, report with a comment on the lab report.

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Initial Calibration Verification Standard (ICV) (Second Source)	4.0 ug/L Low detection waters: 0.100 ug/L	Immediately after calibration.	Immediately after calibration ± 5%R (245.1); ± 10%R (7470/7471).	Re-analyze the ICV. If still out of range, the problem must be identified and corrected before analyzing any samples. Any samples analyzed after the last acceptable ICV/CCV must be re-analyzed.
Continuing Calibration Verification Standard (CCV) (Same Source)	4.00 ug/L for Methods 245.1 & 7470 and 4.0 ug/L used for Method 7471 and Low detection waters: 0.100 ug/L	Immediately after calibration, after every ten samples, and after the last sample.	Immediately after calibration ± 5%R (245.1); ± 10%R (7470/7471). Thereafter it must be within ± 10%R.	Re-analyze the ICV. If still out of range, the problem must be identified and corrected before analyzing any samples. Any samples analyzed after the last acceptable ICV/CCV must be re-analyzed.

* Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

Note: See Section 11.4 for % recovery calculations.

8.3 For samples with results greater than the highest standard will be diluted and reanalyzed until the concentrations are within the calibration range.

8.4 Method detection limits are determined annually using the procedure outlined in the ALSI Quality Assurance Plan. NOTE: If **DoD samples** are to be analyzed, an MDL check sample will be used to verify the MDL. The MDL check sample is at a concentration equal to 2 x the MDL. If a positive response is detected from the MDL check sample, another MDL study is not needed for that calendar year. Theses studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

8.4.1 Practical Quantitation Limits (PQL) or reporting limits are determined by multiplying the MDL by 3-5 times, and adding an appropriate safety factor.

8.5 If the matrix spike fails criteria, a post digestion spike is performed. If the recovery of the post digestion spike is within 85-115%, the results will be reported. If outside of this range, comment on the final report. If the LCS is acceptable and the specific matrix interference is identified, report with a qualifying statement. If the specific matrix interference is unknown, reanalyze the sample and matrix spike to determine matrix effect or analytical error.

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8.6 If the method blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

8.6.1 If the method blank concentration is greater than or equal to ½ the reporting limit AND is greater than 1/10 the sample concentration, the source of the contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement. **(DoD Requirements)**

9 Sample Collection, Preservation and Handling

9.1 Sample Collection:

9.1.1 Samples can be collected in any size plastic or glass bottles. Minimum amount required for analysis is 100 mL.

9.1.2 Aqueous samples requiring dissolved metals shall be filtered immediately on site before adding preservation for dissolved metals.

9.2 Sample Preservation:

9.2.1 Preserve aqueous samples using HNO_3 to a pH <2. Sample preservation shall be performed immediately upon sample collection. If this is not possible, then samples would be preserved as soon as possible when received at the laboratory. If samples are unpreserved in the laboratory, analysis shall not take place for 16 hours.

9.3 Sample Handling:

9.3.1 All samples must be analyzed within 28 days of collection. All samples not analyzed within this time frame must be discarded and resampled for analysis.

9.3.2 All samples require digestion. Refer to the Sample Preparation SOP for procedures.

9.3.3 All samples and sample digestates shall be stored at room temperature, separated from standards.

10 Procedure

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10.1 For P S Analytical Merlin Millennium.

10.1.1 Verify the Argon pressure is set at 40 psi and inspect the tubing. If it has flattened change all four lines and clamp.

10.1.2 Open the Avalon Software and name the folder using the date on appropriate matrix: W for waters, S for soils and LL for low detection waters.

10.1.3 Under the OPTIONS heading select SERVICE OPTIONS and check the Disable dryer gas check and also Disable the analysis gas.

10.1.4 Under the METHODS heading select ALSIW and click OK, if analyzing the low detection samples set the range to 10. Verify that the lines are drawing liquid by using harmless food coloring and adjust the pump clamps to set the proper tension. Allow the instrument to warm up for at least 15 minutes.

10.1.5 Fill the Stannous Chloride reservoir with 10% Stannous Chloride and insert the probe into it. Allow this to flush the system for 10 minutes.

10.1.6 Under the OPTIONS heading select MANUAL CONTROL. Adjust the reference output between 107-110 using the screw on the instrument labeled R. Adjust the Trans Loss by setting the range to 10 and using the screw on the instrument labeled TL set this between 160-190.

10.1.7 Under the CALBRATION heading select NEW CURVE and set the standards and select analyze. To each standard add Hyrdroxylamine hydrochloride. Shake until color disappears and vent. Load into spaces 1 to 6 on the autosampler. In the instrument software set the concentrations that are being used, click OK and verify that the settings are correct and click OK.

10.1.8 After the calibration is complete and acceptable, (r=0.995 or greater) under the PROGRAM heading choose the program editor and OPEN the correct autosampler table.

10.1.9 To each sample add Hydroxylamine hydrochloride. Shake until color disappears and vent. Load the samples into the autosampler tray. To begin the analysis of the samples, under the PROGRAM heading choose RUN and click OK to begin the analysis.

10.1.10 After analysis is complete, any sample that has a result above the reporting limit (0.0005 mg/L for 245.1/7470; 0.001 mg/L for SPLP/7471; 0.006 mg/L for TCLP; 0.0002 mg/L for 245.1 Low and 0.00007 for 245.1 X-Low) must be rerun without stannous chloride to determine if an organic interference is

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present.

10.1.10.1 If the stannous chloride result is greater than the reporting limit, subtract the non-stannous chloride result to get the final mercury concentration.

10.2 For the CETAC M-6100 Mercury Analyzer

10.2.1 Start the Quick Trace M-6100 software (if the software was left in standby, open the M-6100 controls and start the autosampler by selecting PARK - to start the rinse cycle.)

10.2.2 Turn on the lamp and start the gas flow (40 psi). A minimum of 15 minutes is needed.

10.2.3 Inspect the sample tubing and replace if needed clamp the lines. Verify that the capillary is 0.5 mm above the Gas/liquid separator center post.

10.2.4 Wet the Gas/liquid separator (GLS) center post. Disconnect the "11→Hg Vapor→12" from the GLS vapor outlet. Set the gas pressure to 60 psi. Place the reagent line into a beaker containing DI water. Turn on the pump and release the tension on the bottom two lines (drain lines). Allow the liquid level to rise so that the gas will bubble and wet the ENTIRE post of the GLS. Once this happens re-clamp the lines, set the gas pressure to 40 psi and allow the GLS to drain. When the GLS has drained reconnect the "11→Hg Vapor→12" and place the reagent line into 10% Stannous Chloride.

10.2.5 Open the proper worksheet and using SAVE AS name it using the date followed by W for waters; S for soils; LL for low detection

10.2.6 At this point, add Hydroxylamine hydrochloride to each standard shake until color disappears and vent. Load the standards and begin the calibration. Once the calibration is complete and acceptable, (r=0.995 or greater) sample analysis can begin. Decolorize the samples using Hydroxylamine hydrochloride shake until color disappears and vent. Load the samples into the autosampler tray for analysis

10.2.7 After analysis is complete, any sample that has a result above the reporting limit (0.0005 mg/L for 245.1/7470; 0.001 mg/L for SPLP/7471; 0.006 mg/L for TCLP; 0.0002 mg/L for 245.1 Low and 0.00007 for 245.1 X-Low) must be rerun without stannous chloride to determine if an organic interference is present.

10.2.7.1 If the stannous chloride result is greater than the reporting limit,

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subtract the non-stannous chloride result to get the final mercury concentration.

11 Calculations

11.1 Samples results are documented directly form the readout of the instrument in ppb (from the calibration curve).

11.2 The results are converted to ppm and input into the LIMS system.

11.3 Samples requiring dilution at the time of analysis to bring the result into calibration range are multiplied by the dilution factor used before inputting into the LIM system using the following equation:

$$A = \frac{Z\,(B)}{C}$$

where: A= Concentration of mercury in the sample
B= Final volume of the dilution (mL)
Z= Concentration of mercury in the dilution
C= Volume of sample aliquot used in the dilution

11.4 **LCS Recovery**

% recovery = (Cm/Cn)x100
Cm = measured concentration of LCS
Cn = Spiking concentration

Spike Recovery

% recovery = [(Cs-Cu)/Cn]x100
Cs = measured concentration of spiked sample aliquot
Cu = measured concentration of unspiked sample aliquot
Cn = spiking concentration

Precision (RPD)

$$\% \text{ RPD} = \frac{|(R1-R2)|}{(R1+R2)/2} \times 100$$

R1 = sample or spike result
R2 = duplicate or spike duplicate result

12 Reporting Results

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12.1 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.2 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

12.3 Report the actual result, even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit.

12.4 Reporting Results (DEP Reportable Samples)

12.4.1 DEP samples exceeding the Maximum Contaminant Level (MCL) must be reported to the Customer Service Representative immediately following determination in order to comply with the Pennsylvania Code; Title 25, Chapter 109, Section 109.810 for Reporting and Notification Requirements.

12.5 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

12.6 The following must be done to upload data into the Horizon LIMS system. It is instrument specific.

12.6.1 For data from the CETAC M-6100:

Select the REPORTS icon from the instrument software and choose the file to be uploaded. Next select EXPORT FILE. This file is to be exported to the CETAC folder located on the Desktop. Name the file so that it corresponds to the actual run and save. It is now in an EXCEL spreadsheet and shall be in the following format so that uploading can occur *SAMPLE NUMBER*SAMPLE TYPE*BATCH NUMBER*ANALYST INITIALS*DILUTION* (This is only needed if it is something other than 1.) Once all samples have this format the following shall be done to complete the process. Open the MERCURY_CETAC file and click the button called 'import.csv file'. Select the file name to be uploaded from the CETAC DATA folder on the desktop. The reports will print to NuGenesis and can be posted in the Horizon LIMS.

12.6.2 For data from the P S Analytical:

Select the FILE option and select SAVE AS TEXT. Name the file so that it corresponds to the actual run .txt (example: results.txt). Under the drivers select e:\\wmmet001\psadata. Open the spreadsheet named PSA and click

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'import .txt file' Select the file name to be uploaded from the PSA data folder on the Desktop. The data shall be in the following format so that uploading can occur *SAMPLE NUMBER*SAMPLE TYPE*BATCH NUMBER*ANALYST INITALS*DILUTION* (This is only needed if it is something other than 1) Once all the samples have this format the following shall be done to complete the process. Click the 'send to NuGenesis' button. The reports will print to NuGenesis and can be posted in the Horizon LIMS.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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Appendix A – CETAC

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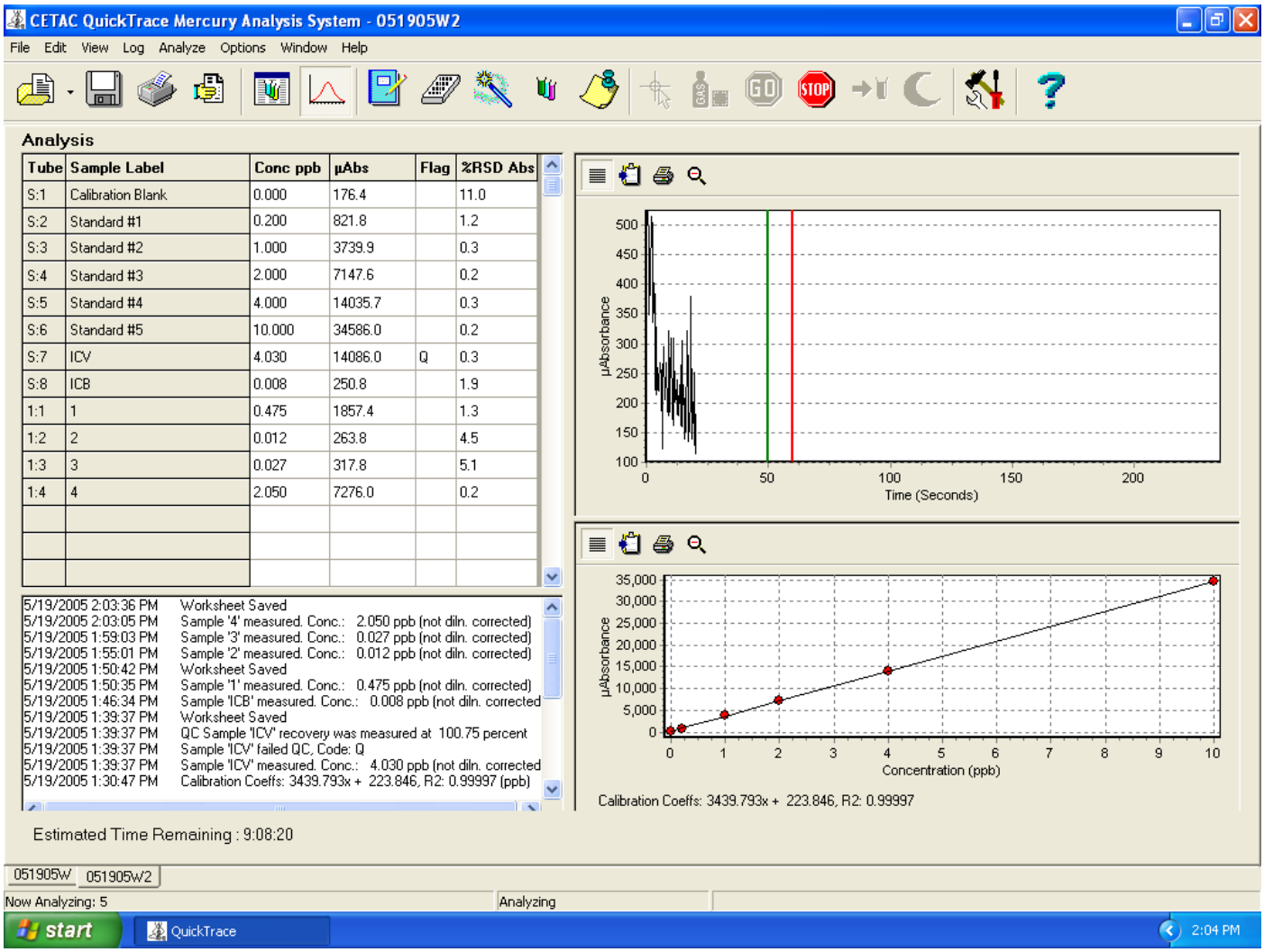

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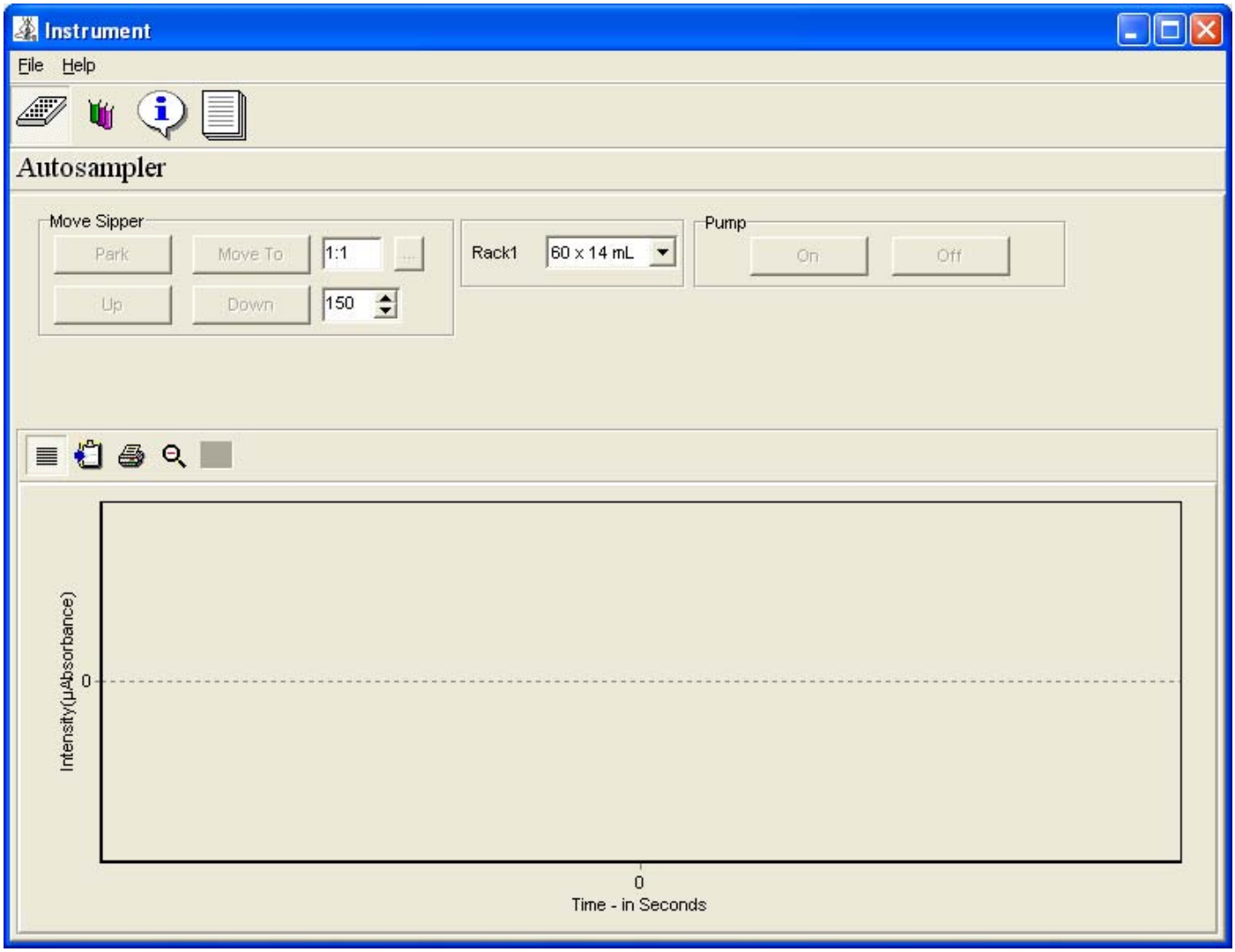

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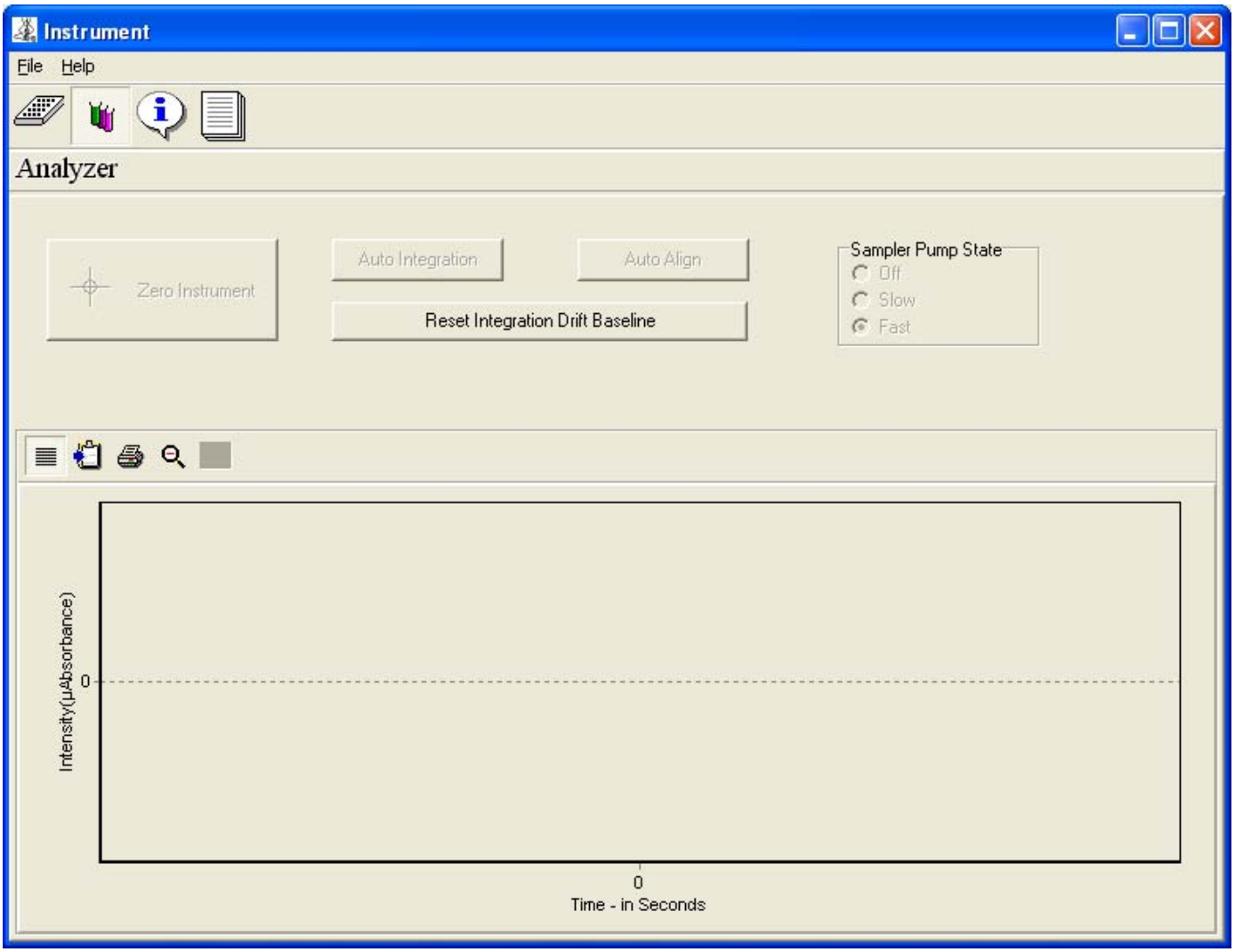

