Appendix A:

Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP)

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FINAL
UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE
PROJECT PLANS FOR ENVIRONMENTAL REMEDIATION SERVICES AT

AT THE FORMER
NIAGARA FALLS-BUFFALO DEFENSE NIKE BATTERY BU-34/35 EAST AURORA AND ORCHARD PARK, NEW YORK

THE FORMERLY USED DEFENSE SITE (FUDS), NO. C02NY007701

CONTRACT NO. W912DR-13-D-0013
Delivery Order No. DB01

Prepared for:

U.S. Army Corps of Engineers
New England District
696 Virginia Road
Concord, MA 01742-2751

Prepared by:

TI2E JV, LLC
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February 2016

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ABBREVIATIONS AND ACRONYMS

ARARs	Applicable or Relevant and Appropriate Requirements
ALS	ALS Environmental Laboratory
APP/SSHP	Accident Prevention Plan/ Site Safety and Health Plan
AST	Above-ground storage tanks
ASTM	American Society for Testing and Materials
BAFs	Bioaccumulation factors
BFB	Balanced Failure Biasing
BSAFs	Biota-sediment accumulation factors
^{o}C	degrees Centigrade
CAS	Chemical Abstract Service
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CHMM	Certified Hazardous Materials Manager
C-O-C	Chain-of-Custody
COPCs	Chemicals of potential concern
COPECs	contaminants of potential ecological concern
CQA	Certified Quality Auditor
CSM	Conceptual site model
CSP	Certified Safety Professional
CVAA	Cold Vapor Atomic Absorption
DOD	Department of Defense
DOT	Department of Transportation
DQO	Data Quality Objective
DRO	Diesel Range Organics
DPT	Direct Push Technology
DQCR	Daily Quality Control Reports
DUP	Laboratory Duplicate
ERAGS	Ecological Risk Assessment Guidance for Superfund
ERS	Environmental Remediation Services
FS	Feasibility Study
FUDS	Formerly Used Defense Site
GC/MS	Gas Chromatograph/ Mass Spectrometer
GRO	Gasoline Range Organics
GSA	General Services Administration
GW	Groundwater
HCl	Hydrochloric acid
HDPE	High density polyethylene
HEM	Hexane Extractable Material
SGT-HEM	Silica Gel Treated Hexane Material
HHRA	Human Health Risk Assessment
HNO_3	Nitric acid
HQ	Hazard Quotient
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICPMS	Inductively Coupled Plasma Mass Spectrometer
ICS	Interference Check Solution
ICV	Initial Calibration Verification
IEI	Inspection Experts, Inc.
IS	Internal standard
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LOD	Limit of Detection
LOQ	Limit of Quantitation
MAC	Maximum Allowable Concentrations
MB	Method Blank
MCL	Maximum Contaminant Levels
MDL	Method Detection Limit
mL	Milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Available

NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NELAC	National Environmental Laboratory Accreditation
NFCS	Niagara Frontier Consulting Services, Inc.
Nike Site	Former Niagara Falls-Buffalo Defense Nike Battery BU-34/35
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PE	Professional Engineer
PG	Professional Geologist
PMP	Project Management Professional
PCBs	Poly Chlorinated Biphenyls
PID	Photoionization detector
PIP	Public Involvement Plan
PM	Project Manager
PQL	Project Quantitation Limit
PQO	Project Quality Objectives
PSL	Project Screening Level
QA	Quality Assurance
QC	Quality Control
QSM	Quality Systems Manual
RAB	Restoration Advisory Board
RAGS	Risk Assessment Guidance for Superfund
RF	Relative Frequency
RI	Remedial Investigation
RPD	Relative Percent Difference
RSD	Relative Standard Distribution
RSL	Regional Screening Level
SCOs	Soil cleanup objectives
SLERA	Screening-level Ecological Risk Assessment
SOP	Standard Operation Procedures
SVOCs	Semi-volatile organic compounds
SW	Surface Water
TI2E	A joint venture between Tidewater, Inc. and Inspection Experts, Inc.
Tidewater	Tidewater, Inc.
TPH	Total Petroleum Hydrocarbons
TSRAWG	Tri-Services Environmental Risk Assessment Work Group
UCL	Upper confidence limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USACE	U.S. Army Corps of Engineers New England District
UST	Underground storage tanks
VOCs	Volatile organic compounds
WRAI	Waste Resource Associates, Inc.