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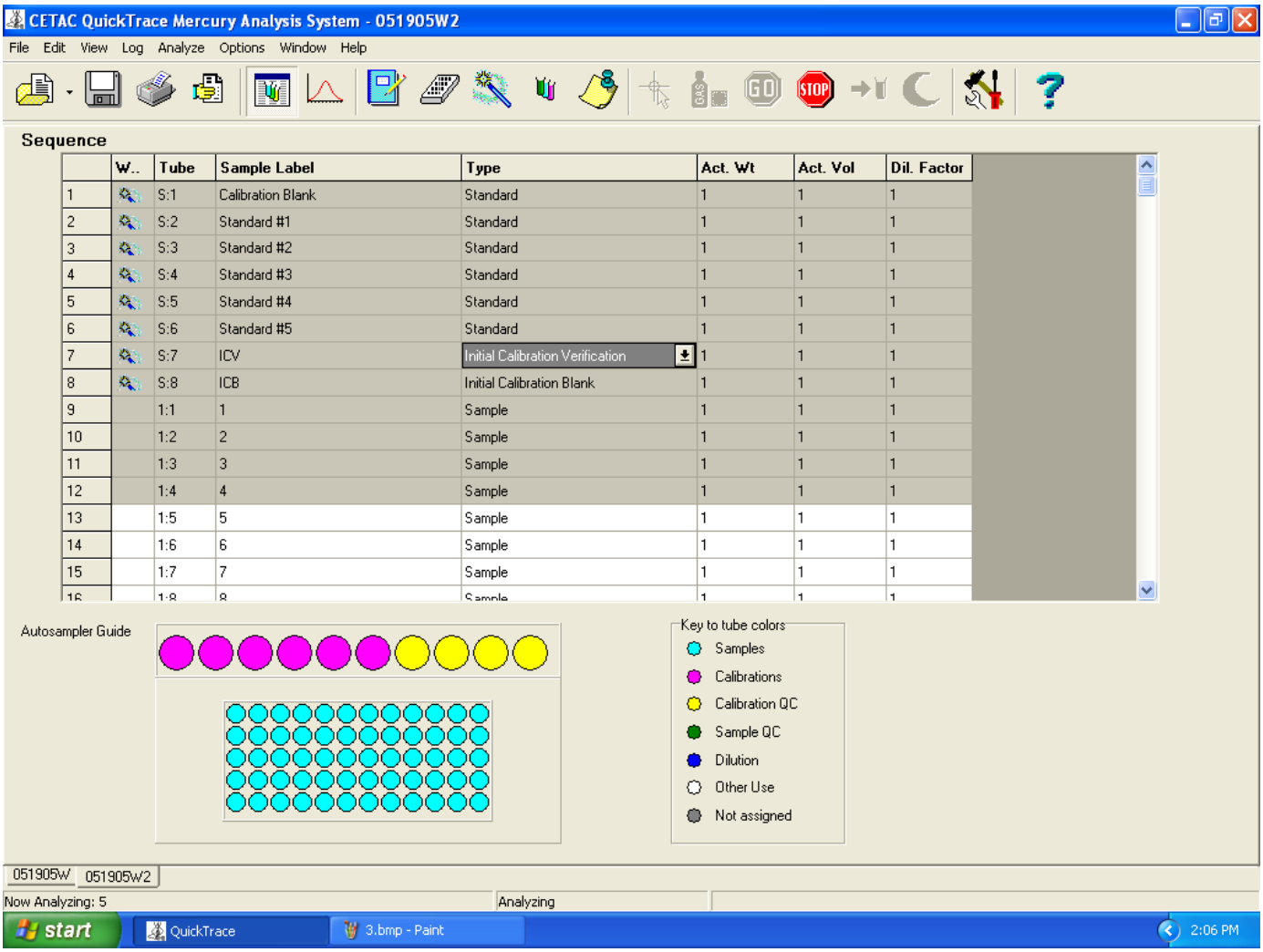

	W..	Tube	Sample Label	Type	Act. Wt	Act. Vol	Dil. Factor
1		S:1	Calibration Blank	Standard	1	1	1
2		S:2	Standard #1	Standard	1	1	1
3		S:3	Standard #2	Standard	1	1	1
4		S:4	Standard #3	Standard	1	1	1
5		S:5	Standard #4	Standard	1	1	1
6		S:6	Standard #5	Standard	1	1	1
7		S:7	ICV	Initial Calibration Verification	1	1	1
8		S:8	ICB	Initial Calibration Blank	1	1	1
9		1:1	1	Sample	1	1	1
10		1:2	2	Sample	1	1	1
11		1:3	3	Sample	1	1	1
12		1:4	4	Sample	1	1	1
13		1:5	5	Sample	1	1	1
14		1:6	6	Sample	1	1	1
15		1:7	7	Sample	1	1	1
16		1:8	8	Sample	1	1	1

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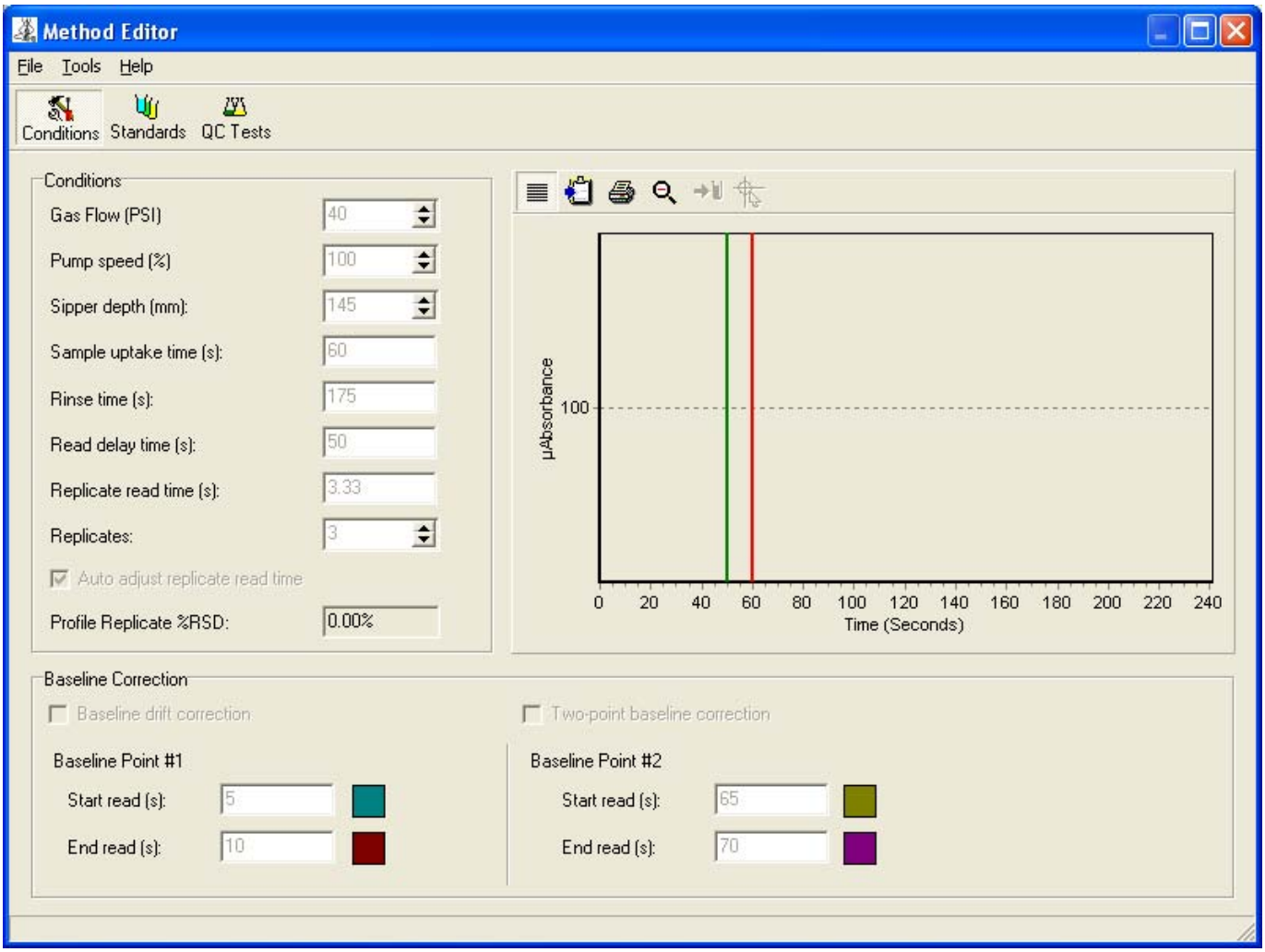

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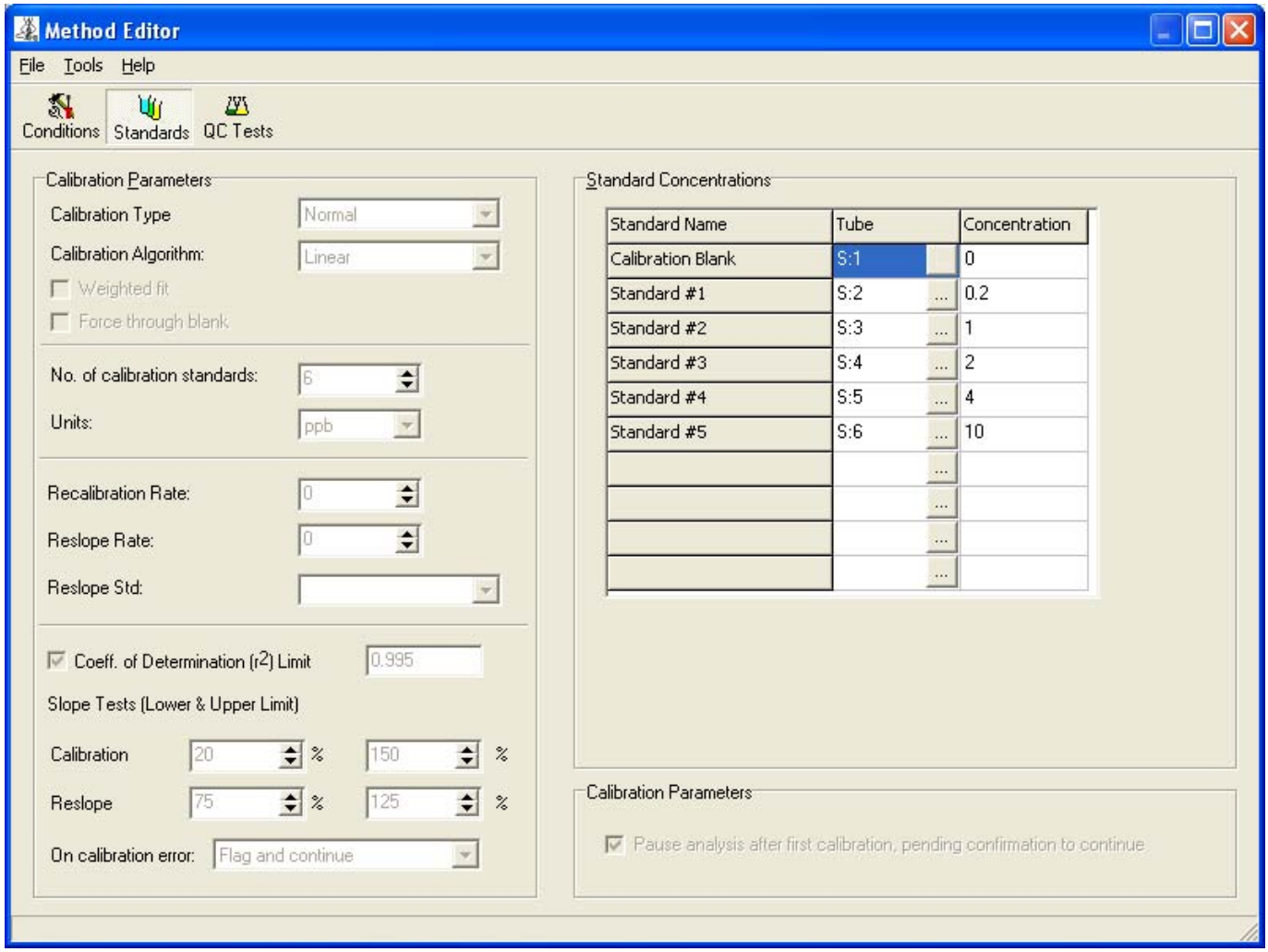

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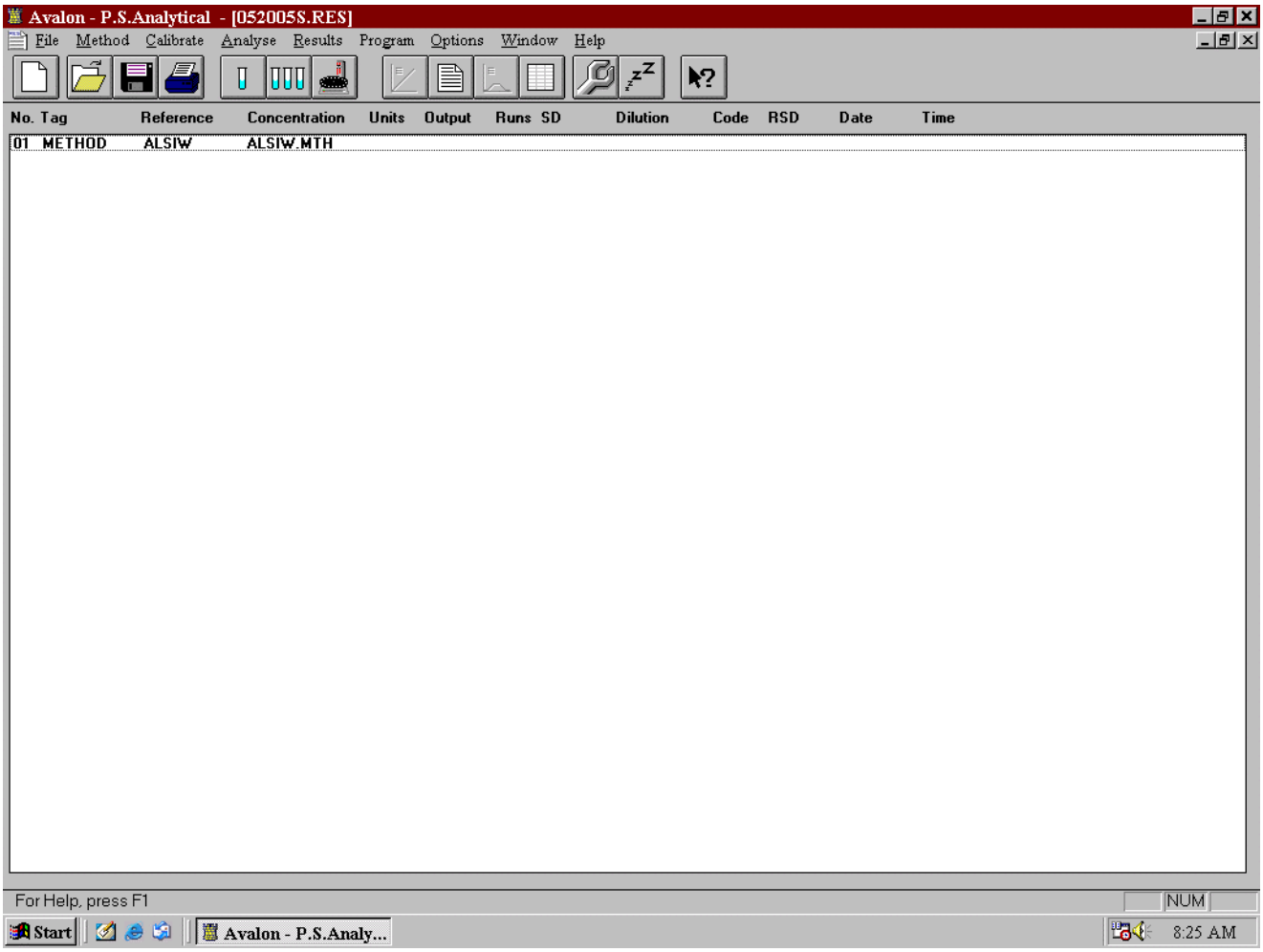

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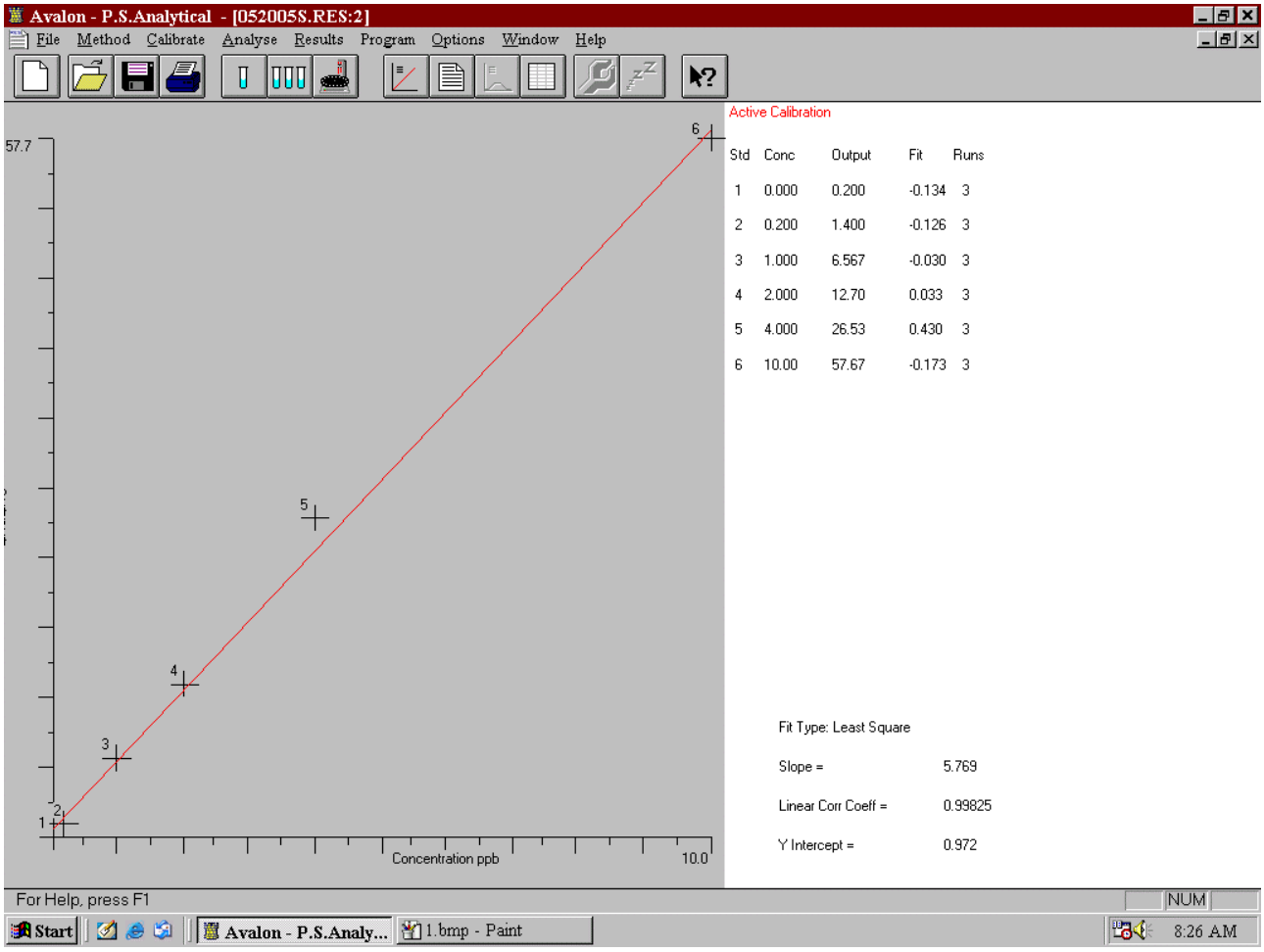

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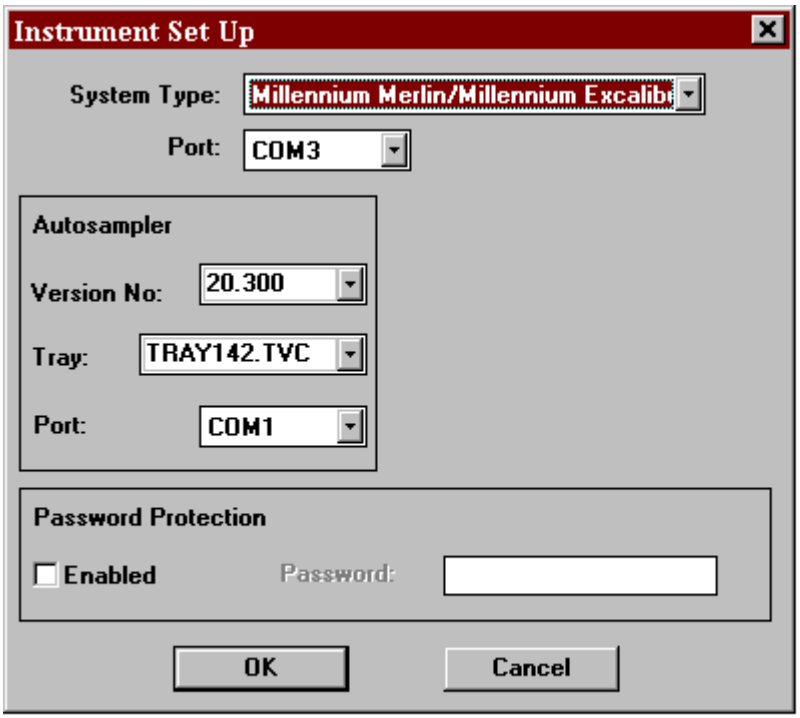

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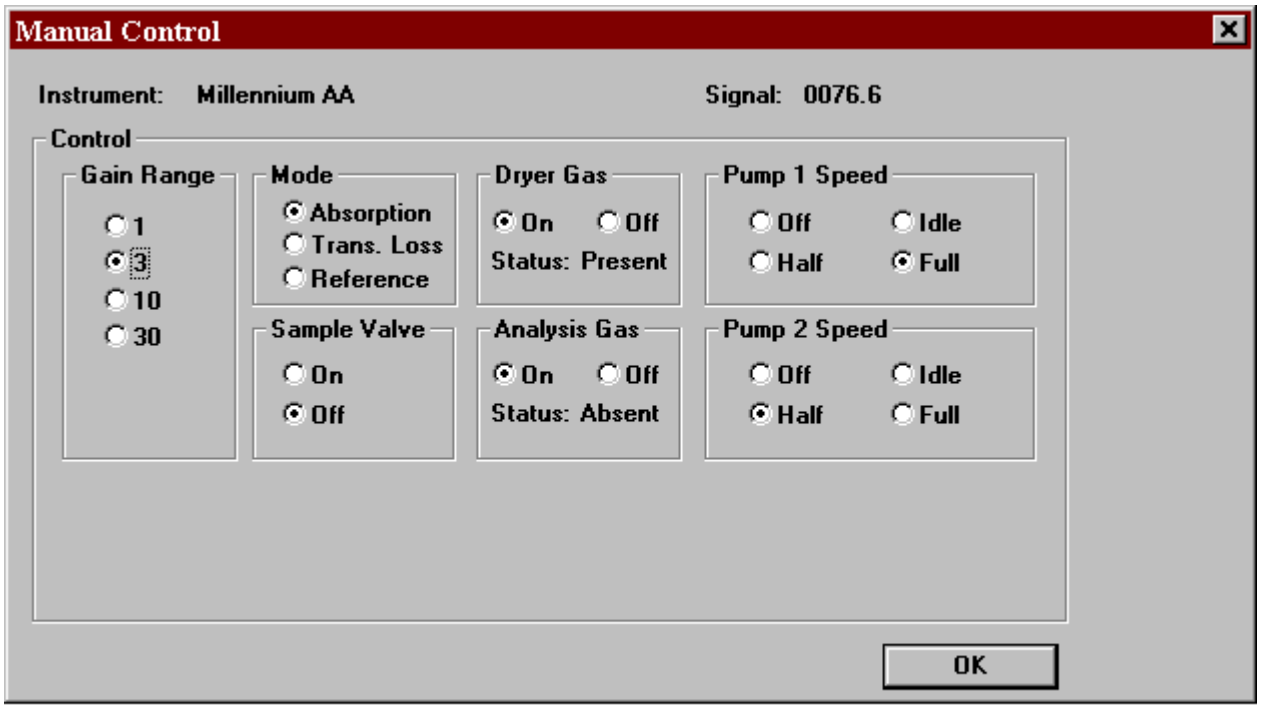

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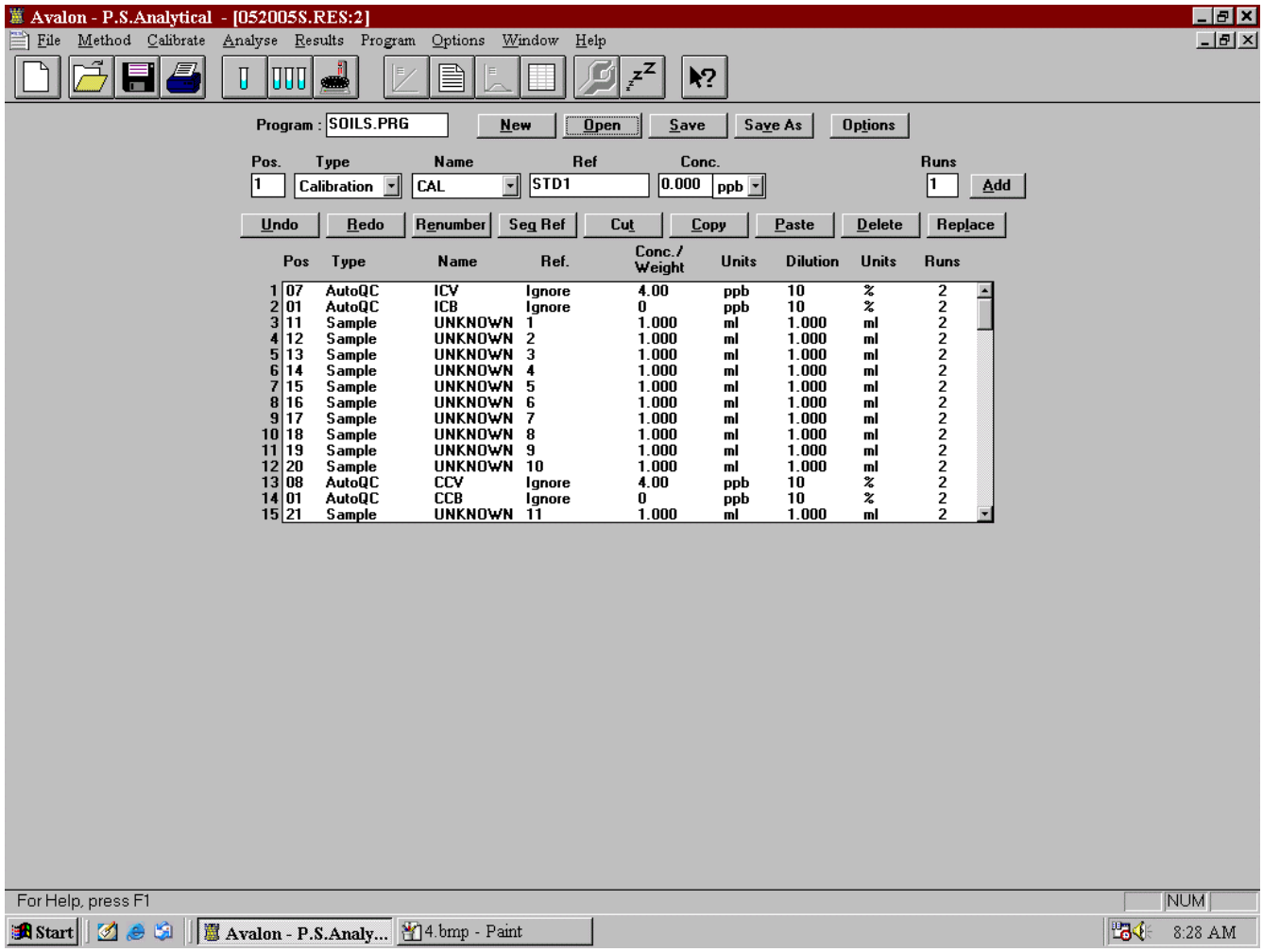

	Pos	Type	Name	Ref.	Conc./ Weight	Units	Dilution	Units	Runs
1	07	AutoQC	ICV	Ignore	4.00	ppb	10	%	2
2	01	AutoQC	ICB	Ignore	0	ppb	10	%	2
3	11	Sample	UNKNOWN	1	1.000	ml	1.000	ml	2
4	12	Sample	UNKNOWN	2	1.000	ml	1.000	ml	2
5	13	Sample	UNKNOWN	3	1.000	ml	1.000	ml	2
6	14	Sample	UNKNOWN	4	1.000	ml	1.000	ml	2
7	15	Sample	UNKNOWN	5	1.000	ml	1.000	ml	2
8	16	Sample	UNKNOWN	6	1.000	ml	1.000	ml	2
9	17	Sample	UNKNOWN	7	1.000	ml	1.000	ml	2
10	18	Sample	UNKNOWN	8	1.000	ml	1.000	ml	2
11	19	Sample	UNKNOWN	9	1.000	ml	1.000	ml	2
12	20	Sample	UNKNOWN	10	1.000	ml	1.000	ml	2
13	08	AutoQC	CCV	Ignore	4.00	ppb	10	%	2
14	01	AutoQC	CCB	Ignore	0	ppb	10	%	2
15	21	Sample	UNKNOWN	11	1.000	ml	1.000	ml	2

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Appendix C

CETAC HG WORKSHEET

File:_____
Matrix:_____
Analyst:_____
Date:_____

$SnCl_2$ solution: _____g $SnCl_2$ in _____of 1.4% H_2SO_4
NH2OH / HCl log#: _____ Pipette ID#_____
Post Spike: 50 ul_____ into 10 ml of sample.

Sample	Sample
1	37
2	38
3	39
4	40
5	41
6	42
7	43
8	44
9	45
10	46
11	47
12	48
13	49
14	50
15	51
16	52
17	53
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30	66
31	67
32	68
33	69
34	70
35	71
36	72

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Appendix D

PSA HG WORKSHEET

File:_____ SnCl$_2$ solution: _____g SnCl$_2$ in _____of 1.4% H$_2$SO$_4$
Matrix:_____ NH2OH / HCl log#: _____ Pipette ID#_____
Analyst:_____ Post Spike: 50 ul_____ into 10 ml of sample.
Date:_____

Sample	Sample
1	37
2	38
3	39
4	40
5	41
6	42
7	43
8	44
9	45
10	46
11	47
12	48
13	49
14	50
15	51
16	52
17	53
18	54
19	55
20	56
21	57
22	58
23	59
24	60
25	61
26	62
27	63
28	64
29	65
30	66
31	67
32	68
33	69
34	70
35	71
36	72

SOP Change History Sheet

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Section No.	Section	Reason for Change
1	Scope and Application	SOP update
8.2	Quality Control	PADEP Audit response
8	Quality Control Requirements	USACE audit response
12	Reporting Results	Updated for new LIMS
5	Apparatus and Materials	A2LA Audit Response SOP update 5/3/05
7	Instrument Calibration	SOP update to calibration levels 5/03/05
10	Procedure	SOP update 5/3/05
	Appendix A	Inserted New Instrument Set-up Records for CETAC 5/19/05
	Appendix B	Inserted New Instrument Set-up Records for PSA 5/19/05
	Appendix C	Insert New Instrument Logbook for CETAC 5/19/05
	Appendix D	Insert New Instrument Logbook for PSA 5/19/05

Revision 23: 03/23/2006

1.2	Scope and Application	DoD audit response, updated methods
1.8	Scope and Application	Added project requirement verbiage
6.3	Reagents	Updated vendor information

SOP Change History Sheet (continued)

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Section No.	Section	Reason for Change
6.6	Reagents	Added decolorizing agent volume as per PADEP audit response
7.3.1	Instrument Calibration	Updated reference method and standards reference
7.6	Instrument Calibration	Added corrective actions for calibration blank concentrations as per PADEP/DoD audit responses
8.2	Quality Control	Added ongoing proficiency verbiage
8.2	Quality Control	Updated table to reflect method addition/revision and concentration revisions as per DoD audit response
8.3-8.5	Quality Control	Major revisions/additions throughout
10.1.7, 10.1.9, 10.2.6	Procedure	Added verbiage about Hydroxylamine addition as per PADEP audit response
10.1.8	Procedure	Added r-value
11.4	Calculations	Added specific calculations as per DoD audit response
12	Reporting Results	Major revisions/additions throughout

SOP Change History Sheet (continued)

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Section No.	Section	Reason for Change
16	Troubleshooting	Added section as per DoD audit response

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Hexavalent Chromium by SW-846 Methods 7196A and 3060A

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Susan Magness, Date
Validator

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1 Scope and Application

1.1 This method is adapted from SW-846 Method 7196A "Hexavalent Chromium (Colorimetric) and SW-846 Method 3060A "Alkaline Digestion for Hexavalent Chromium."

1.2 This method is applicable to the determination of hexavalent chromium in TCLP leachates, groundwater, and certain domestic and industrial waters, providing no interfering substances are present. The method is also applicable to the determination of hexavalent chromium in soils, sludges, and similar waste materials providing the samples undergo the alkaline digestion procedure prior to quantitation.

3.3 The current method detection limits can be found in the wet chemistry method detection limit notebook. The detection limit for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix. Method detection limits should be performed annually, when a new analyst performs the analysis, or when a significant change is made to the procedure.

1.4 This method is restricted for use by or under the supervision of analysts experienced in the use of a spectrophotometer.

1.5 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

2 Summary of Method

2.1 Soils, sludges, and solid waste samples are digested using a 0.28 M Na_2CO_3/0.5 M NaOH solution to dissolve the hexavalent chromium and stabilize it in an aqueous solution.

2.2 Hexavalent chromium is determined colorimetrically in aqueous samples or the alkaline digestates by reaction with diphenylcarbazide in acid solution. A red-violet color is produced. The reaction is very sensitive. The absorbance is measured on a spectrophotometer at 540 nm.

3.3 With the exception of filtration to remove particulate material, aqueous samples require no pretreatment prior to color development.

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3.3 Due to the reporting limit requirements of many projects, a calibration curve from 0.01 to 1.0 mg/L has been established. This is considered to be a method modification.

3.4 For soil samples, after digestion, the final volume is diluted to 200 ml to accommodate any further dilutions that may be required. This is considered a method modification.

3 Interferences

3.1 For waste materials or soils containing soluble Cr3 concentrations greater than four times the laboratory Cr6 reporting limit, Cr6 results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^2+ in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation.

3.2 The reaction with diphenylcarbazide is nearly specific for chromium. Extreme amounts of molybdenum, vanadium and iron will interfere. Normally, however, the concentrations of these are not high enough to cause a problem. If interferences are suspected, refer to referenced method.

3.3 Some client specific projects may require determinations of the reducing/oxidizing tendency of each sample matrix. This can be accomplished by characterization of each sample for additional analytical parameters, such as pH, ferrous iron, sulfides, Oxidation Reduction Potential (ORP), Total Organic Carbon TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD). Analysis of these additional parameters establishes the tendency of Cr6 to exist or not exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

4 Safety

4.1 This method utilizes chemical compounds that are significant health hazards. All possible steps should be taken to limit analyst contact with these chemicals. The minimum personal protective equipment requirements for performing this test are PVC gloves, safety glasses, and a fully-buttoned lab coat. This PPE should reduce the possibility of contact to a safe level, but the analysts should not limit themselves to these PPE minimums.

4.2 ALSI maintains material safety data sheets on all these chemicals. An analyst new to this test should not start this test without first reading all the relative MSDS.

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5 Apparatus and Materials

5.1 Spectrophotometer – for use at 540 nm, with a light path of 1 cm or longer. Shimatzu UV-1201, is currently in use.

5.2 150 ml beakers - plastic or glass – plastic beakers: VWR catalog no. 13890-068, or equivalent, glass beakers: VWR catalog no. WL54675-1, or equivalent.

5.3 pH paper - ranging from 1 – 6, VWR catalog no. 60777-027, or equivalent.

5.4 pH Meter – Corning Model 350 is currently in use.

5.5 Pipets - Class A, various sizes

5.6 Volumetric Flasks - Class A, various sizes

5.7 Glass Fiber Filters - Gelman A/E, VWR catalog no. 38150-190, or equivalent.

5.8 0.45 µm Membrane Filter Paper - VWR catalog no. 28148-584, or equivalent.

5.9 Filter Flask - 500 ml or 1000 ml, VWR catalog nos. 29410-990 and 29410-993, or equivalent.

5.10 Filter Funnel – magnetic, VWR catalog no. 28143-550.

5.11 Automatic pipet capable of dispensing 0.500 ml. An Eppendorf 2000 variable volume pipet, VWR catalog no. 53511-582, or equivalent.

5.12 Digestion Vessel: borosilicate glass 250 ml beakers, VWR catalog no 13910-201, or equivalent.

5.13 Graduated Cylinder 100-mL, VWR catalog no. 24710-124, or equivalent.

5.14 Heating Device - capable of maintaining the digestion solution at 90~95^{0}C with continuous auto stirring capability.

5.15 Thermometer capable of measuring up to 100 degrees centigrade (with NIST traceable calibration), VWR catalog no. 61017-867, or equivalent.

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5.16 An automated continuous stirring hotplate.

6 Reagents

6.1 Reagent Water. Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose ALSI uses a Filson Water Purification System which provides analyte-free, greater than 16.0 megohm-cm DI water on demand.

6.2 Hexavalent Chromium Calibration Stock Solution (50 mg/L) – Purchased from HACH catalog no. 810-42, or equivalent. Store at 4°C. Discard according to the manufacturer's expiration date.

6.3 Hexavalent Chromium Calibration Working Solution (1.0 mg/L) - Pipet 10.0 ml of Stock Chromium Solution (6.2) into a 500 ml volumetric flask. Bring to volume with reagent water. This solution is used for the calibration curve preparation. The dilution must be recorded in the standards logbook. Prepare fresh for each use.

6.4 Hexavalent Chromium Verification Stock Solution (50 mg/L) – Dissolve 141.4 mg of $K_2Cr_2O_7$ (VWR catalog no. EM-PX1445-1 or equivalent) in reagent water and dilute to 1000 ml. This solution should be remade every six months. The preparation of this standard should be recorded in the standards logbook. Store at 4°C.

6.5 Calibration Verification Standard (0.5 mg/L) – This standard must be prepared daily. Using a Class A pipet transfer 5.0 ml Stock Verification Chromium Standard (6.4) into a 500 ml volumetric flask. Bring to volume using reagent water. This must be made fresh for each use. The dilution must be recorded in the standards logbook.

6.6 Aqueous Matrix Spike (0.50 mg/L) – Pipet 1.0 ml of the Calibration Stock Solution (6.2) into 100 ml of sample.

6.7 Solid LCS (20 mg/Kg) – Pipet 1.0 ml of the calibration stock solution (6.2) into 2.5 grams of analyte-free sand.

6.8 Solid Pre-digestion Soluble Matrix Spike (40 mg/Kg) – Pipet 2.0 ml of the calibration stock solution (6.2) into 2.5 grams of sample. The exact concentration of the spike can be calculated:

$$Cr^{+6} \text{ Spike mg/Kg} = \frac{(\text{ml spike sol'n})\ (\text{conc spike sol'n})}{\text{Grams of Sample}}$$

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6.9 Solid Pre-digestion Insoluble Matrix Spike (644 mg/Kg) – Add 10 mg of $PbCrO_4$ to 2.5 grams of sample. This concentration is above the calibration range and will require a minimum 20X dilution prior to analysis. The exact concentration of the spike can be calculated:

$$Cr^{+6} \text{ Spike mg/KG} = \frac{(\text{mg } PbCrO_4)\ (160.9)}{\text{Grams of Sample}}$$

6.10 Solid Post-digestion Matrix Spike (40 mg/Kg) – Pipet 2.0 ml of the calibration stock solution (6.2) into the final digestate immediately before dilution to the final volume.

6.11 Acetone – For spectrophotometers. VWR catalog no. BJ010-4, or equivalent. Store in a flammable liquids cabinet. Discard according to the manufacturer's expiration date.

6.12 1,5-Diphenylcarbazide (1.5-Diphenylcarbohydrazide) – Manufactured by J.T. Baker, purchased from VWR catalog no. K620-03, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.13 Diphenylcarbazide Solution – Dissolve 0.500 gram 1.5-diphenylcarbazide in 100 ml acetone. Store in an amber bottle at 4°C. Discard when solution becomes discolored, or after 6 months. The lot number of the chemical used to make this reagent should be recorded in the reagent logbook.

6.14 Sulfuric Acid (Conc.) – Purchased from VWR catalog no. JT9681-33, or equivalent. Store in an acid storage cabinet. Discard according to the manufacturer's expiration date.

6.15 Sulfuric Acid (10%) - Add 100 ml concentrated sulfuric acid to 500 ml of reagent water in a 1 liter volumetric flask. Bring to volume with reagent water. Store at room temperature. Discard after 6 months.

6.16 Nitric Acid(concentrated) – Analytical reagent grade - Do not use if it has a yellow tinge; this is indicative of photoreduction of NO_3 to $N0_2$, a reducing agent for Cr(VI). Purchased from VWR catalog no. JT9601-5, or equivalent. Store in an acid storage cabinet. Discard according to the manufacturer's expiration date.

6.17 Nitric acid - 5.0 M HNO_3 - Add 320 ml of concentrated nitric acid (6.16) to 500 ml of reagent water. Mix, and dilute to volume in a 1 liter volumetric flask. Store at room temperature. Discard after 6 months.

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6.18 Sodium Carbonate: Na_2CO_3, anhydrous, analytical grade, purchased from VWR catalog no. JT3602-1, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.19 Sodium Hydroxide: NaOH, analytical grade, purchased from VWR catalog no JT3722-7, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.20 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical grade, purchased from Aldrich catalog number 20833-7, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.21 Potassium Hydrogenphosphate: K_2HPO_4: Analytical reagent grade, purchased from VWR catalog number JT3252-01, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.22 Potassium diHydrogenphosphate: KH2PO4: Analytical reagent grade, purchased from VWR catalog number JT3246-1, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.23 Phosphate Buffer (0.5 M): Dissolve 87.09 grams of K_2HPO_4 and 68.04 grams of KH_2PO_4 into 700 ml of reagent water. Transfer to a 1 liter volumetric flask and dilute to volume. Store at room temperature. Discard after 6 months.

6.24 Lead Chromate: $PbCrO_4$, analytical reagent grade, purchased from VWR catalog number JT2314-04, or equivalent. Store at room temperature. Discard according to the manufacturer's expiration or after 5 years if an expiration date is not provided.

6.25 Digestion Solution: Dissolve 20.0 +/- 0.05 grams of NaOH and 30.0 ± 0.05 grams of Na_2CO_3 in reagent water in a 1 liter volumetric flask and dilute to volume. Store in a tightly capped polyethylene bottle at room temperature and prepare fresh monthly. The pH of the digestion solution must be checked prior to using. The pH must be 11.5 or greater, if not, discard.

6.26 Aqueous LCS (0.50 mg/L) – Pipet 1.0 ml of the calibration stock solution (6.2) into 100 ml of reagent water.

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7 Instrument Calibration

7.1 Turn on spectrophotometer and adjust to 540 nm. Allow it to warm up for 20 minutes.

7.2 A full five-point curve must be made each days samples are analyzed.

7.3 Prepare a calibration curve by making a series of standards diluted from the 1 mg/l calibration standard (6.3) according to the following procedure. Standards should be prepared using Class A pipets and volumetric flasks.

ml of 1 mg/L Cr+6 Standard	Concentration when diluted to 100 ml (mg/L) (95 ml used for analysis)
1	0.01
10	0.10
25	0.25
50	0.50
100	1.0

7.4 Measure into separate beakers 95 ml of each of the five calibration standards, two (2) reagent blanks, and a 0.5 mg/l verification standard (6.5).

7.5 Add 2.0 ml of diphenylcarbazide solution to each beaker using a Class A pipet or automatic pipet. Swirl to mix.

7.6 Using 10% H_2SO_4 adjust the pH of the solutions to pH 2.0 ± 0.5. Check that the pH is within this range using pH paper. Record the pH in the laboratory book. No more than 3 milliliters of 10% H_2SO_4 should be needed to bring the pH to 2.0.

7.7 Pour each standard or solution back into a 100 ml volumetric flask and dilute to volume with reagent water. After bringing to volume, pour the liquid back into its original beaker.

7.8 Allow solutions to stand 10 minutes for full color development.

7.9 Zero the spectrophotometer using the reagent blank.

7.10 Rinse the cuvette with the sample to be measured, then transfer an aliquot of the sample to the cuvette and measure the absorbance.

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7.11 Read and record the absorbance of each calibration standard, the verification standard, and follow with the second reagent blank to verify that the spectrophotometer has not drifted.

7.12 Access the curve generation program that uses linear regression analysis in the ALSI 02 Server accessed through Network Neighborhood.

Open Network Neighborhood
Open ALSI 02
Open Folder "E"
Open Folder "Wet Chem"
Open Folder "Curves"
Open File "Cr^{+6} Curve"

7.13 Enter the concentrations of the calibration standards in mg/L in the "x" column starting with the 0.01 mg/L standard. Enter the corresponding absorbances in the "y" column.