EXECUTIVE SUMMARY

TI2E (an SBA-approved joint venture between Tidewater, Incorporated [Tidewater] and Inspection Experts, Incorporated [IEI]) prepared this Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) to support its Environmental Remediation Services (ERS) activities at the Former Niagara Falls-Buffalo Defense Nike Battery BU-34/35 (Nike Site) located in East Aurora and Orchard Park, New York. The field efforts will be performed under the U.S. Army Corps of Engineers New England District (USACE) Contract No. W912DR-13-D-0013.

Site location and Background

Site Description

The Niagara Falls-Buffalo Defense Nike Battery Unit BU-34/35 (BU 34/35) is located in Erie County, New York and consisted of two operational areas located on separate parcels of land. These include the battery control area (Control Area) (also called Integrated Fire Control, and the launch area (Launch Area) with underground missile magazines (also called silos in previous reports), launchers, and adjacent assembly, missile fueling, and service areas. The Launch Area, subject of this investigation, is a 19.84-acre parcel of land located at 601 Willardshire Road near the intersection of North Davis Road in the Town of Aurora, New York (Figure 1).

History and Operations

The Launch Area fee parcel was transferred from Marjorie Klopp to U.S. Army via deed dated December 9, 1955. The U.S. Army constructed the surface to air missile Launch Area and ancillary buildings between December 1955 and January 1957 (Malcolm Pirnie, Inc., 1996). The Launch Area formerly was occupied by barracks (subsequently converted to apartments) and a silo area consisting of six underground Nike missile magazines. The missile magazines were configured in two rows of three magazines each situated southeast of the former barracks. The underground structures were made of reinforced concrete and were accessed at the surface by steel doors.

The Site was deactivated on April 8, 1965 and Launch Area property fee parcels and easements reverted to the original property owner Marjorie K. C. Klopp. The Launch Area property and its improvements were transferred by the GSA to the Klopp estate, with no provisions for restoration of the land. Subsequently, the estate of Marjorie K. C. Klopp transferred title of the Launch Area Property to the H.G.M. Land Corporation. The Launch Area is currently owned by Waterhill Evergreen Holdings, LLC.

The GSA Report of Excess Real Property Schedule A listed several Launch Area buildings, and structures, utilities, and facilities that conveyed when the property deed reverted back to the property owner. The listed Launch Area buildings and structures that conveyed included the following:

- Launch Area Buildings included: missile assembly and test building, two acid storage shed, two barracks buildings one with administrative offices and the other mess hall,

generator building, gas meter house, chlorinator house, sewage pump house, sentry station and six underground missile magazines.

- Launch Area Structures included concrete pads, multi-court area (physical fitness), acid fueling station, electric distribution lines (above and underground), seven transformers, gas pipelines, sewage treatment plant, septic tank, sanitary sewer line, storm sewers, potable water lines, fencing, and vehicle parking areas.

Based on the above and USACE Guidance (2003), the Nike missiles, were assembled, serviced, maintained and prepared for firing at the Launch Area. Below TI2E summarize USACE guidance regarding the nature of the facilities and operations conducted at the assembly and services area; the fueling station, the above ground missile racks, and underground missile magazines.

The assembly and service area was divided into two sections: 1) the assembly area and 2) service areas located near the launch area. Nike missiles arrived on-site in major assembly components, unassembled, and unarmed. The assembly consisted of the installing missile fins and missile body sections and testing the missile system. Large overhead doors of the assembly building allowed the missiles to be rolled in and out of the test and assembly room.

The fueling of the Nike Ajax missile was performed at a fuel station. The missile was tilted by 3 to 5 degrees due to the ramp built in the acid fueling station. The acid platform hoist, consisting of crank-operated lift approximately 12 feet high, lifted the acid fuel drum onto the platform and allowed the fuel to flow by gravity into the missile. The acid fuel was stored in two acid storage sheds.