7.14 The software will generate an equation for the curve and a correlation coefficient. The correlation coefficient must be at least 0.995, and the concentration of the verification standard calculated using the equation must be within 10% of its true value for the curve to be accepted.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Manual shall be followed when performing this procedure.

8.2 Method Detection Limit (MDL) studies shall be performed annually. MDL's will be performed as specified in 99-MDL, which is derived from the procedures outlined in 40 CFR. MDL's will be processed in the same manner as a field sample. The calculated MDL must be between 10% and 100% of the spike concentration.

8.3 Initial Demonstration of Capability

8.3.1 Prepare and analyze for replicate Laboratory Control Samples (Aqueous Samples (6.26)/Solid Samples (6.7)). All four results should be within 15% of their true value for aqueous samples and within 20% of their true value for solid samples. If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples begins.

9.4.1 Using the data generated in Section 8.3.1, calculate the percent relative standard deviation (%RSD) of the replicate analysis as indicated below. To be

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acceptable, the %RSD must be less than 15% for aqueous samples and less than 20% for solid samples.

$$\% \, RPD = \frac{\text{Sample Standard Deviation (S)}}{\text{Mean Recovered Concentration}} \times 100$$

If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples is begun.

8.4 QC Requirements Aqueous Samples:

Quality Control Requirements Aqueous Samples
(Specific project requirements may override these requirements.)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Method Blank	---	Following the ICV, repeated every 10 samples, and at the end of the analysis	< Reporting Limit. For DOD QSM samples <1/2 Reporting Limit RL = 0.02 mg/L	Reanalyze all associated samples. If samples cannot be reanalyzed, report with a qualifying statement.
Initial Calibration Verification Standard (6.5)	0.50 mg/L	Following the initial calibration	90-110%	Reanalyze all associated samples. If samples cannot be reanalyzed, report with a qualifying statement.
Continuing Calibration Verification Standard (6.5)	0.50 mg/L	After every 10 samples and at the end of the analysis batch.	90-110%	Reanalyze all associated samples. If samples cannot be reanalyzed, report with a qualifying statement.
Laboratory Control Sample (6.26)	0.50 mg/L	One every 20 samples with a minimum of one per batch.	85-115%	Reanalyze all associated samples. If samples cannot be reanalyzed, report with a qualifying statement.
Duplicate*	--	One every 10 samples with a minimum of one per batch.	RPD</=15%	Reanalyze once. If reanalysis is not possible, or if RPD is still unacceptable, report with a qualifying statement.
Matrix Spike* (6.6)	0.50 mg/L	One every 10 samples with a minimum of one per batch.	85-115%**	Reanalyze once. If reanalysis is still unacceptable and calibration verification standards are acceptable, problem is judged to be matrix related. Report with a qualifying statement.
Matrix Spike Duplicate * (6.6)	0.50 mg/L	One every 10 samples with a minimum of one per batch.	RPD</=15%	Reanalyze once. If reanalysis is not possible, or if RPD is still unacceptable, report with a qualifying statement.

(*) Samples selected for duplicate and spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor

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performance in a duplicate or spike analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

(**) If the sample has a spike recovery of less than 85% and is an acidic extract, the sample is to be retested to determine if the low recovery is due to the presence of residual reducing agent. This determination shall be performed by first making an aliquot of the extract alkaline (pH 8.0 – 8.5) using 1 N NaOH and respiking and reanalyzing. If a spike recovery of 85 – 115% is obtained in the alkaline aliquot of an acidic extract that initially was found to contain <5 mg/L Cr+6, one can conclude that the analytical method has been verified.

8.5 QC Requirements Soil, Sludge, Solid Waste Samples:

QC Requirements Soil, Sludge, Solid Waste Samples
(Specific project requirements may override these requirements.)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Reagent/ Calibration Blank	---	Following the ICV, repeated every 10 samples, and at the end of the analysis batch.	<0.01 mg/L	Reanalyze all associated samples since the last acceptable blank.
Method Blank	---	One every 20 samples with a minimum of one per batch.	Less than RL of 5 mg/kg or less than 1/10 the regulatory or action level, whichever is greater. Less than ½ RL for DOD QSM samples.	Redigest and reanalyze all samples with a concentration of less than 10 times the blank result.
Initial Calibration Verification Standard (6.5)	0.50 mg/L	Following the initial calibration	90 – 110%	Reanalyze all associated samples. If samples cannot be reanalyzed report with a qualifying statement.
Continuing Calibration Verification Standard (6.5)	0.50 mg/L	After every 10 samples and at the end of the analysis batch.	90-110%	Reanalyze all associated samples. If samples cannot be reanalyzed report with a qualifying statement.
Laboratory Control Sample (6.7)	20 mg/kg	One every 20 samples with a minimum of one per batch	80 – 120%	Redigest and reanalyze all associated samples. If samples cannot be reanalyzed, report with a qualifying statement.
Duplicate*	---	One per 20 samples with at least one per batch.	20% RPD if both samples are > or = 4 times reporting limit, +/- reporting limit if one or both samples are <4 times the reporting limit.	If all of the method performance criteria are acceptable, the precision problem is judged to be matrix related. Report with a qualifying statement.

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Pre-digestion Soluble Matrix Spike* (6.8)	40 mg/kg	One every 20 samples. Minimum of one per batch.	75 – 125%	If recovery is outside limits and LCS is in control recovery problem is judged to be matrix related. Report with a qualifying statement.
Pre-Digestion Insoluble Matrix Spike* (6.9)	644 mg/kg	One every 20 samples. Minimum one per batch.	75 – 125%	If recovery is outside limits and LCS is in control recovery problem is judged to be matrix related. Report with a qualifying statement
Post-Digestion Matrix Spike* (6.10)	40 mg/kg	One every 20 samples. Minimum one per batch.	85 - 115%	If recovery is outside limits and LCS is in control, recovery problem is judged to be matrix related. Report with a qualifying statement.

*Samples selected for duplicate and spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

9.4 All extracts and samples that are part of a delisting petition must be analyzed by the method of standard additions (SW-846 Method 7000, Section 8.7). Prior to sample receipt, contact should be made with the customer to determine if the method of standard additions is to be used.

9.5 All water samples that suffer from matrix interferences must be analyzed by the method of standard additions. The need for the method of standard additions on soil samples will be based on the post-digestion spike. A minimum of one sample from each project site must be utilized as a matrix spike. If the recovery of the spike is not within the established or project defined control limits, all samples of the sample matrix from that site must be analyzed by the method of standard additions.

9 Sample Collection, Preservation and Handling

9.1 Collect samples in plastic or glass bottles. A minimum of 200 mL for aqueous. Samples and 20 grams for solid samples is required.

9.2 Samples should be unpreserved, and maintained at 4 +/- 2 degrees C until analysis.

9.3 The maximum holding time for hexavalent chromium solid samples is 30 days until digestion and an additional 7 days after digestion.

9.4 The maximum holding time for hexavalent chromium aqueous samples is 24 hours.

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9.5 For EP and TCLP leachates there is a 24 hour holding time for extraction and a 24 hour holding time until analysis for a possible holding time of 48 hours for sampling to analysis.

10 Soil Digestion Procedure (Aqueous samples proceed to section 11 analysis procedure)

10.1 Place 2.5 +/- 0.10 gram of sample into a clean and labeled 250 ml glass beaker. The sample should have been mixed thoroughly before the aliquot is removed.

10.2 For samples being pre-digestion spiked, the spike material should be added directly to the sample at this point.

10.3 Using a graduated cylinder, add 50 ml +/- 1 ml of digestion solution to each sample.

10.4 Add approximately 400 mg of MgCl2 (6.16) to each sample.

10.5 Add 0.5 ml of 1.0 M Phosphate Buffer (6.19) to each sample.

10.6 Cover each beaker with a watch glass.

10.7 Place beakers on a magnetic stirrer (unheated) for at least 5 minutes.

10.8 Place beakers on a heated stirrer at 90-95 degrees centigrade. Maintain the samples at 90-95 degrees centigrade for at least 60 minutes with continuous stirring.

10.9 Turn hotplate off and continue to stir the samples until they have reached room temperature.

10.10 Transfer the contents quantitatively to the filtration apparatus; rinsing the beaker with 3 successive portions of reagent water. Transfer the rinsates to the filtration apparatus. Filter through a 0.45 um membrane filter. If necessary, use a prefilter to prevent clogging. Rinse the inside of the filter flask and filter pad with reagent water and transfer the filtrate and rinses to a clean 250 ml beaker.

10.11 Place the sample beaker on the magnetic stirrer in a fume hood, and, with constant stirring, slowly add 5.0 M nitric acid (6.17) solution to the beaker dropwise. Adjust the pH of the solution to 7.5 +/- 0.5 and measure with pH paper. If too much acid is added, the sample must be discarded and redigested.

10.12 If a flocculent precipitate should form during the addition of acid, the sample must be filtered through 0.45 um membrane filter again.

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10.13 For samples being post-digestion spiked, the spike material should be added directly to the digestate at this point.

10.14 Transfer the contents of the beaker to a 200 ml volumetric flask and dilute to volume with reagent water. The sample is now ready to be analyzed.

11 Analysis Procedure

11.1 Turn on spectrophotometer and adjust to 540 nm. Allow it to warm up for 20 minutes.

11.2 Into separate beakers, pour 95 ml of each sample (on an aliquot diluted to 95 ml), blank, verification standard (6.5), matrix spikes and matrix spike duplicate. If samples are turbid or contain suspended material they should first be filtered through a glass fiber and/or 0.45 µm filter.

11.3 All 2.0 ml diphenylcarbazide solution using a Class A or automatic pipet.

11.4 Use 10% H_2SO_4 solution to adjust the pH to 2 ± 0.5.

11.5 Dilute to 100 ml with reagent water and let stand 10 minutes for full color development.

11.6 Zero the spectrophotometer using a reagent blank.

11.7 Transfer an aliquot of each solution to the absorption cell for the spectrophotometer and measure the absorbance at 540 nm.

11.8 An aliquot of the sample containing all reagents except diphenylcarbazide should be prepared and used to correct the absorbance for turbidity. From the corrected absorbance determine the concentration of chromium.

12 Calculations

12.1 From the computer generated equation record the concentrations of all samples, verification standards, etc…

12.2 If the sample was turbid and required a sample blank analysis, correct the absorbance reading of the final colored solution by subtracting the absorbance of the pre-colored sample prior to calculating the final concentration.

12.3 Sample concentration for solid samples is calculated as:

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$$Cr^6, \text{ mg/kg} = \frac{\text{Result from Curve, mg/l x 200ml}}{\text{Sample Size, grams}} \times \text{(Any Additional Dilution)}$$

12.4 Standard recovery is calculated as:

$$\% \text{ Recovery} = \frac{\text{(Result, mg/kg)}}{\text{True Value, mg/kg}} \times 100$$

12.5 Spike recovery is calculated as:

$$\% \text{ Recovery} = \left(\frac{\text{Spike Result – Unspiked Sample Result, mg/kg)}}{\text{Spike Added, mg/kg}} \right. \times 100$$

12.6 Precision (RPD) is calculated as:

$$\% \text{ RPD} = \frac{\text{(Result 1 – Result 2)}}{\text{Average Result}} \times 100$$

13 Reporting Results

13.1 Report results only for samples that fall within the lowest and highest calibration standards. Any sample that is above the highest concentration standard must be diluted and rerun until the concentration falls within the range of the curve.

13.2 Report all detectable results to three significant figures.

13.3 All results are entered into the LIMS on a “Wet” basis. The LIMS will automatically adjust soil samples to a dry weight basis by:

$$\text{Dry Weight} = \text{Result} \times \frac{100}{\text{Total Solids}}$$

14 Waste Disposal

14.1 Refer to ALSI SOP 19-Waste Disposal.

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15 Pollution Prevention

15.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

16 Definitions

16.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

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Appendix A
HEXAVALENT CHROMIUM

Sample	ABS	Blank ABS	Corr. ABS	Chromium (mg/L)	pH	Curve Used	Date	Tech	Time Started

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Appendix B

Hexavalent Chromium Soil Digestion Logbook

Sample	(grams)	Date	Tech	Sample	(grams)	Date	Tech

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SOP Change History Sheet

Section Number	Section Description	Reason for Change
1.3	Scope and Application	NELAC additions
5	Apparatus and Materials	NELAC additions
6	Reagents and Standards	NELAC additions
7.3	Instrument Calibrations	Correction to current SOP
8	Quality Control	Correction to current SOP
9	Sample Colletion, Preservation & Handling	Correction to current SOP
16	Definitions	NELAC additions
	Appendix A	Added
	Appendix B	Added

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Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Microwave Assisted Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP or GFAA Spectroscopy

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC.
(ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

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Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Alan Lopez, Date
Laboratory Technical Manager

_____ _____
KaTonna Williams Date
Validator

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1 Scope and Application

1.1 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.2 This digestion procedure is used for the preparation of aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for analysis by graphite furnace (GFAA), and inductively coupled argon plasma spectroscopy (ICP). The procedure is an acid digestion for determining total available metals on samples requiring digestion by Method 3015 of EPA SW846 "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," Revision 0, September 1994.

1.3 Samples prepared by Method 3015 may be analyzed by GFAA or ICP for the following metals:

Aluminum	Lead
Antimony	Magnesium
Arsenic	Manganese
Barium	Molybdenum
Beryllium	Nickel
Cadmium	Potassium
Calcium	Selenium
Chromium	Silver
Cobalt	Sodium
Copper	Thallium
Iron	Vanadium
	Zinc

1.4 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 A representative 45 mL aqueous sample is digested with 5 mL of concentrated nitric acid in a Teflon digestion vessel for 20 minutes using microwave heating. The sample is transferred, with filtering if necessary, to a clean sample bottle for analysis.

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3 Interferences

3.1 In order to identify problem matrices and method error, blanks, spikes, spike duplicates and check samples are run at regular intervals, as specified in each relevant analytical method.

3.2 Samples that are oily or continuously vent in the microwave shall be digested by an alternate hot plate digestion method (see prep SOP 09-3050B).

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Therefore, each chemical shall be treated as a potential health hazard.

4.2 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.

4.3 Analysts shall consult the Material Safety Data Sheets (MSDS) for each chemical used in the digestion process.

4.4 Since the chemical make-up of the samples is not known, analysts shall treat the samples with extreme caution. Proper protective equipment must be used including PVC gloves, lab coats, safety glasses and a fume hood.

4.5 Although there are many safety features built into the microwave, technicians must remember that the samples will be acidified to 10% nitric acid, heated to approximately 170°C and will be under pressures of 70 psi. All parts of the digestion vessels must be examined to ensure that there are no visible faults that could release the sample during digestion.

4.6 Analysts must be familiar with the proper assembly and use of the vessels and all related safety equipment. Information regarding the safe use of the MSD-2100 can be found in the *MSD-2100 Operation Manual* to be found in the microwave supplies drawer in the Inorganic Prep Lab.

4.7 The addition of Conc. Nitric acid (6.2) to water in Sections 7.2 and 10.7 may cause significant heat generation or a vigorous chemical reaction. The analysts must always add acid to water, not water to acid. This step must also be

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performed in a fume hood.

5 Apparatus and Materials

5.1 Microwave, CEM Model MDS-2100 with advanced composite digestion vessels.

5.2 100mL graduated cylinder, Class A or Class B.

5.3 Filter Paper, Whatman #2.

5.4 Filter funnels, plastic.

5.5 8 fl-oz. Polyethylene bottles, disposable. VWR #16059-068, or equivalent.

5.6 Acid Dispensers (Finnipipettes), from Thermo capable of dispensing 0.5mL to 10mL of acid.

5.7 Thermo Finntip tips, VWR catalogue #53515-050 or equivalent.

5.8 Balance, capable of weighing to 0.01g, Mettler PM2000.

6 Reagents

6.1 Reagent Water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System, which provides analyte-free, >16.0 megohm-cm deionized water on demand. All references to water in the method refer to reagent water unless otherwise specified.

6.2 Nitric Acid, concentrated, (HNO_3). J.T. Baker 'Baker analyzed' grade, Cat. #JT-9598-34 purchased from VWR or equivalent.

Note: Acid bottles must be contained inside Teflon carriers at all times. If an expiration date is not supplied by the manufacturer, the expiration will be five years from the date opened. This date shall be marked directly on the acid bottle.

6.3 Spiking solution Standard Mix #1-R purchased from, High Purity Standards, Cat. #SM-1339-001, or equivalent NIST certified standard. This is a pre-

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mixed standard of Al, Ba, Ca, Li, Mg, and Se at 100 mg/l, As, Cd, Cr, Pb, and Sr at 10 mg/l, K and Na at 2000 mg/l, Be at 20 mg/l, and V at 5 mg/l in 5% HNO_3.

6.4 Spiking Solution Standard Mix #2 purchased from High Purity Standards, Cat. #SM-1339-002, or equivalent NIST certified standard. This is a pre-mixed standard of Bi, B, Co, Cu, Fe, Mo, and Ni at 100 mg/l, Mn, Ag, Tl, Sn, and Ti at 10 mg/l, Sb at 20 mg/l, and Zn at 50 mg/l in 5% HNO_3.

7 Glassware Cleaning

7.1 Glassware used in this procedure is to be cleaned as noted in the Glassware Washing Standard Operating Procedure (09-GLWH) for metals glassware. This procedure includes the acid washing of all glassware used for metal digestions.

7.2 Glassware used in this procedure is stored in metals prep area and rinsed with a 10% Nitric Acid (HNO_3) solution before use. Exact measurements are not necessary to produce this cleaning reagent. Production is approximately 100 mL concentrated HNO_3 into 900 mL of deionized water.

Note: Some heat will be generated from the addition of acid to water.

7.3 For determination of trace elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory glassware and apparatus and general contamination within the laboratory environment from dust, etc. A clean laboratory work area designated for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface adsorption or leaching, (2) depleting element concentrations through adsorption processes. All reusable lab ware (glass, quartz, polyethylene, PTFE, FEP, etc.) shall be sufficiently clean for the task objectives.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Demonstration of Capability (DOC) – DOCs must be performed by each analyst prior to performing this method and repeated at any time there is a significant

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change in instrument type. To perform DOCs, four consecutive Laboratory Fortified Blanks (LCSs), with a matrix matching that of the calibration standards, are analyzed. The recoveries obtained must be within 85-115% of the known values for each associated metal, and consecutive reads must have an RSD less than 10%. If the DOCs are outside these acceptance limits, a new set of LCS's must be digested and analyzed. This process is repeated until the DOCs are completed successfully.

8.2.1 Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.3 For each analytical batch of samples processed, a method blank shall be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated. A method blank is a 45 mL aliquot of reagent water digested as a sample.

Note: The method blank concentration must be <1/2 the reporting limit for USACE / DOD samples. If the blank recoveries are outside this acceptance limit, the samples associated with the batch will need to be redigested in a new batch.

8.4 Spiked samples or standard reference materials shall be employed to determine accuracy. A spike and spike duplicate sample must be prepared for every twenty (20) samples.

Note: Some client contracts (ex. DOD) require the preparation of matrix spikes for every new matrix received. The Horizon LIMS will designate that these samples are to be prep spiked. The 1 per 20 batch spiking rule will not always be applicable to these samples. If the LIMS designates a sample MS/MSD, it is not to be disregarded by the analyst.

8.4.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.4.2 Samples to be spiked for (ICP) analysis shall be spiked in the following manner. Using a Finnipipette (5.6), add 500 µL of Standard Mix #1-R (6.3) and 500 µL of Standard Mix #2 (6.4), to the samples before the

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addition of Nitric Acid. Record spike amounts and lot #'s used in the digestion logbook.

Note**:** The final volume for samples spiked in this manner will be 51 mL.

8.5 A Laboratory Fortified Blank / Laboratory Control Sample (LCS) is to be run minimally every 20 samples or at least once on each 8-hour shift. The blank spike is a 45 mL aliquot of reagent water spiked as noted in Section 8.4.2. Special projects may require more frequent analysis of the LCS.

Note: The final volume for LCS's spiked in this manner will be 51 mL.

8.6 The expiration date of all standards and reagents shall be labeled on their respective containers. If an expiration date is not supplied by the manufacturer, the default expiration date shall be one year from the date opened.

8.7 The ALSI Lab I.D. number of all mechanical pipettes used in this digestion process must be documented in the prep logbook.

8.8 All mechanical pipettes used in this digestion process must calibrated annually to verify the ***entire*** range of use. Select four to five settings throughout the pipette range, and perform a full calibration at each setting. This calibration data must be recorded in the metals autopipette calibration logbook. (See 09-AP SOP for calibration procedure.)

8.8.1 A single setting pipette calibration is also required on a weekly basis to verify dispensing volumes. Select a mid-range volume to perform this calibration, and be sure to document the procedure in the metals autopipette logbook. (See 09-AP SOP for calibration procedure.)

8.9 The Mettler balance used to weigh the digestion vessels must be calibrated each day. Prior to use, the analyst must verify that daily calibration has been recorded in the appropriate Balance Calibration Logbook. The procedure for calibrating the balance can be found in SOP 09-PM2000.

8.10 All QC spiking solutions used in this method must be stored in area separate from samples and sample digestates. If the spiking solutions are going to be kept in the same room as samples, then a separate drawer or enclosed cabinet

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must be designated for their storage.

Quality Control Requirements
(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Corrective Action
Method Blank (LRB)	--	One per batch of 20 samples or less.	Dependent upon analysis results. Samples in the batch must be < the reporting limit or ≥ 10X the method blank. For batches involving samples from USACE / DOD the method blank recovery must be <1/2 the reporting limit. If not, the samples associated with the blank must be redigested.
Laboratory Fortified Blank (LFB or LCS)	See Sections 8.4 & 8.5	One per 20 samples or at least once per 8-hour shift.	Dependent upon analysis results. If LCS recoveries remain outside acceptable limits of 80-120% after reanalysis, the samples associated with it must be redigested and reanalyzed.
* Matrix Spike (MS)	See Sections 8.4 & 8.5	One per batch of no more than 20 samples.	The matrix spike recovery must be within 75-125% of the expected value. If the spike fails, a post digestion spike is performed at the time of analysis, and a comment is added to the client's lab report. The metals analyst will add these comments when the sample results are entered.
*Duplicate or matrix spike duplicate (MSD)	For MSD see sections 8.4 & 8.5	One MSD per batch of no more than 20 samples. Sample duplicates are performed only when requested by the client.	The Relative Percent Difference (RPD) between the Spike and Spike duplicate must be within 20%. If the RPD is above 20%, a comment must be added to the report.

Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.11 MDL studies must be performed according to SOP 99-MDL or the reference methods, whichever is more frequent.

9 Sample Collection, Preservation and Handling

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9.1 Both plastic and glass containers are suitable.

9.2 Aqueous waste waters must be acidified to a pH of <2 with HNO_3 at the time of sampling as a means of preservation.

9.3 Samples and sample digestates must be stored separately from prep spiking solutions.

10 Procedure

10.1 Any apparatus coming into contact with the sample (containers, graduated cylinders, filter paper, filter funnels, etc) must be rinsed with both 10% HNO_3 and reagent water. Also, the graduated cylinder used to measure out samples shall be rinsed at least twice with both 10% HNO_3 and reagent water in between transferring each sample.

10.2 Section 10.1.4 of EPA Method 3015A states that a microwave power check shall be periodically performed to verify the integrity of the calibration. Check to make sure that the power check has been performed on a weekly basis. If not, perform the microwave power check as specified in the Microwave Power Check SOP (09-PWRCK). According to the EPA Method, a partial power setting, obtained from the three-point calibration, shall be used for the power check. The wattage obtained from the power check shall be ± 10 w from the selected partial power setting. If the wattage is outside allowable limits, repeat the power check. If the wattage remains outside acceptable limits, the entire calibration needs to be re-evaluated. If the wattage is within allowable limits, the multiple point calibration remains valid, and the digestion process can continue.

Note: A service contract for the microwave, CEM Model MDS-2100, is maintained with the manufacturer. As per that agreement, a service technician performs a calibration twice a year. A certificate of calibration is provided and stored in the metals prep area.

10.3 Assemble, according to the manufacturer's instruction, a microwave tray with nine of each of the following: advanced composite sleeves, thread rings, and Teflon liners. These are to be placed in positions 1,2,3,5,6,7,9,10, and 11 as labeled on the carrousels. Counter balances shall be used if necessary so as to evenly distribute the weight on the turntable.

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10.4 Using a graduated cylinder, Class A or Class B, transfer a 45 mL representative aliquot of the well-mixed sample to a digestion vessel. **Note:** The number of the digestion vessels must be documented with COC# of the sample it contains in the microwave digestion logbook. This is necessary because there is no place to record the sample number on the digestion vessel itself. Also, record the sample volume used in the "sample amount" column of the microwave digestion logbook. (App. A)

10.5 Using the method explained in Section 10.3, fill the rest of the tray including the blank which is a 45 mL aliquot of reagent water. Note: If fewer than nine (9) samples are to be digested, fill out the rest of the tray with vessels containing 45 mL of deionized water and 5 mL of nitric acid to balance the energy inside the cavity of the microwave.

10.6 If any of the samples are to be spiked, add the spike solutions at this time as described in Section 8.4. Record spike amounts and lot numbers in the designated columns in the microwave digestion logbook.

10.7 Using the Finnipipette acid dispenser from Thermo, add 5 mL of concentrated nitric acid to each digestion vessel. This step shall be performed in a fume hood because of the possibility of a vigorous reaction between the sample and concentrated nitric acid. If a vigorous reaction does occur, allow the sample to cool to room temperature before capping the vessel.

10.8 Place a cover and cap on each of the vessels remembering to include a pressure regulating cover on one of the samples. Tighten the cap just hand tight. The threads will expand during heating in the microwave.

10.9 Using an analytical balance, weigh each digestion liner/cover/cap assembly to the nearest 0.01g and record this weight in the "initial weight of vessel" column of the microwave digestion logbook.

10.10 Assemble the overflow reservoir making sure that a vent tube from the reservoir is connected to each digestion vessel.

10.11 Place an **OPEN** transparent valve on the pressure control cover.

10.12 Place the tray in the microwave cavity making sure to connect the pressure line to the transparent valve on the tray.

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Note: The pressure line must be purged of any air bubbles in order to accurately read the pressure of the sample. This may require filling the syringe with deionized water.

10.13 Recall the stored program (3015) from the stored methods menu. Review the method to make sure that changes were not inadvertently made to the program. The program calls for the samples to reach a pressure of 70 psi in less than ten minutes and then hold that pressure for 10 additional minutes.

10.13.1 From information received from the manufacturer of the microwave, the pressure of 70 psi is said to place the samples in the correct temperature range specified by Method 3015. That is, the samples will be brought to with ± 4 degrees of 160ºC within the first ten minutes, and allowed to slowly rise to 165ºC-170ºC for the second ten minutes.

10.14 Verify that the fume hood is turned on and start the program.

10.15 If the tray of samples has reached 70 psi in the specified time frame, a reversed type "TAP P" will be displayed in the microwave's LCD.

10.16 If the "TAP P" is not displayed when the program is completed, the batch will need to be cooled to room temperature and the batch redone with a fewer number of vessels per tray.

10.17 If the "TAP P" is displayed, close the transparent value in the pressure monitored sample, and remove the tray from the microwave, and place in a fume hood to cool.

10.18 After the vessels have cooled to room temperature, remove the vent tubes from the covers and the transparent valve from the pressure control cover.

10.19 Reweigh the vessels and record the weights in the "final weight of vessel" column of the microwave digestion logbook.

10.19.1 If the difference in weights from the initial weight of vessel to the final weight of vessel is greater than 5g, the sample must be discarded and redigested. This indicates a loss of sample through the rupture membrane. Re-evaluate the sample and

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consider digesting by the appropriate hot plate method if the sample cannot be digested in the microwave.

10.19.2 If the difference in weights from the initial weight of the vessel to the final weight of the vessels is less than or equal to 5g, the "final volume" column can be filled out in the microwave digestion logbook. The final volume is the total volume of sample plus nitric acid plus any spike solutions added. (See Sections 8.4 and 8.5 for final volumes of spiked samples.)

10.20 Carefully uncap and vent each sample in a fume hood.

10.21 Transfer the sample into an 8 fl-oz. Polyethylene digestion bottle that has been pre-rinsed with 10% Nitric Acid and D.I water. Label the bottle with sample number, digestion type, method number, preparation factors, and batch number.

10.22 If the sample contains particulates that may be large enough to clog the nebulizer, filtration of the sample through a 10% HNO_3 and reagent water rinsed Whatman #2 filter paper will be necessary. Do not filter with any additional amount of reagent water, as this will change the preparation factors for the sample. The filtration may be performed directly into a digestion bottle that has been rinsed and labeled as noted in Section 10.21.

11 Calculations

11.1 Not applicable to this method.

12 Reporting Results

12.1 Verify that all required information has been entered into the digestion logbook.

12.2 Open the production Horizon LIMS, and go to "batch posting" under the Operations TAB. Enter the Horizon Batch Number (HBN), which corresponds to MDIG digestion batch. When the batch opens, the samples will already have default initial volumes of 45 mL and final digestate volumes of 50 mL. These volumes will need to be adjusted to match the volumes in the microwave digestion logbook. Remember that the final digestate volume for all spiked samples is 51 mL.

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12.3 Once the sample prep factors have been updated, the analyst initials and final prep date and time need to be entered in their appropriate fields.

12.4 When entering data into Horizon LIMS do not round off results; Horizon will automatically perform rounding appropriate to the method.

12.5 Double check that all information entered in steps 12.1 through 12.3 is correct, have information reviewed by second analyst, and hit the save TAP in the upper left corner of the Horizon page. Once the batch is saved, the prep factors are applied to the samples, the META analysis code becomes available for the Metals Dept.)

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this

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method.

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Appendix A

Microwave Digestion Logbook

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EPA Method: 3015	Date of Microwave Power Check:_____			Date	Time	Tech
ALSI SOP: 09-3015	Date of Microwave Calibration:_____		Start	_____	_____	_____
Microwave ID Number:ZR8053	Was pressure reached in	YES: _____	Finish	_____	_____	_____
Batch Number:_____	time required by method?	NO: _____				
Batch Number:_____	T - TCLP	M - 6020				
Batch Number:_____	S - SPLP	W - 6010B				
Batch Number:_____	A - ASTM					

Position #	Sample #	Initial Weight Of Vessel (g)	Final Weight Of Vessel (g)	Spike Amount (ul of each)	Spike Lot #	Sample Amount (45 ml)	Final Volume (50 ml)	Batch Type	Pipette ID #	QC Type
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										

Comments: 1.) Concentrated HNO_3 Lot #_____

2.)All TCLP matrix spikes are spiked prior to preservation as noted in the TCLP logbook.

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SOP Change History Sheet

Section No.	Section	Reason for Change
3.1	Interferences	SOP Update 05/1/205
3.2	Interferences	SOP Update 05/12/05
4.6	Safety	SOP Update 05/12/05
5.5	Apparatus and Materials	DOD Audit Response 05/12/05
5.6	Apparatus and Materials	DOD Audit Response 05/12/05
6.2	Reagents	SOP Update 05/12/05
6.3	Reagents	SOP Update 05/12/05
6.4	Reagents	SOP Update 05/12/05
8.2	Quality Control	SOP Update 05/12/05
8.3	Quality Control	SOP Update 05/12/05
8.4	Quality Control	DOD Audit Response 05/12/05
8.4.1	Quality Control	SOP Update 05/12/05
8.4.2	Quality Control	SOP Update 05/12/05
8.5	Quality Control	SOP Update 05/12/05
8.7	Quality Control	A2AL Audit Response 04/21/05
8.8.	Quality Control	A2LA Audit Response 04/21/05
8.8.1	Quality Control	A2LA Audit Response 04/21/05

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SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
8.9	Quality Control	DOD Audit Response 05/12/05
8.10	Quality Control	DOD Audit Response 05/12/05
10.1	Procedure	SOP Update 05/12/05
10.3	Procedure	SOP Update 05/12/05
10.4	Procedure	SOP Update 05/12/05
10.6	Procedure	SOP Update 05/12/05
10.7	Procedure	SOP Update 05/12/05
10.13.1	Procedure	SOP Update 05/12/05
10.14	Procedure	SOP Update 05/12/05
10.17	Procedure	SOP Update 05/12/05
10.21	Procedure	SOP Update 05/12/05
12.1	Reporting Results	SOP Update 04/21/05
12.2	Reporting Results	SOP Update 05/12/05
12.3	Reporting Results	SOP Update 05/12/05
12.4	Reporting Results	SOP Update 05/12/05
Revision 7: 2/20/2006		
1.4	Scope and Application	Added as per DoD audit
2.1	Summary of Method	Added time parameters

SOP Change History Sheet (continued)

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Section No.	Section	Reason for Change
4.2	Safety	Added statements of MSDS availability
6.2	Reagents	Revised expiration dating
7.2	Glassware Cleaning	Added storage instructions
7.3	Glassware Cleaning	Added contamination considerations
8.2.1	Quality Control	Added ongoing DOC proficiency
8.11	Quality Control	MDL performance frequency
10.2	Procedure	Note added about microwave service contract
12.4	Reporting Results	Data entry instructions
12.5	Reporting Results	Review by second analyst
16	Troubleshooting	Section added as per DoD audit

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Standard Operating Procedure for Chain of Custody Entry

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Steve Smith, Date
Sample Receiving

_____ _____
Deborah J. Wood, Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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1 Scope and Application

1.1 This standard operating procedure addresses the entry of chain-of-custodies for all customers of ALSI. This document shall serve as a guideline to entering the chain-of-custody when the sample(s) are received in the Sample Receiving Department.

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 Individual projects may override criteria listed in this SOP.

2 Summary of Method

2.1 Enter your user name and password to access the Horizon LIMS.

2.2 From the Samples Menu select Login, then select By Container.

2.3 Access the "sample login" section and enter all data pertaining to the individual sample such as date/time sampled and analyses required.

3 Interferences

3.1 Not Applicable.

4 Safety

4.1 Samples may contain compounds and preservatives that are significant health hazards. All possible steps should be taken to limit analyst contact with these samples. The minimum personal protective equipment requirements when handling samples are PVC gloves, safety glasses, and a fully-buttoned lab coat. This PPE should reduce the possibility of contact to a safe level, but the analyst should not limit themselves to these PPE requirements.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available in hard copy in the QA office and electronically on the ALSI network.

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5 Apparatus and Materials

5.1 Laboratory computer station having access to the current version of Horizon; Horizon 9.0 is in use at the time of SOP 19-COC, revision 4.

6 Reagents

6.1 Not Applicable

7 Instrument Calibration

7.1 Not Applicable

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 An Initial Demonstration of Capability shall be documented as follows:

8.2.1 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Sample Login." See Appendix C.

8.2.2 Corrective action for DOC failure:

8.2.2.1 Repeat training shall be performed until all forms are successfully completed.

8.2.2.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.3 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation, and Handling

9.1 Not Applicable

10 Procedure

10.1 Accessing Horizon

10.1.1 Double-click on the Horizon icon on the desktop menu. The Horizon login screen will appear.

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10.1.2 Enter your user name (first initial and last name) and password (password provided to the IT department.) The Horizon main menu will appear.

10.2 Creating Workorders

10.2.1 From the main menu select "Samples".

10.2.2 Select "Login".

10.2.2.1 Select "By Container"

10.2.3 From the Client field, click on the LOV (List Of Values) and select the client from the list.

10.2.4 Select the "Profile" field and click on the LOV (List of Values) icon.

10.2.5 Select the "Profile" field and click on the LOV (List of Values) icon.

10.2.6 The list shown will be only the profiles available for the client you have selected. Scroll down the list and pick the profile that best matches the COC you are logging in.

10.2.6.1 Profiles for DEP reportable drinking water samples will be identified by the 7 digit DEP identification number.

10.2.7 Selecting the profile will fill in all client information on the "Customer Login" screen. This information includes the client number and name, the type of deliverable, the report format, the sequence, the status, charges, the earliest due date, and the date created.

10.2.7.1 The type of deliverable can be changed if the COC requests a type different from the listed in the profile. Options under the LOV menu include: CM (commercial w/o deliverable), CT (commercial timeframe invoice), IL (invoicing at large), CQ (commercial QA/QC package), and SD (EPA sample deliverable group).

10.2.7.2 The report format can be changed if the COC requests a type different from that listed in the profile. Options under the LOV menu include: 40CFR (40 CFR report deliverable), SDWA (SDWA deliverable), Standard (standard

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deliverable), and UCMR (UCMR EDD deliverable).

10.2.8 Under "Work ID" field, type in a keyword(s) that will identify that project. These keywords can be identifiers such as "monthly DEP", weekly wastewater, etc. or they can be project or job numbers provided by a specific client. You can also press enter from this field and the computer will fill in the default workorder ID associated with the profile you have chosen. The workorder ID field must be blank for the default ID to work.

10.2.9 The PO field will be empty at this point. If a purchase order number is available enter it her. Unless the client has a PO that is used consistently, the PO in the "case" can be saved in the "Client Info". If the PO is stored in the client profile, the PO field will be filled in by the LIMS.

10.2.10 From the Collector field click on the LOV (List Of Values) Icon. A list will appear. Begin typing the last name of the collector, this will begin reducing the list of names making it easier to find the collector you are looking for.

10.3 Sample Login

10.3.1 From the Sample ID field, click on the LOV (List Of Values) Icon. If any valid ID's are stored in the client profile, they will appear in a list. If no valid ID's are available, the user can manually type in a Sample ID. Refrain from using abbreviations, all small cases or all upper case.

10.3.2 The "Phone", "Report to", "PO", "Location", "Description", "Rec Codes", "Chain", "Original", "Keywords", and "Paired" fields are empty fields.

10.3.3 Enter the date and time collected.

10.3.4 The "Matrix" field is filled in according to the profile, but can be changed if necessary. A list of matrix options can be seen by accessing the LOV menu for the "Matrix" field.

10.3.5 Enter the date and time received. The date and time received is the date and time the last signature on the COC was completed upon receipt by the sample receiving department.

10.3.6 The "Type" field and "Mgr" fields are filled in according to the profile.

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10.3.7 The "Turn" field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the "Turn" field. When selecting an option, always pick one expressed as "Workdays from Receipt".

10.3.8 The "Priority" field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the "Priority" field.

10.3.9 Click on the "Load Line" button from the top of the Horizon Screen. A list of available testing will be displayed associated with the client and profile chosen.

10.3.9.1 Line Item 1 for the profile will initially appear on the first line of the "Line Item" section. Click on the "Line Item"

10.3.9.2 All the line items available under that profile will appear. Line items can be a single test or a group of several different tests.

10.3.9.3 Select a line item that includes tests codes necessary for this sample. Click on "Set Line Item" Unchecking any tests will cause that test or tests not to load in the workorder.

10.3.9.4 You can continue to add testing from the list by "appending line items. Appending will add to what has already been loaded into the workorder. Setting A Line will cause everything loaded in the workorder to be replaced. Select the Line Item you wish to append then, click on "Append Line Item" to add these codes to the test codes already selected. Again unchecking any test will cause that test not to load.

10.3.9.5 Continue in this manner until all test codes needed are selected.

10.3.9.6 Once all test codes are loaded that are needed, click on OK on the Line Items window.

10.3.9.7 If a needed test code is not included as a line item, it can be entered manually. In the next unused Type field key in the type of container, or click on the LOV Icon to select from a list.

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10.3.9.8 Next key in the preservative or click on the LOV Icon and select the preservative from the list.

10.3.9.9 In the CC field key in "OK"

10.3.9.10 In the Count field key in the number of containers received.

10.3.9.11 Click in the ACODE 1 Field and then click on the LOV Icon and select the test code you are trying to add. You can continue to add additional testing to that bottle by going to the next ACODE to the Right. There are 16 available ACODES on a single line. You more is needed for a single bottle continue on the next line making sure The "Type" field remains blank. When the LIMS sees a blank type field, it automatically associates the testing for that line with the bottle listed above that line.

10.3.9.12 Once all test codes are entered, and all bottles are correct, click on "Save".

10.3.9.13 If auxiliary data is required for the sample, such as for DEP reportable samples, the auxiliary data field will appear.

10.3.9.14 Auxiliary data information will include: 1) Raw or Finished Water; Level of Compositing (Usually 0); Source (Surface (1), GUDI (2), or Groundwater (3); Start of Sampling Period; and End of Sampling Period.

10.3.9.15 Once the Auxiliary Data is entered, click "Save".

10.3.9.16 A "Lab ID" number will be generated, and unique bottle IDs will also be generated for each container, and the sample will be saved, and labels will print.

NOTE: If a second sample is included on the workorder, the LIMS will advance to the next sample. Proceed starting with Section 10.3.1. Information from the first sample will be carried over to the second and can be saved as is or changed. DO NOT changed the Work ID Field. If this field is altered in any way, the LIMS will begin a new workorder.

10.3.9.17 To begin a new workorder simply start from Section 10.2.3 and remember that you must changed the Work ID field to clear the

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WO number that the LIMS last assigned. You can also hold down the shift key while pressing F7 to clear the enter Container Login Screen the begin again from section 10.2.3. This is probably the "safest" way until you are comfortable with changing workorders.

10.4 Creating DEP Sample Identifier

10.4.1 From the Horizon main menu select the "Clients" icon.

10.4.2 Select "Setup/Edit"

10.4.3 Select the profile the DEP identified is to be added to.

10.4.4 Select "Valid ID" (Double check)

10.4.5 Add the new DEP identifier.

11 Calculations

11.1 Not Applicable

12 Reporting Results

12.1 Not Applicable

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

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15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to the maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this method.

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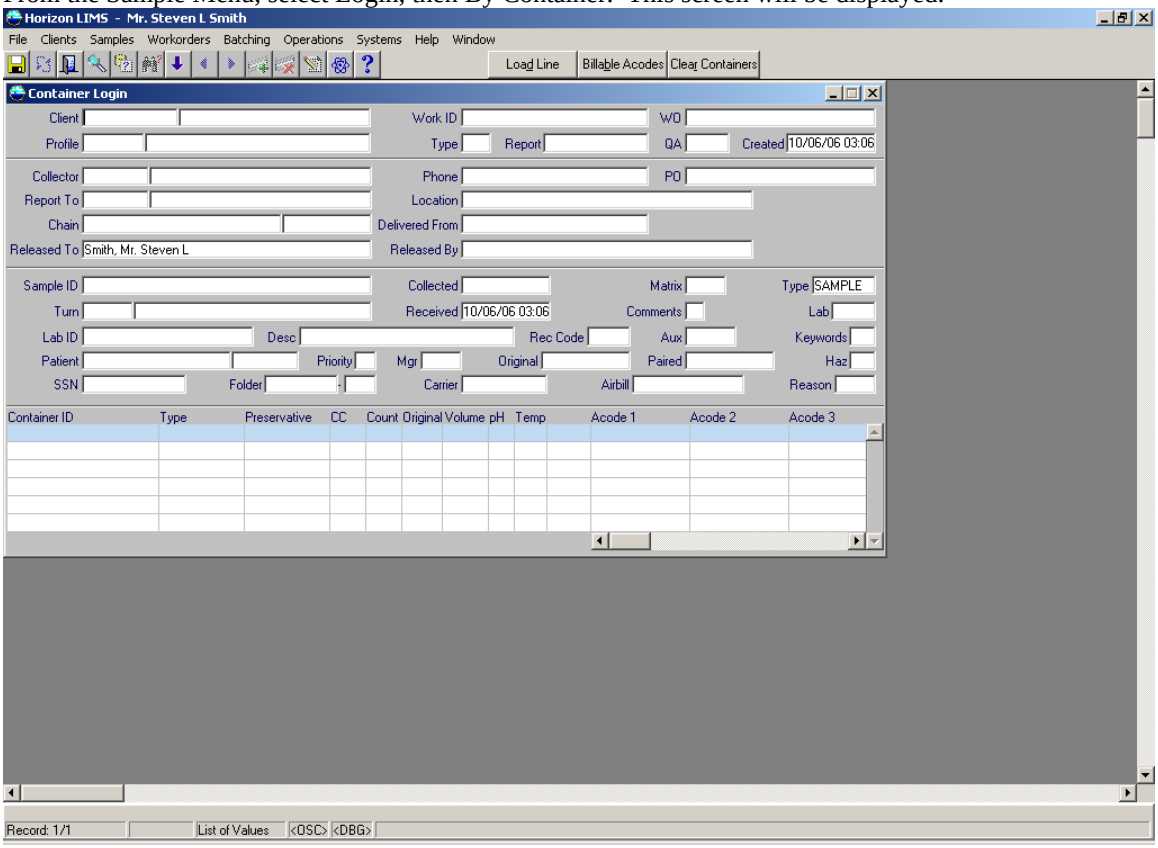
APPENDIX A

From the Sample Menu, select Login, then By Container. This screen will be displayed:



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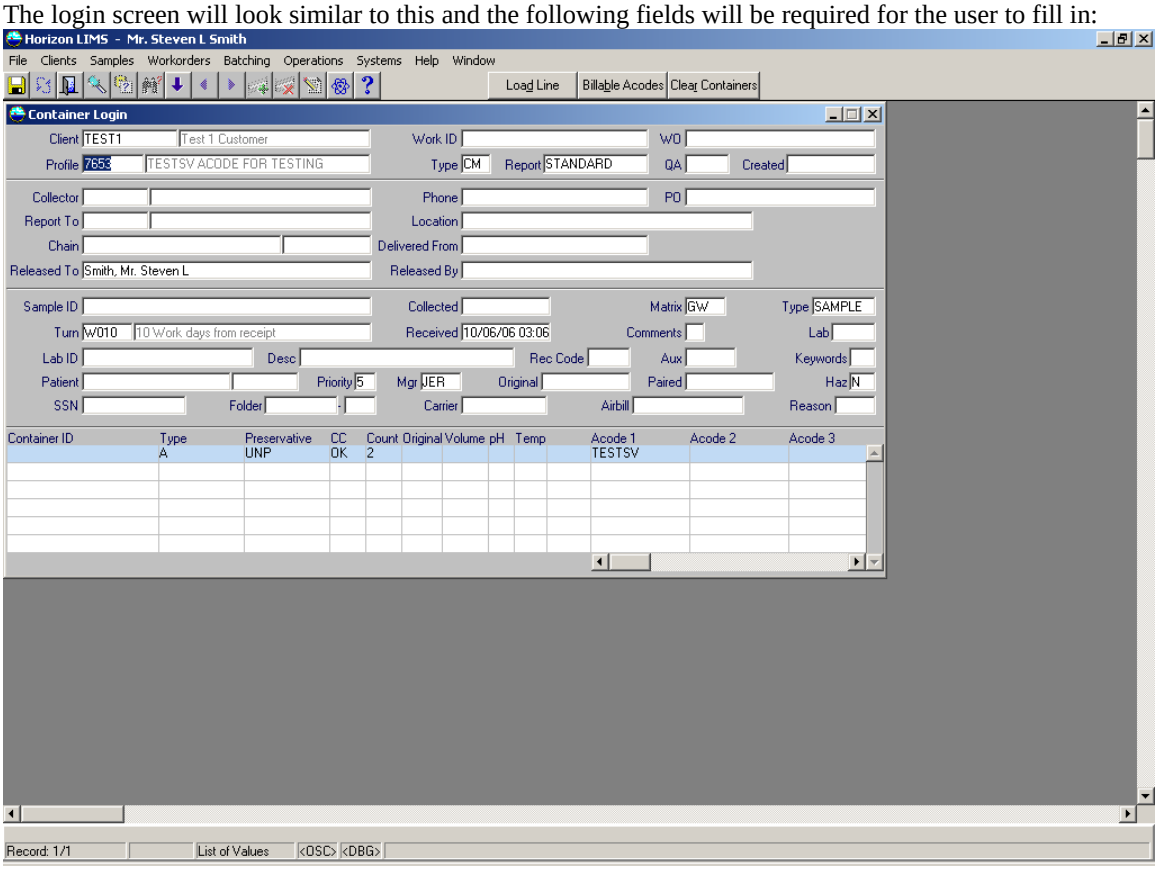
In the Client Field, Click on the List Of Values button. The client for the workorder will be displayed here. The login screen will look similar to this and the following fields will be required for the user to fill in:



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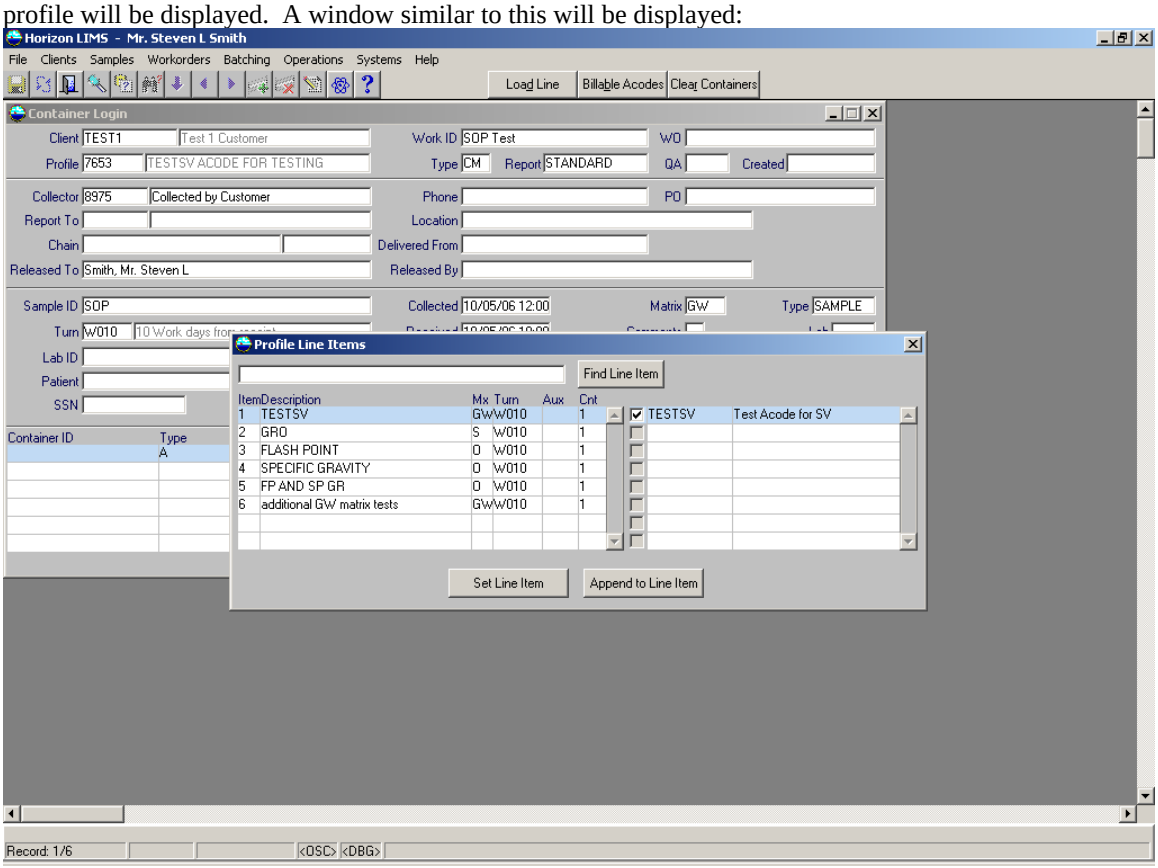
When the user is ready to add testing, simply click on the Load Line button. All available testing for the client and profile will be displayed. A window similar to this will be displayed:



From the Profile Line Items window, select the line item that best matches the COC being worked on. If a line item contains more testing then what is requested, simply uncheck what isn't to be added to the workorder. Click on the Set Line Item button. If additional line items are to be appended, simply click on the line item to be appended, uncheck any testing not needed to be added and click Append to Line Item.

NOTE: Clicking on the Set Line Item Button replaces everything.

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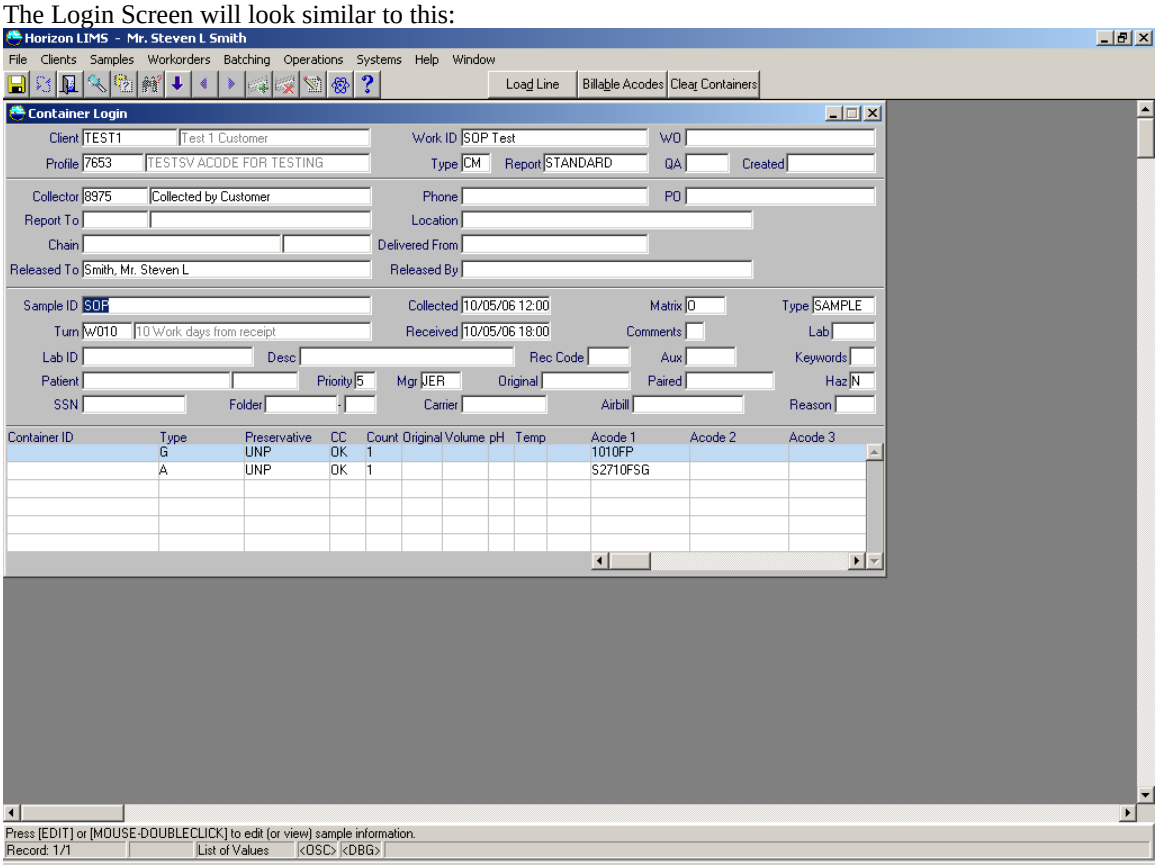
The Login Screen will look similar to this:



At this point the user can manually change a bottle or preservation for any bottle listed.
Once the user is satisfied to save and print labels, Click on the save button in the upper left corner of the login screen.

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APPENDIX B

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Sample Log-in

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-COC and signed the concurrence form.	_____	_____
2) Analyst can access the Horizon LIMS.	_____	_____
3) Analyst can create a new workorder.	_____	_____
3) Analyst knows how to select a client profile.	_____	_____
4) Analyst knows how to change deliverable type and report format if necessary.	_____	_____
5) Analyst knows how to access the sample log-in screen.	_____	_____
6) Analyst knows how to select a sample collector.	_____	_____
7) Analyst knows how to select a sample ID, including an ID for a DEP reportable sample.	_____	_____
8) Analyst knows how to enter the date/time collected and received.	_____	_____
9) Analyst knows how to change the sample matrix if necessary.	_____	_____
10) Analyst knows how to modify the priority and turn-around fields if necessary.	_____	_____
12) Analyst knows how to add test codes using the "set line item" function.	_____	_____
13) Analyst knows how to append line items.	_____	_____

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Rev. 5/05

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	Analyst	Trainer
14) Analyst knows how to add test codes manually that are not included in line items.	_____	_____
15) Analyst knows how to enter auxiliary data.	_____	_____
16) Analyst knows how to create DEP sample identifiers.	_____	_____
17) Analyst demonstrated this ability on at least five separate projects/workorders, one of which is a DEP sample.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date
_____	_____	_____
Supervisor Name	Supervisor Signature	Date

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SOP Change Summary

Section No.	Section	Reason for Change
Revision 4: 11/22/2006:		
1.2	Scope and Application	Added references to the most current NELAC standards
1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
5.1	Apparatus and Materials	Revised reference to a specific Horizon version to read "current"
8.1	Quality Control	Added standard verbiage concerning ALSI QA Plan
8.2	Quality Control	Added DOC requirements
8.2.2	Quality Control	Added DOC failure corrective action
8.3	Quality Control	Added comment concerning ongoing proficiency
16	Troubleshooting	Added section
A	Appendix	Updated appendix
B	Appendix	Deleted screenshots; added training documentation

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Standard Operating Procedure for Disposal of Samples, Extracts, Digestates, and Leachates

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Susan Magness, Date
Quality Assurance Manager

_____ _____
Ray Martrano, Date
Laboratory Manager

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8 Quality Control 4

9 Sample Collection, Preservation and Handling 4

10 Procedure 4

SOP Concurrence Form 16

1 Scope and Application

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1.1 The purpose of this Standard Operating Procedure (SOP) is to document the procedures for disposing of all samples, extracts, leachates, and digestates.

2 Summary of Method

2.1 Samples are mainly disposed of by the Sample Receiving Department, but all departments dispose of waste in some form or another.

2.2 There are four categories of waste for disposal

2.2.1 Non-hazardous aqueous waste (which can be samples, extracts, leachates, or digetates)

2.2.2 Harzardous aqueous waste (which can be samples, extracts, leachates, or digetates)

2.2.3 Non-hazardous non-aqueous waste (which can be samples, extracts, leachates, or digetates)

2.2.4 Harzardous non-aqueous waste (which can be samples, extracts, leachates, or digetates)

3 Interferences

3.1 Not applicable.

4 Safety

4.1 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and PVC gloves. Tyvek suits, dust masks, or respirators may be required depending on the waste being handled (hazardous waste).

5 Apparatus and Materials

5.1 pH monitoring system

5.2 55 gallon drums

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6 Reagents

6.1 Not applicable.

7 Instrument Calibration

7.1 Not applicable.

8 Quality Control

8.1 Not applicable.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 As noted in the ALSI SOP 19-Rec/Han, Section 10.8, samples are stored at least two weeks following completion of the last analysis on the lab report, with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations, such as USACE samples requiring 6 month storage, are handled project by project. This also applies to extracts, leachates, and digestates (from this point on to be noted as waste in this SOP).

10.2 Prior to any disposal, a Sample Discard Report is printed from the ALSI LIM system. The date used for disposal is two weeks after the disposal date. Data from the LIMS (laboratory reports) are also used to determine if the waste is hazardous or non-hazardous.

10.3 The non-hazardous aqueous waste is disposed of using pre-approved drains. The waste created by this disposal goes into a pH monitoring system. The system adjusts the pH level to within the limits allowable by our local POTW. After this adjustment, the waste is disposed into the local septic system.

10.4 The non-hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A pre-determined disposal company disposes the 55-gallon drums.

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10.5 The hazardous aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company disposes of the 55-gallon drums. The individual departments dispose of the majority of this waste as it is created.

10.6 The hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company then disposes of the 55-gallon drums.

10.7 All waste is disposed of in accordance with all federal, state, and local regulations.

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Document Title: Standard Operating Procedure for Disposal of Samples, Extracts, Digestates, and Leachates

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Susan Magness, Date
Quality Assurance Manager

_____ _____
Ray Martrano, Date
Laboratory Manager

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SOP Concurrence Form 16

1 Scope and Application

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2.2.3 Non-hazardous non-aqueous waste (which can be samples, extracts, leachates, or digetates)

2.2.4 Harzardous non-aqueous waste (which can be samples, extracts, leachates, or digetates)

3 Interferences

3.1 Not applicable.

4 Safety

4.1 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and PVC gloves. Tyvek suits, dust masks, or respirators may be required depending on the waste being handled (hazardous waste).

5 Apparatus and Materials

5.1 pH monitoring system

5.2 55 gallon drums

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6 Reagents

6.1 Not applicable.

7 Instrument Calibration

7.1 Not applicable.

8 Quality Control

8.1 Not applicable.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 As noted in the ALSI SOP 19-Rec/Han, Section 10.8, samples are stored at least two weeks following completion of the last analysis on the lab report, with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations, such as USACE samples requiring 6 month storage, are handled project by project. This also applies to extracts, leachates, and digestates (from this point on to be noted as waste in this SOP).

10.2 Prior to any disposal, a Sample Discard Report is printed from the ALSI LIM system. The date used for disposal is two weeks after the disposal date. Data from the LIMS (laboratory reports) are also used to determine if the waste is hazardous or non-hazardous.

10.3 The non-hazardous aqueous waste is disposed of using pre-approved drains. The waste created by this disposal goes into a pH monitoring system. The system adjusts the pH level to within the limits allowable by our local POTW. After this adjustment, the waste is disposed into the local septic system.

10.4 The non-hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A pre-determined disposal company disposes the 55-gallon drums.

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10.5 The hazardous aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company disposes of the 55-gallon drums. The individual departments dispose of the majority of this waste as it is created.

10.6 The hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company then disposes of the 55-gallon drums.

10.7 All waste is disposed of in accordance with all federal, state, and local regulations.

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Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Anna Milliken, Date
Laboratory Manager

_____ _____
Deborah J. Wood, Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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1 Scope and Application

1.1 The purpose of this Standard Operating Procedure (SOP) is to demonstrate the procedures for receiving and handling of all samples coming into the laboratory.

1.2 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 The sample receiving area is staffed Monday through Friday from 08:00 to 22:00. Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers.

2.2 Upon receipt of samples, sample receiving personnel complete the appropriate documentation which may include, but not be limited to, the signing of Chain of Custody forms and the completion of DC-2 Forms (EPA projects only.)

2.3 Sample receiving personnel also check for any non-conformances regarding the condition of the samples upon receipt. These non-conformances may include but not be limited to, incorrect containers, preservatives, or packaging, unacceptable sample temperatures, expired holding times, and incomplete or inaccurate documentation.

2.4 Weekend receipt of samples is discussed in Section 10.15.

3 Interferences

3.1 Not applicable to this method.

4 Safety

4.1 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available to all staff and are located in the QA office.

4.2 Incoming samples may contain a variety of unknown and potentially serious safety hazards associated with both the samples themselves and the preservatives added to them and as such, shall be treated with the utmost caution.

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4.3 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and PVC gloves.

4.4 Incoming coolers will be opened in or next to a ventilation hood when sample conditions or accompanying information dictate.

5 Apparatus and Materials

5.1 pH paper (1-6 range), purchased from VWR catalog #66777-027, or equivalent

5.2 pH paper (9-14 range), purchased from VWR catalog #34175-499, or equivalent.

5.3 5-3/4" Pasteur pipettes, purchased from VWR catalog #14672-200, or equivalent.

5.4 Potassium iodide starch paper purchased from VWR catalog #60799-008, or equivalent.

5.5 Total chlorine paper, purchased from Fisher Scientific catalog #3108T34, or equivalent.

5.6 Thermometers, capable of measuring to 1°C in the range which includes -10°C to 40°C, purchased from VWR catalog #61222-504, or equivalent. Thermometers are calibrated annually by a representative of the QA department. Thermometer information, including correction factors, is maintained in the temperature logbook.

5.7 Calibrated infrared temperature gun, capable of measuring to 1°C; HB Infrared Thermometer 900LS, purchased from VWR, or equivalent. The infrared thermometer is calibrated quarterly by a representative of the QA department. Thermometer information, including correction factors, is maintained in the temperature logbook.

5.8 Refrigerators. Each refrigerator used for storage of samples is maintained above the freezing point of water up to 6ºC. Temperatures are monitored 7 days a week and are recorded in the temperature logbook after the correction factor is applied. The refrigerators include:

5.8.1 Walk-in Refrigerator by AmeriKooler, Inc. with a Winland Electronics, Inc. temperature alarm located on upper level.

5.8.2 Walk-in Refrigerator by Bally, Serial #DX9101996-01 located on lower level.

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5.8.3 Walk-in Refrigerator by Harford, Model DL3676W487-V located on upper level

5.8.4 Refrigerator #7 - Gibson Market Master located in the GC lab.

5.8.5 Refrigerator #10 - Gibson Market Master located in the GCMS lab.

5.8.6 Refrigerator #18 – Admiral. (Used for microbiology samples only.)

5.8.7 Refrigerator #26 – Kenmore located in the GC/MS lab.

5.8.8 Refrigerator #27 – General Electric. (Used for holding volatile samples in sample Receiving before distribution to departments.)

6 Reagents

6.1 Reagent water provided by a Filson water purification system, which generates analyte-free greater than 16.0 megohm water on demand, used for the preparation of reagents.

6.2 Distilled reagent water as provided by the Corning Mega-Pure system, MP-3A and Barnstead, Fistreem II, in the Wet Chemistry department, used in non-volatile trip blank preparation.

6.3 GC/MS reagent water used in volatile trip blank preparation.

6.4 Preservatives are prepared as follows:

Note: Sample matrix and buffering capacity determine, to a large extent, the effectiveness of the added preservatives. Therefore, each appropriate bottle is pH checked upon receipt at the laboratory to insure that the preservative was adequate. All preservatives are kept at room temperature.

6.4.1 Concentrated Sulfuric acid - Sulfuric acid is purchased from VWR item #JT9681-33 or equivalent. Sulfuric acid has an expiration date of five years after receipt, and is stored in an acid chemical storage cabinet. The dispenser top is set to dispense 2 mL. For a 250 mL bottle, 1 squirt is used. For a 500 mL bottle, 2 squirts are used. For a 1000 mL bottle, 4 squirts are used.

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6.4.2 Nitric Acid (1:1) – Nitric acid is prepared by the Prep department as a 1:1 dilution and recorded in the reagent preparation logbook. The concentrated nitric acid is purchased from VWR, item #JT9598-34 or equivalent. The concentrated nitric acid has an expiration date of five years after receipt, and is stored in an acid storage cabinet. The 1:1 nitric acid is prepared by adding 500 mL of concentrated nitric acid to 500 mL of reagent water in a fume hood. The 1:1 nitric acid has an expiration date of six months after preparation and is stored in the Sample Receiving department. The dispenser top is set to dispense 2 mL. For a 250 mL bottle, 1 squirt is used. For a 500 mL bottle, 2 squirts are used. For a 1000 mL bottle, 4 squirts are used.

6.4.3 Hydrochloric acid (1:1) – Hydrochloric acid (1:1) can be purchased directly from the manufacturer or prepared by the Prep department from concentrated hydrochloric acid.

6.4.3.1 The manufacturer prepared hydrochloric acid (1:1) is purchased from VWR, item #MKH16807 or equivalent. This hydrochloric acid has an expiration date of five years after receipt, and is purchased on an as needed basis.

6.4.3.2 The hydrochloric acid prepared by the Prep department is also a 1:1 dilution. Concentrated hydrochloric acid is purchased from VWR, item #MK261246 or equivalent. The concentrated hydrochloric acid has an expiration date of five years after receipt, and is stored in an acid storage cabinet. The 1:1 dilution hydrochloric acid is prepared by adding 500 mL of concentrated hydrochloric acid to 500 mL of DI water in a fume hood. The 1:1 hydrochloric acid has an expiration date of six months after preparation and is stored in the Sample Receiving department.

6.4.3.3 For EPA method 525.2, 3 mL is used for a 1000 mL bottle. For TOCs (125 mL bottle), 1 drop is used.

6.4.4 Zinc Acetate Reagent – The zinc acetate reagent is prepared by the Wet Chemistry department, and recorded in the reagent preparation logbook. Zinc acetate is purchased from VWR, item #JT4296-1 or equivalent. The zinc acetate has an expiration date of five years after receipt, and is stored in the wet chemistry department. The zinc acetate reagent used in sample receiving is prepared by diluting 55 grams of zinc acetate in 250 mL of reagent water. The prepared zinc acetate has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 250 mL zero headspace bottle, 4 mL of zinc acetate is

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added.

6.4.5 10 N Sodium hydroxide – 10 N sodium hydroxide is prepared by the prep department, and recorded in the reagent preparation logbook. Sodium hydroxide pellets are purchased from VWR, item #JT3722-7 or equivalent. The sodium hydroxide has an expiration date of five years after receipt, and is stored in the prep department. The 10 N sodium hydroxide used in sample receiving is prepared by diluting 400 grams of sodium hydroxide in 1000 mL of reagent water. The prepared sodium hydroxide has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 500 mL bottle, 4 mL of sodium hydroxide is added.

6.4.6 EDTA Reagent – EDTA reagent is prepared by the Wet Chemistry department, and recorded in the reagent preparation logbook. Solid disodium EDTA is purchased from VWR, item #JT8993-1 or equivalent. The disodium EDTA has an expiration date of five years after receipt, and is stored in the Wet Chemistry department. The EDTA used in sample receiving is prepared by diluting 5 grams of disodium EDTA in 200 mL of DI water. The prepared EDTA has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 250 mL bottle, 2.5 mL of EDTA is added.

6.4.7 Sodium thiosulfate – Sodium thiosulfate is used directly from the manufacturer. Sodium thiosulfate is purchased from VWR, item #JT3954-1 or equivalent. Sodium thiosulfate has an expiration date of five years after receipt, and is stored in the Sample Receiving department. Refer to the current revision of the QA Plan, Appendix B-Container, Preservation, Storage, and Holding Time, for guidance in using sodium thiosulfate as a dechlorinating agent for organic samples.

6.4.8 Sodium sulfite – Sodium sulfite is used directly from the manufacturer. Sodium sulfite is purchased from VWR item #JT3722-1 or equivalent. Sodium sulfite has an expiration date of five years after receipt, and is stored in the Sample Receiving department. Refer to the Refer to the current revision of the QA Plan, Appendix B-Container, Preservation, Storage, and Holding Time, for guidance in using sodium thiosulfate as a dechlorinating agent for organic samples.

6.4.9 Ascorbic acid – Ascorbic acid is used directly from the manufacturer. Ascorbic acid is purchased from VWR, item #JT0938-7 or equivalent. Ascorbic acid has an expiration date of five years after receipt, and is stored in the Sample Receiving department. For a 40 mL vial, 25 mg of

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ascorbic is used for organic samples containing residual chlorine.

6.4.10 Ethylenediamine (EDA) Solution – EDA solution is prepared by the wet chemistry department. EDA is purchased from Aldrich, catalog # 24,072-9 or equivalent. The EDA has an expiration date of five years after receipt, or sooner if specified by the manufacturer. The EDA solution used in sample receiving is prepared by diluting 5.6 mL of EDA to 50 mL with reagent water. The prepared EDA has an expiration period of 30 days and is stored in the wet chemistry department.

7 Instrument Calibration

7.1 IR Thermometer Gun. The IR thermometer will be calibrated on a quarterly basis by the QA/QC department. Records of calibrations will be retained in the QA/QC department and the correction factors will be listed on the IR thermometer for use by the sample receiving department. The IR thermometer shall be calibrated at 0ºC, 4ºC, and 10ºC. The 0ºC correction factor shall be applied to all measurements less than 2ºC. The 4ºC correction factor shall be applied to all measurements between 2ºC and 6ºC. The 10ºC correction factor shall be applied to all measurements above 6ºC. The recorded measurement shall have the correction factor applied.

7.2 Thermometers. All other thermometers used for sample temperature measurement will be calibrated on an annual basis by the QA/QC department. Records of calibrations will be retained in the QA/QC department and the correction factors will be listed on a label affixed to the thermometer for use by the Sample Receiving department. The thermometer shall be calibrated at 0ºC, 4ºC, and 10 ºC. The 0ºC correction factor shall be applied to all measurements less than 2ºC. The 4ºC correction factor shall be applied to all measurements between 2ºC and 6ºC. The 10ºC correction factor shall be applied to all measurements above 6ºC. The recorded measurement shall have the correction factor applied.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 It is the responsibility of Sample Receiving personnel to check for and document any non-conformances regarding the condition of the samples upon receipt. These non-conformances may include but not be limited to, incorrect containers, preservatives, or packaging, unacceptable sample temperatures, expired holding times, and incomplete or inaccurate documentation. These non-conformances shall be recorded on the COC and communicated to the appropriate customer

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Service Representative so that the client can be notified. Any non-conformances associated with sample preservation shall also be recorded in the sample preservation logbook (Appendix A).

8.3 ALSI reserves the right to reject a sample upon receipt in the laboratory if any of the following conditions occur:

8.3.1 The sample is not properly identified on the sample label and/or the Chain-of-custody form.

8.3.2 The sample has exceeded the holding time for the requested analysis.

8.3.3 The incorrect preservative was used during sample collection.

8.3.4 Incorrect sampling protocols were used during sampling.

8.3.5 Improper sample container was used.

8.3.6 Insufficient sample is present to perform the requested analysis.

8.3.7 Improper storage or transport of sample has occurred prior to receipt.

8.3.8 Excessive amount of sample has been collected or other conditions exist which would make disposal difficult.

8.3.9 Excessive air bubbles are present in samples requiring zero headspace.

8.4 An Initial Demonstration of Capability shall be documented as follows:

8.4.1 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Checking Sample Preservatives." See Appendix E.

8.4.2 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Measuring Cooler Temperatures." See Appendix E.

8.4.3 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Receiving Samples." See Appendix E.

8.4.4 Reading, understanding and acceptance of the contents of this SOP shall be documented through the signing of the 19-Rec/Han Concurrence form.

8.4.5 Corrective action for DOC failure:

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8.4.5.1 Repeat training shall be performed until all forms are successfully completed.

8.4.5.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.5 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 The Sample Receiving area is staffed Monday through Friday from 08:00 to 22:00. Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers.

10.1.1 Samples received from a carrier (Fed-Ex, UPS, etc.) shall be accepted through a signature by a laboratory employee upon receipt at the facility and taken to the Sample Receiving area.

10.1.2 ALSI field personnel shall deliver all samples to the Sample Receiving area.

10.1.3 Walk-in clients and other client's field personnel shall be directed to the Sample Receiving area to deliver their samples.

10.2 All samples shall be accompanied by a Chain of Custody. An example of ALSI's Chain of Custody is included in Appendix C. All information required in the gray shaded area of the ALSI Chain shall be filled in by the client/sampler. If the sample is accompanied by a Chain of Custody from another source, the following information shall be included: client information (name, address, contact person, and phone number), sample(s) description, date/time sampled, analyses requested, date/time results required, PWSID information (if applicable), and any special deliverable or handling instructions (if applicable). The Chain of Custody shall also be signed by each individual having possession of the samples since collection (both when they receive the sample and when they relinquish it). A representative of the sample receiving department shall sign the Chain of Custody when the samples are received at the laboratory. If a walk-in customer does not have a Chain of Custody, one must be completed before samples will be accepted.

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10.2.1 If a Chain of Custody references microbiological samples that require immediate attention due to holding time restrictions, the Chain of Custody may also be signed by a representative of the microbiology department. In signing the Chain of Custody, the representative from the microbiology department shall follow the same procedures as sample receiving personnel.

10.3 The condition of the samples at the time of receipt must be documented.

10.3.1 If the sample is from an EPA project, a DC-2 form (Appendix D) must be completed to document the condition of the samples at the time of receipt.

10.3.2 Any other checklist or cooler receipt form provided by a client must be completed in its entirety.

10.3.3 The condition of all other samples at the time of receipt shall be documented in the receipt information section of the ALSI Chain of Custody. If the Chain of Custody is from another source other than ALSI, a receipt information label shall be affixed to the Chain to record this information.

10.3.4 If the project requires an internal Chain of Custody, this procedure must be initiated by the Sample Receiving Department at login. Refer to the Legal Chain of Custody SOP (99-LCOC) for details.

10.3.5 The appropriate customer service representative shall be notified of non-conformances so that the client can be contacted and a decision made whether to run the sample as received or to resample.

10.4 The temperature of the cooler/container must be taken at the time of sample receipt.

10.4.1 If a temperature blank is provided with the cooler, a calibrated mercury thermometer shall be used to determine the cooler temperature. The correction factor as discussed in Section 7.2 shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.

10.4.2 If a temperature blank is not provided, the temperature of the sample is taken with a calibrated IR gun held no more than one inch away from the sample bottle or temperature blank. The reading must be taken directly

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through the side of the bottle. It shall not be taken through the lid or through any label, etc., adhered to the bottle. The correction factor as discussed in Section 7.1 shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.

10.4.3 If this protocol is not feasible, a small amount of sample shall be decanted from a non-volatile bottle and a calibrated thermometer shall be used to read the temperature of the sample as discussed in Section 10.4.1.

10.4.4 If the temperature reading does not fall within the acceptable range (above the freezing point of water up to 6ºC), multiple readings shall be taken using at least two other bottles contained in the same cooler to insure that a representative reading has been obtained. All readings shall then be recorded along with the identification of the bottles from which they were taken.

10.4.5 Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

10.4.6 All samples shall be observed for receipt on ice. The observation shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain of Custody. The individual noting this fact shall also initial the corresponding line.

10.4.7 Samples shall remain on ice or be transferred to an appropriate refrigerator until processing. Microbiological samples shall be stored in Refrigerator 18 and volatile samples shall be stored in Refrigerator 27 to prevent contamination until transport to the appropriate departments.

10.5 All samples shall be checked for custody seals. The presence of custody seals shall be noted on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. If custody seals are present, the presence or absence of tampering shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Details involving any tampering with custody seals shall be noted in the sample/COC comments section of the Chain.

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10.6 All samples shall be checked for breakage/leakage. The presence or absence of damage shall be noted by checking the appropriate Y/N box under "Cont. in Good Cond." in the receipt information section of the Chain and initialing the corresponding line. Details involving any breakage shall be noted in the sample/COC comments section of the Chain.

10.7 All samples shall be checked to insure that the information on the Chain of Custody agrees with the information on the container labels. This information would include sample descriptions, date/time sampled, sampler ID, and analyses requested. Whether or not the Chain of Custody and labels agree shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any discrepancies between the Chain and labels shall be specifically mentioned in the sample/COC comments section of the Chain.

10.8 All samples shall be checked to insure that the containers received are appropriate for the analyses requested and that a sufficient sample volume has been provided. This information shall be documented by checking the appropriate Y/N boxes under "Correct Containers" and "Correct Sample Volumes" in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.9 Samples contained in a septa-sealed container to be analyzed for volatile analytes, shall be checked for the appearance of headspace. Headspace is the appearance of air in a septum-lined container. A pea sized amount of air or smaller is allowable. Whether or not headspace is present shall be noted by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

All volatile samples shall be held in the Sample Receiving refrigerator designated for VOC only (Refrigerator 27) and protected from contamination until the COC is entered into the LIMS system and the identifying labels are attached to each vial. The samples will then be transported to the VOC refrigerators in the organics lab for analysis.

10.10 The pH of all acid or caustic preserved bottles (with the exception of volatile containers) shall be taken using the pH paper listed in Section 5. Bottles shall be preserved according to the SOP for Bottle Preservation (19-BOTTLE). (See the most recent revision of the QA Manual for a list of analyses that require pH testing.)

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10.10.1 The pH of the samples is measured by inserting a 5-3/4" Pasteur pipette into the sample. After the pipette is drawn out, a drop of the sample is placed on the pH paper. The pH is read using the color charts on each package of the pH strips. If the pH falls within the correct range, it is recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation logbook. (See Appendix A).

10.10.2 If the pH is not within the acceptable range, the pH is adjusted by adding the appropriate preservative (see SOP for bottle preservation). The initial pH, the amount of preservative added, the lot number of the preservative used, and the final pH are recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation logbook.

10.10.3 For samples requiring **DoD** certification, a small amount of sample shall be poured out of each bottle (excluding volatile samples) to check for the appropriate pH.

10.10.4 The following information must be recorded in the sample preservation logbook: the sample number, date received, preservative, initial pH, and the initials of the person taking the pH. In cases when the pH was adjusted, the amount of preservative added and the final pH are also recorded along with the lot of the preservative added. The logbook is generated in a Microsoft Excel spreadsheet that is protected against making any changes to the posted results. Each bottle is scanned using a Symbol Pocket PC with Pocket Excel installed.

10.10.5 Whether or not samples are preserved properly shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.10.6 If a metals bottle was acidified upon receipt at the laboratory, a copy of the preservation log shall be attached to the bottle with a rubber band when it is delivered to the department. This will notify the Metals Department of the date and time of preservation.

10.11 All samples received must be checked for holding times.

10.11.1 Particular attention shall be given to any microbiological samples

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received, especially fecal coliforms which have a holding time of 6 hours for wastewater samples and frequently arrive at the laboratory with little time remaining prior to expiration. A representative of the microbiology department shall be notified immediately upon the arrival of these samples.

10.11.2 The presence of any other short holding time tests (less than 48 hours) that require immediate attention shall also be noted. If any short holding time tests require attention, the appropriate analytical department shall be notified. A list of holding times is maintained in the most recent revision of the QA Manual.

10.11.3 Particular attention shall also be given to soil samples that require any type of volatile analysis (8260, 8021, GRO, etc.). If these samples are received as encores or in soil jars they must be extracted within 48 hours of collection. Pre-weigh vials do not need any special treatment. The procedure for processing encores and soil jars is detailed in Appendix F.

10.11.4 Any samples received more than five days after the time of collection shall be evaluated for all holding times. Some extractions and wet chemistry procedure have 7 day holding times and while these would not normally be considered “short holds”, they may be in jeopardy due to the excessive time elapsed between sampling and receipt. Analytical departments shall be notified of any such samples. Customer service personnel or a laboratory manager shall be consulted if a holding time is in question.

10.11.5 For any samples received at the laboratory with analyses that are already past hold at the time of receipt, the Chain of Custody shall be stamped with the red “EXPIRED” stamp and the expired tests noted in the stamp’s box area.

10.12 The chlorine content shall be checked for samples requiring sodium thiosulfate or sodium sulfite as a preservative or samples requesting cyanide. See the most recent revision of the ALSI QA Manual for a list of analyses that require sodium thiosulfate or sodium sulfite as a preservative.

10.12.1 In order to verify that the preservative has been added, the samples are tested for the absence of chlorine by inserting a 5-3/4" Pasteur pipette into the sample and placing a drop on KI paper for non drinking water samples. For drinking water samples, the residual chlorine is verified by using the total chlorine paper. If a color

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change to blue or violet is indicated, sodium thiosulfate must be added to the sample until the chlorine has been neutralized.

10.12.2 The presence/absence of chlorine must be documented in the sample preservation logbook file. Whether or not samples are preserved properly shall be documented on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.13 The turbidity of drinking water samples for metals analysis shall be determined by the Metals or Wet Chemistry department.

10.14 At this time, the samples are entered into the Horizon LIMS. See SOP for COC Entry (19-COC).

10.15 Labels generated by the Horizon LIMS are then affixed to the bottles and the bottles are stored as follows:

10.15.1 Volatile aqueous samples for GC are stored in Refrigerator #26 and 29 located in the GC lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator.)

10.15.2 Volatile aqueous samples for GC/MS are stored in Refrigerators #26 and 29 located in the GC/MS lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator #10.)

10.15.3 Metals samples are stored in numerical order on shelves in the Inorganic Prep Lab excluding drinking water metals samples. Drinking water metals samples that do not require a digestion are stored in the Metals lab. Dissolved metals are stored in the Metals Department.

10.15.4 Total Organic Carbon and Total Organic Halogen samples are stored in the walk-in refrigerator on the lower level.

10.15.5 Samples requiring any sort of organic extractions are stored on the right

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side of the Harford walk-in refrigerator located on the upper level.

10.15.6 All other liquid samples not mentioned are stored in the remaining two walk-in refrigerators in numerical order.

10.15.7 Solid samples are stored on the left side of the Harford walk-in refrigerator on the upper level.

10.15.8 Solid volatile samples and petroleum samples are stored in either GC or GCMS, wherever the testing is done.

10.15.9 A very limited number of samples do not require refrigeration. These are often associated with atypical analysis. Approval from a department supervisor is required to store samples without refrigeration.

10.15.10 Samples with special storage considerations like solvents or free product are stored in the prep hoods.

10.16 All samples are stored at least two (2) weeks following the completion of the last analysis on the lab report with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations are handled project by project by the appropriate customer service representative. Samples requiring **DoD** certification must be stored for 60 days.

10.17 Samples received over the weekend and during off-peak hours shall be handled in the manner outlined in Appendix B.

11 Calculations

11.1 Not applicable

12 Reporting Results

12.1 Not applicable

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the

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quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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APPENDIX A

Preservation Log available on the ALSI Network

Container Number	pH Received	Adjustment Amount Needed	Final pH	Preservation Lot Number	Residual Chlorine (+ or -)	Date	Time	Initials

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APPENDIX B

The following steps can be followed for samples received after hours and on weekends:

1. Sign the Chain of Custody (COC) in the "received by" section on the bottom of the COC form only after inspecting samples to ensure that all information on the COC matches the samples being submitted for client submission.

2. Tear off the gold copy of the COC (last page), and give it to the client submitting the sample.

3. Check the analyses required for the samples received on the COC for any analyses on the following list which require short holding times (HT). If any of these tests are required and there are no trained personnel available to complete the analysis within the holding time, contact the supervisor of the department that performs the analysis. If the supervisor cannot be reached, contact the Laboratory Manager or appropriate Customer Service Representative.

Analyze Immediately	24 Hour HT	48 Hour HT
Chlorine Demand	Hexavalent Chromium	BOD/CBOD
Turbidity	Total Coliform (30 hr)	Nitrate
Residual Chlorine		Nitrite
Total Chlorine		Orthophosphate
Odor		MBAS
Dissolved Oxygen		Osmotic Pressure
pH		Color
Sulfite		Settleable Solids
Fecal Coliform (6hr)		UV254
		Encore Extraction

4. Check the temperature of the samples as specified in Section 10.4

5. Place samples and COC form together in the lower level walk-in.

6. Leave an E-mail message for the appropriate Customer Service Representative letting them know what was received, and what actions were taken.

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APPENDIX C

Analytical Laboratory Services, Inc.
Environmental • Industrial Hygiene • Field Services
34 Dogwood Lane • Middletown, PA 17057 • 717.944.5541 • Fax: 717.944.1430

CHAIN OF CUSTODY/ REQUEST FOR ANALYSIS

ALL SHADED AREAS MUST BE COMPLETED BY THE CLIENT / SAMPLER. INSTRUCTIONS ON THE BACK.

COC #: _____ of _____
ALSI Quote #:

Client Name:
Address:
Contact:
Phone#:
Project Name/#:
Bill To:

TAT: ☐ Normal-Standard TAT is 10-12 business days. ☐ Rush-Subject to ALSI approval and surcharges.
Date Required: _____ Approved By: _____
Email? ☐ -Y
Fax? ☐ -Y No.:

***Container Type
***Container Size
*** Preservative

ANALYSES/METHOD REQUESTED

Sample Description/Location (as it will appear on the lab report)	Sample Date	Military Time	*G or C	**Matrix	Enter Number of Containers Per Analysis Requested or Field Results Below.										Sample/COC Comments
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															

Receipt Information (completed by Receiving Lab)
Cooler Temp: _____ Therm. ID: _____
No. of Coolers: _____

	Y	N	Initial
Custody Seals Present?			
(If present) Seals Intact?			
Received on Ice?			
COC/Labels Complete/Accurate?			
Cont. in Good Cond.?			
Correct Containers?			
Correct Sample Volumes?			
Correct Preservation?			
Headspace/Volatiles?			

Courier/Tracking #:

SAMPLED BY (Please Print):
LOGGED BY(signature): DATE: TIME:
REVIEWED BY(signature): DATE: TIME:

Relinquished By / Company Name	Date	Time	Received By / Company Name	Date	Time
1 SAMPLED BY			2		
3			4		
5			6		
7			8		
9			10		

Data Deliverables: ☐ Standard ☐ CLP-like ☐ NJ-Reduced ☐ NJ-Full ☐ _____ (specify)
EDDs Required? ☐ If yes, format type:
DOD Criteria Required?

State Samples Collected In:	SDWA Forms Required?
NC	yes
NJ	yes
NY	yes
PA	yes
Other:	
PWSID	

ALSI Field Services: ☐ Pickup ☐ Labor ☐ Composite Sampling ☐ Rental Equipment ☐ Other:

* G=Grab; C=Composite **Matrix: AI=Air; DW=Drinking Water; GW=Groundwater; OI=Oil; OL=Other Liquid; SL=Sludge; SO=Soil; WP=Wipe; WW=Wastewater

Copies: WHITE - ORIGINAL CANARY - CUSTOMER COPY ***Container Type: AG-Amber Glass; CG-Clear Glass, PL-Plastic. Container Size: 250ml, 500ml, 1L, 8oz., etc. Preservative: HCl, HNO3, NaOH, etc. Rev 5/06

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APPENDIX D

DC-2 Forms

Lab Name: Analytical Laboratory Services, Inc.	DAS Number:	SDG Number:
City: Middletown	State: Pennsylvania	Zip Code: 17057
Order Number:	Parameter:	

	Page Numbers From	To	Check Lab	EPA
Inventory Sheet	XXXXXXX	XXXXXXX		
SDG Narrative				
SDG Cover Sheet/Traffic Report				
QC Data				
Sample Data				
Standard Data				
Blank Data				
Raw Data				
Preparation Logs				
Clean-up Logs				
Analysis Logs				
Internal Chain of Custody Logs				
Shipping / Receiving Documents				
Telephone / e-mail Logs				
Other Records				

Organization	Lab Inventory	Region 3 Auditor	EPA Verifier
Print Name			
Title			
Date			
Signature			

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** Reference: Critical Elements for Certification of Drinking Water Laboratories for Chemistry, October 1, 1997.*

[1] In addition cool to 4ºC.
[2] The holding time for Heptachlor under this method is 7 days.
[3] Samples that have been preserved with mercuric chloride may be disposed of in at least two ways: as a hazardous waste, or by passing over an absorbent column (i.e., alumina, activated with carbon, etc.) for mercury absorption, with the effluent analyzed periodically for breakthrough. The absorbent would then be disposed of as a hazardous waste. Other techniques may be applicable.
[4]Mercuric chloride is only required in drinking water samples in which biological degradation of the target pesticides and herbicides are exhibited.
[5]No extract.

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APPENDIX E

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Checking Sample Preservatives

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst knows where to find the list of sample types that require preservation.	_____	_____
3) Analyst knows which pH paper to use for checking the pH of acidified samples and where to find it.	_____	_____
4) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for acidified samples.	_____	_____
5) Analyst knows which pH paper to use for checking the pH of basic samples and where to find it.	_____	_____
6) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for basic samples.	_____	_____
7) Analyst knows how to adjust the pH of any sample if the measured value is not acceptable.	_____	_____
8) Analyst knows which test papers to use for checking chlorine content and where to find them.	_____	_____
9) Analyst knows how to apply the sample to the test papers and how to interpret the reading for chlorine content.	_____	_____

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Rev. 5/05

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	Analyst	Trainer
10) Analyst knows how to properly complete the preservation logbook.	_____	_____
11) Analyst knows that USACE samples require a small amount of Sample to be poured out for sampling pH.	_____	_____
12) Analyst knows how to complete all information on the chain of custody regarding preservation including where to record non-conformances.	_____	_____
13) Analyst is aware that when metals samples are acidified upon Receipt a copy of the preservation log must accompany the sample to the metals prep department.	_____	_____
14) Analyst has demonstrated his/her ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date
Supervisor Name	Supervisor Signature	Date

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New Employee Orientation

Sample Receiving Training Checklist

Procedure: Measuring Cooler Temperatures

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst read and understands SOP 19-Rec/Han and signed the concurrence form.	____	____
2) Analyst knows the location of the mercury thermometer.	____	____
3) Analyst knows where to find and how to apply the correction factor for the mercury thermometer.	____	____
4) Analyst can properly take and record a cooler temperature using a provided temperature blank.	____	____
5) Analyst knows the acceptable limits for cooler temperatures are above the freezing point of water to 6 degrees C.	____	____
6) Analyst knows the location of the IR temperature gun.	____	____
7) Analyst knows how to properly use the IR gun to take a sample temperature (i.e. where to shoot the gun through the bottle).	____	____
8) Analyst knows where to find and how to apply the correction factors for the IR temperature gun.	____	____
9) Analyst knows to take multiple temperatures if the initial bottle used is outside the acceptable limits.	____	____
10) Analyst is aware that samples received same day as sampling are acceptable if evidence exists that the chilling process was begun.	____	____

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	Analyst	Trainer
11) Analyst is aware that designated refrigerators are available for "holding" micro and volatile samples during processing.	_____	_____
12) Analyst has demonstrated this ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

Analyst Name	Analyst Signature	Date
Supervisor Name	Supervisor Signature	Date

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New Employee Orientation

Sample Receiving Training Checklist

Procedure: Receiving Samples

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	____	____
2) Analyst will ensure that all samples are accompanied by a chain of custody.	____	____
3) If a walk-in client attempts to drop off samples not accompanied by a chain of custody, the analyst provides a chain for them to complete on-site.	____	____
4) Analyst receives custody of the sample(s) by signing the chain of custody upon receipt of samples, including the date and time received.	____	____
5) When samples are received with a chain of custody from a source other than ALSI, the analyst will confirm client and sample information is present.	____	____
6) Analyst checks for the presence of custody seals and whether they are intact and records all findings including any non-conformances appropriately on the chain of custody.	____	____
7) Analyst checks the sample temperature and for the presence of ice and records all findings including any non-conformances appropriately on the chain of custody.	____	____
8) Analyst checks for breakage and leakage and records all findings including any non-conformances appropriately on the chain of custody.	____	____

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	Analyst	Trainer
9) Analyst compares the information on the chain of custody to the information on the bottle labels and records all findings including any non-conformances on the chain of custody.	_____	_____
10) Analyst checks to see that the sample containers are appropriate for the analyses requested and records all findings including any non-conformances on the chain of custody.	_____	_____
11) Analyst checks all volatile containers for headspace and records all findings including any non-conformances on the chain of custody.	_____	_____
12) Analyst properly checks for preservatives and records all findings including any non-conformances on the chain of custody.	_____	_____
13) Analyst knows what analytes have short holding times and notifies the appropriate departments upon receipt.	_____	_____
14) Analyst has demonstrated this ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date
_____	_____	_____
Supervisor Name	Supervisor Signature	Date

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Appendix F

Receipt of Soil Volatile Samples in Encores or Soil Jars

Volatile soil samples received in soil jars or encores must be extracted into sodium bisulfate and/or methanol vials within 48 hours from the time of collection. Samples received with little time remaining prior to expiration require immediate attention. Proceed as follows:

1) When these types of samples are received, check the sample containers against the COC. Following procedure, note any discrepancies on the COC.

2) Log the samples into Horizon.

3) Initiate the ALSI Encore Internal COC Logbook. Even if the samples cannot be logged into Horizon for any reason, continue with this step. In such a case, use the sample description in lieu of a Horizon sample number.

4) Transfer the samples to the GC/MS laboratory along with a copy of the COC.

   NOTE: Samples must be delivered to an actual person who acknowledges receipt of the samples by initialing the Encore Internal COC Logbook. The samples cannot be left on a counter or placed in the refrigerator without documented receipt. If an individual qualified to extract encores is not available, refer to the GC/MS phone list located in sample receiving and contact an individual to come in and perform the extractions.

5) Complete the Encore Internal COC Logbook. (See Appendix G)

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Appendix G

ALSI ENCORE INTERNAL COC LOGBOOK

Sample #	Date Sampled	Time Sampled	Analysis Method	Date Received in Lab	Time Received in Lab	Received within 48 hours (Y/N)	Relinquished By	Received By	Date Delivered to Analyst	Time Delivered to Analyst	Comments

Rev. 4/03

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SOP Change Summary

Section No.	Section	Reason for Change
Revision 7:		
5.9	Apparatus and Materials	Correction to current SOP
6.0	Reagents	Correction to current SOP
10.2.8	Procedure	A2LA Audit Response
10.6-10.7	Procedure	Horizon LIMS Implementation
Revision 8:		
10.2.4	Procedure	New Jersey DEP audit response
Revision 9:		
2	Summary of Method	DoD audit response 5/13/05
4	Safety	DoD audit response 5/13/05
5	Apparatus and Materials	DoD audit response 5/13/05
6	Reagents	DoD audit response 5/13/05
7	Instrument Calibrations	DoD audit response 5/13/05
8	Quality Control	DoD audit response 5/13/05
10	Procedure	DoD audit response 5/13/05
Appendix C	Sample Collection, Containers & Preservation For Organic Contaminants	DoD audit response 5/13/05
Appendix D	ALSI Chain of Custody	DoD audit response 5/13/05
Appendix E	DC-2 Data Package Inventory Checklist	DoD audit response 5/13/05

SOP Change Summary (continued)

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Section No.	Section	Reason for Change

Revision 10: 10/06/2006

Revised refrigeration temperatures to read "…above the freezing point of water up to 6ºC…" to reflect NELAC verbiage.

1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
6.4.1, 6.4.2	Reagents	Revised volume of dispenser tops from 1 mL to 2 mL; revised number of squirts to accommodate new dispenser volume
6.4.3.3	Reagents	Revised volume added to TOC bottle
8.1	Quality Control	Added verbiage indicating that the SOP falls under the QA Plan umbrella
8.4, 8.5	Quality Control	Added initial DOC and statement about ongoing proficiency
10.10.4	Procedure	Removed recording of tests required in sample preservation logbook; added generation of logbook in a Microsoft Excel spreadsheet
11	Calculations	Added section
12	Reporting Results	Added section
16	Troubleshooting	Added section

Removed Appendix duplicating QA Plan Appendix B, Container, Preservation, Storage and Holding Times

C	Appendix	Updated COC to current form
E	Appendix	Added examples of training forms

SOP Change Summary (continued)

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Section No.	Section	Reason for Change
Revision 11: 02/05/07		
5.8.3	Apparatus and Materials	Added new walk-in refrigerator
6.1	Reagents	Added ALSI generated reagent water
6.3	Reagents	Replaced "well/tap" water with "reagent" water
6.4.10	Reagents	Added Ethylenediamine (EDA) Solution
10.2.1	Procedure	Added specific verbiage for microbial samples
10.11	Procedure	Added verbiage about sample holding times, microbial sample test times, soil samples and corrective actions for samples past holding time
10.13	Procedure	Added Wet Chem as lab for turbidity analysis
10.15.5, 10.15.7	Procedure	Added specific sample storage locations; deleted defunct refrigerators
B	Appendix	Added Fecal Coliform to "Analyze Immediately" column; Added Total Coliform to "24 Hour HT" column
F	Appendix	Added directions for the receipt of soil volatile samples in Encores or Soil Jars
G	Appendix	Added example of Encore Internal COC logbook page

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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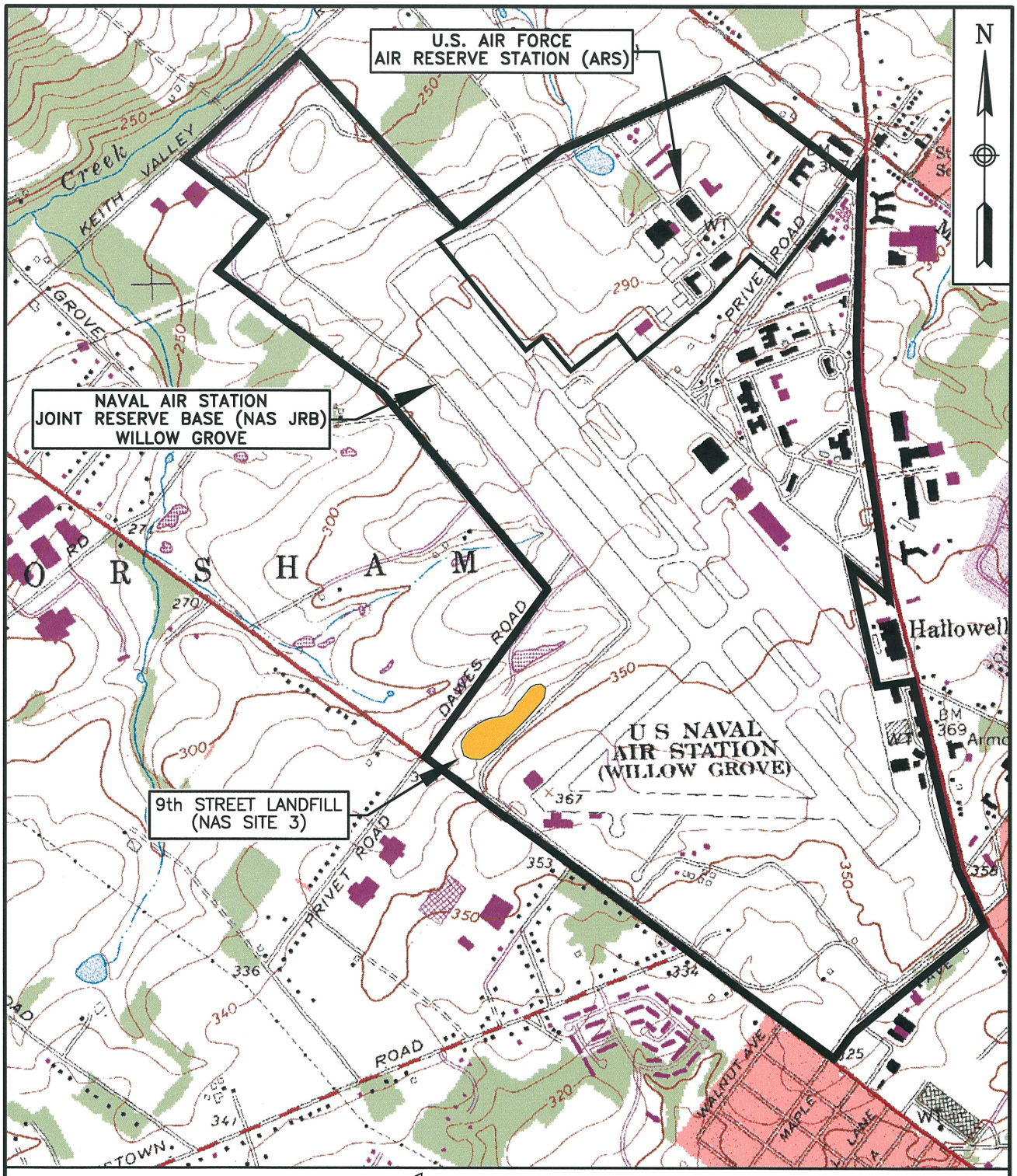
APPENDIX C

FIGURES

112G00845\0310\112G00845BM01.DWG 02/29/08 MKB



BASE MAP IS A PORTION OF THE AMBLER, PA U.S.G.S. 7.5 MINUTE QUADRANGLE MAP, DATED 1963, PHOTOREVISED IN 1983.

QUADRANGLE LOCATION

0 1500 3000

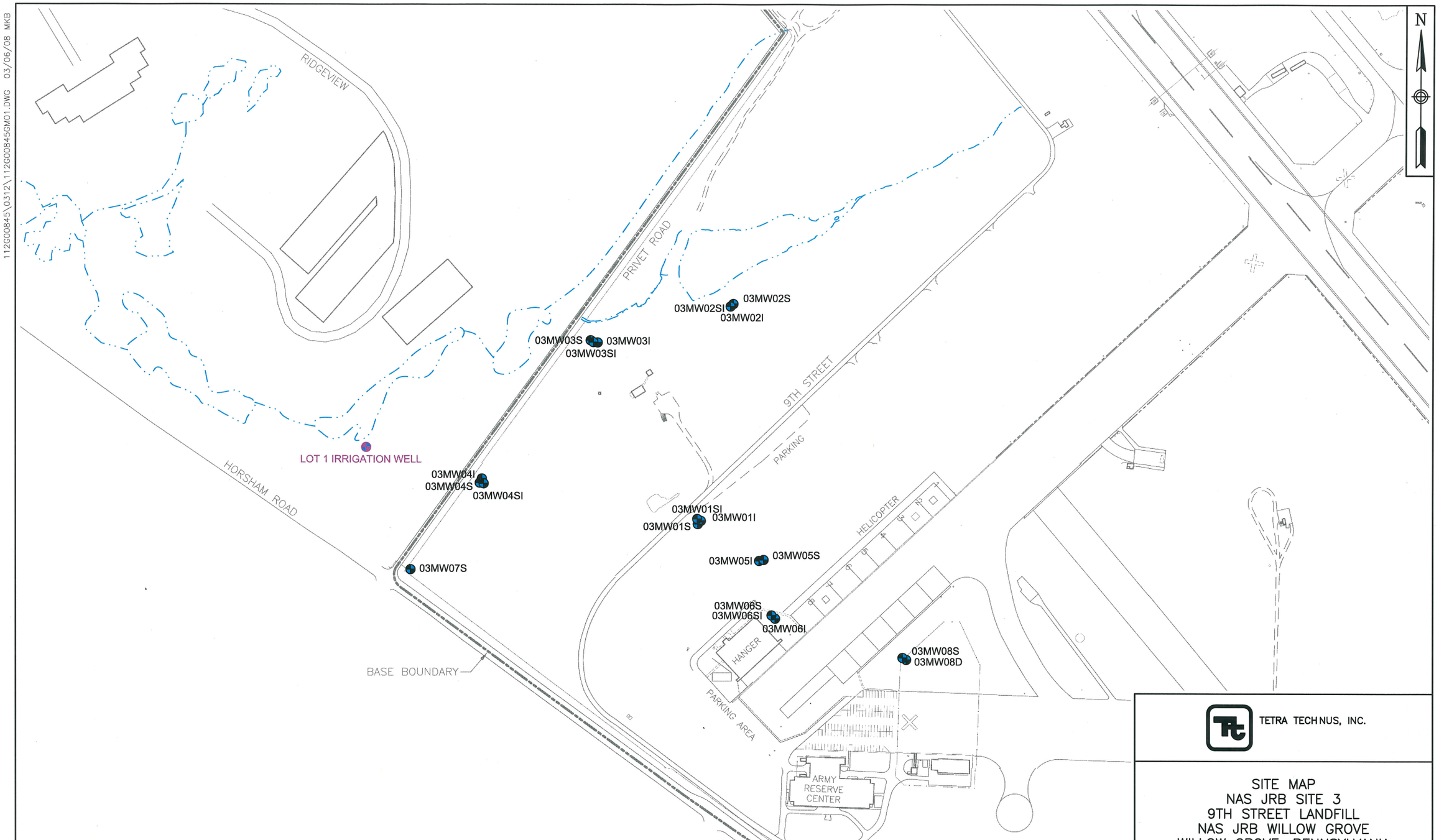
SCALE IN FEET

TETRA TECHNUS, INC.

FACILITY LOCATION MAP
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

SCALE	AS NOTED
FILE	112G00845BM01
REV	0
DATE	02/29/08
FIGURE NUMBER	FIGURE 10–1

112G00845\0312\112G00845GM01.DWG 03/06/08 MKB



0 300 600

SCALE IN FEET

LEGEND

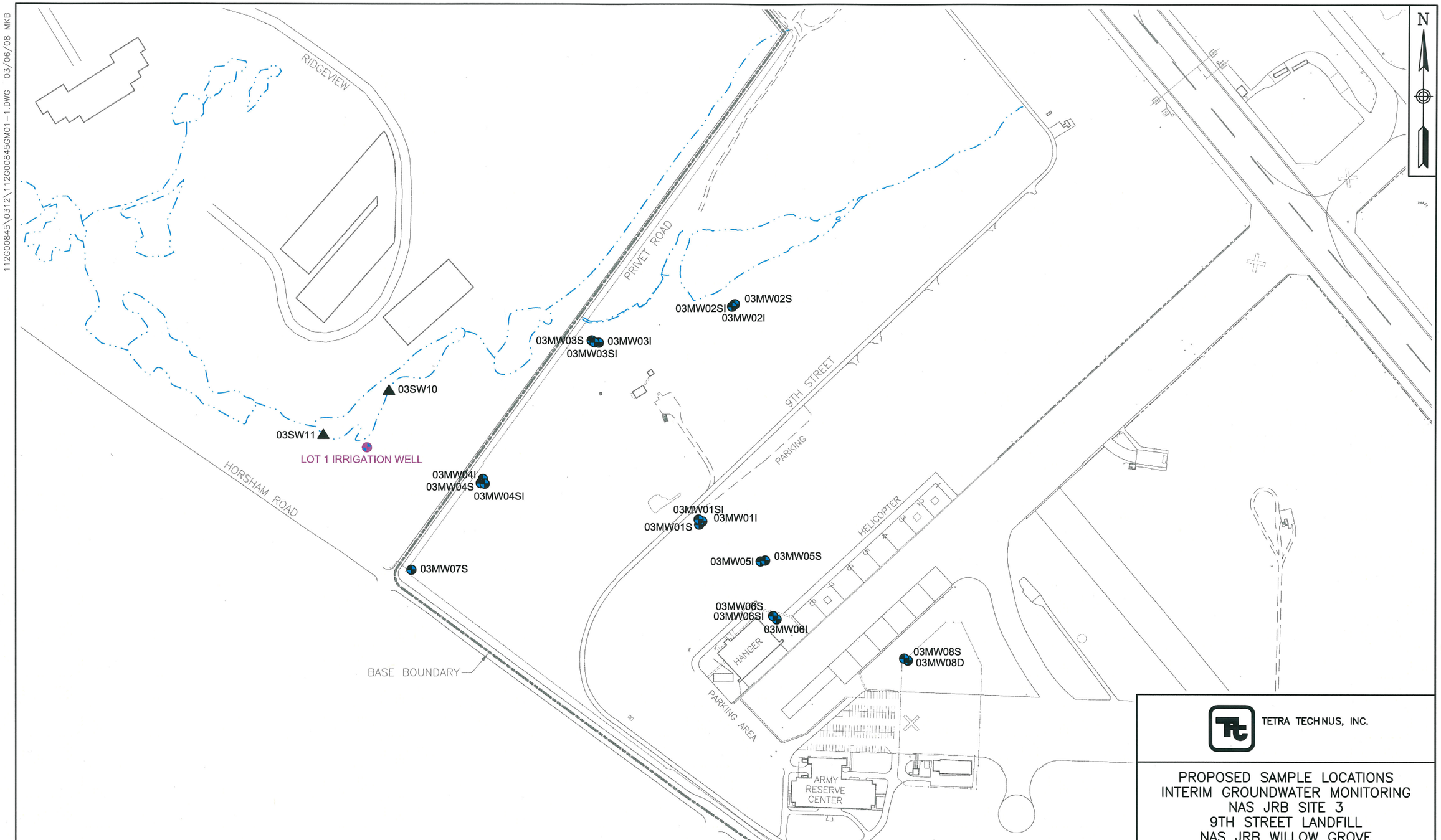
- ON-SITE MONITORING WELL LOCATION
- OFF BASE WELL LOCATION

TETRA TECHNUS, INC.

SITE MAP
NAS JRB SITE 3
9TH STREET LANDFILL
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

FILE	SCALE
112G00845GM01	AS NOTED
FIGURE NUMBER	REV / DATE
FIGURE 10-2	0 / 03/06/08

112G00845\0312\112G00845GM01-1.DWG 03/06/08 MKB



LEGEND

- ON-SITE MONITORING WELL LOCATION
- OFF BASE WELL LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION

TETRA TECH NUS, INC.

PROPOSED SAMPLE LOCATIONS
INTERIM GROUNDWATER MONITORING
NAS JRB SITE 3
9TH STREET LANDFILL
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

FILE	SCALE
112G00845GM01-1	AS NOTED
FIGURE NUMBER: FIGURE 14-1	REV: 0 / DATE: 03/06/08