The missile launchers and steel structures were located at the surface at each of the six underground missile magazine. The monorail launcher, constructed of welded steel beams, consisted of the basic structure, erecting beam, loading and storage racks, four test stations, a launcher control indicator, and electrical and hydraulic power units. The monorail launcher was used in conjunction with the launching and handling rail as a firing platform for the missile and it served as a test station for missile pre-firing tests and operations. The erecting beam was raised or lowered by means of hydraulic pressure from the hydraulic power package. The beam was used to support the launching and handling rail and it contained one of the launcher's four test stations. Loading and storage rack sections extended from each side of the monorail launcher. The sections on the left side of the launcher were used as storage and test stations for the missiles. The sections on the right side of the launcher were used for storing the launching and handling rails after the missiles had been fired, and they were used to store rejected missiles until they are transported to the assembly and service area.

The underground missile magazines contained storage racks that held the missiles in the underground area using a system of locking pins. The missiles were rolled to the elevator and on to the elevator launcher. The Nike Ajax magazines housed up to twelve missiles. The missiles were lifted to the surface in a horizontal position by means of a hydraulic elevator and manually pushed along the launch rails to one of the launchers. The launcher elevated the missiles to an

angle of 88 degrees for launching. This angle prevented the booster from falling back on the launch area. During a launch, the crew took their positions in a small personnel room located behind three blast-proof doors.

The magazine pad had a double elevator door, which swung down to open. Stairways led to the double-door main entrances to the magazines. Access to Nike magazines was accomplished via armored hatches, vertical ladders and/or staircases, in some cases staircases. Emergency escape hatches, with counter-weights for easy opening, led from the underground personnel rooms to the outside. The magazines were made of reinforced concrete and fresh air was provided via a ventilation unit. Each magazine unit had several vent shafts.

Both natural gas and electric were commercially available to the Launch Area. This is evidenced by the list of conveyed structures including the gas meter house and gas pipelines and above ground and below ground electrical lines. The Launch Area also had its own electrical power as evidenced by the conveyance of the Generator House. The fuel and storage tanks associated with the generator house are unknown. In addition the GSA (1963) did not report that UST/ASTs used for the storage of fuel (if any) conveyed to the former property owner. The Launch Area potable water facilities included pump house, chlorinator, and potable water lines. Sanitary sewage treatment facilities included sewage treatment plant - sand filter and septic tank. The facility also had storm water facilities to direct storm water away from the underground missile magazines. These structures included bermed areas surrounding swales connected with culverts (USACE, 1957).

The Launch Area was only subject to DoD use and control between 1957 and 1964, and the only facility utilized post-DoD ownership and operation was the former enlisted men's barracks and bachelor officers' quarters building. These building structures were converted into apartments and then to a private residence, and then used for custodial purposes including vehicle and machinery storage for H.G.M. (Malcolm Pirnie, 1996).


20
16
Golf Center
Conley Rd
Michael Rd
Willardshire Rd
N Davis Rd
N Star Rd
Cazenovia Creek
187
Launch Area Site
Willardshire Rd
Quaker Rd
126
Cazenovia Creek
20A
Transit Rd
Grover Rd
N
0
2000
4000
Approx. Scale in Feet
NIKE
BU 34/35
Michigan
New York
Pennsylvania
Ohio
November 2015
TI2E, A Joint Venture
Between Tidewater and IEI
Prepared by:
K. DeSilva
Approved by:
K. Fields
Figure 1. Launch Area Location Map
Nike BU 34/35
East Aurora, NY

QAPP WORKSHEET #1 & 2: TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)

1. Project Identifying Information

a. Site name/project name: Former Niagara Falls-Buffalo Defense Nike Battery BU 34/35 East Aurora and Orchard Park, New York
b. Site location/number: FUDS No. C02NY007701
c. Contract Number: W912DR-13-D-0013
Contract Title: Remedial Investigation, Feasibility Study, Proposed Plan, and Decision Document, FUDS No. C02NY007701, Former Niagara Falls-Buffalo Defense Nike Battery BU 34/35, East Aurora and Orchard Park, New York

2. Lead Organization: U.S Army Corps of Engineers, New England District

Lead Organization's Project Manager (PM): _____
Signature
Mr. Greg Goepfert, USACE, New York District
March 1, 2016

Investigative Organization: TI2E

Investigative Organization's Project Manager: _____
Signature
Mr. Keith Fields, P.E., PMP; TI2E
March 1, 2016

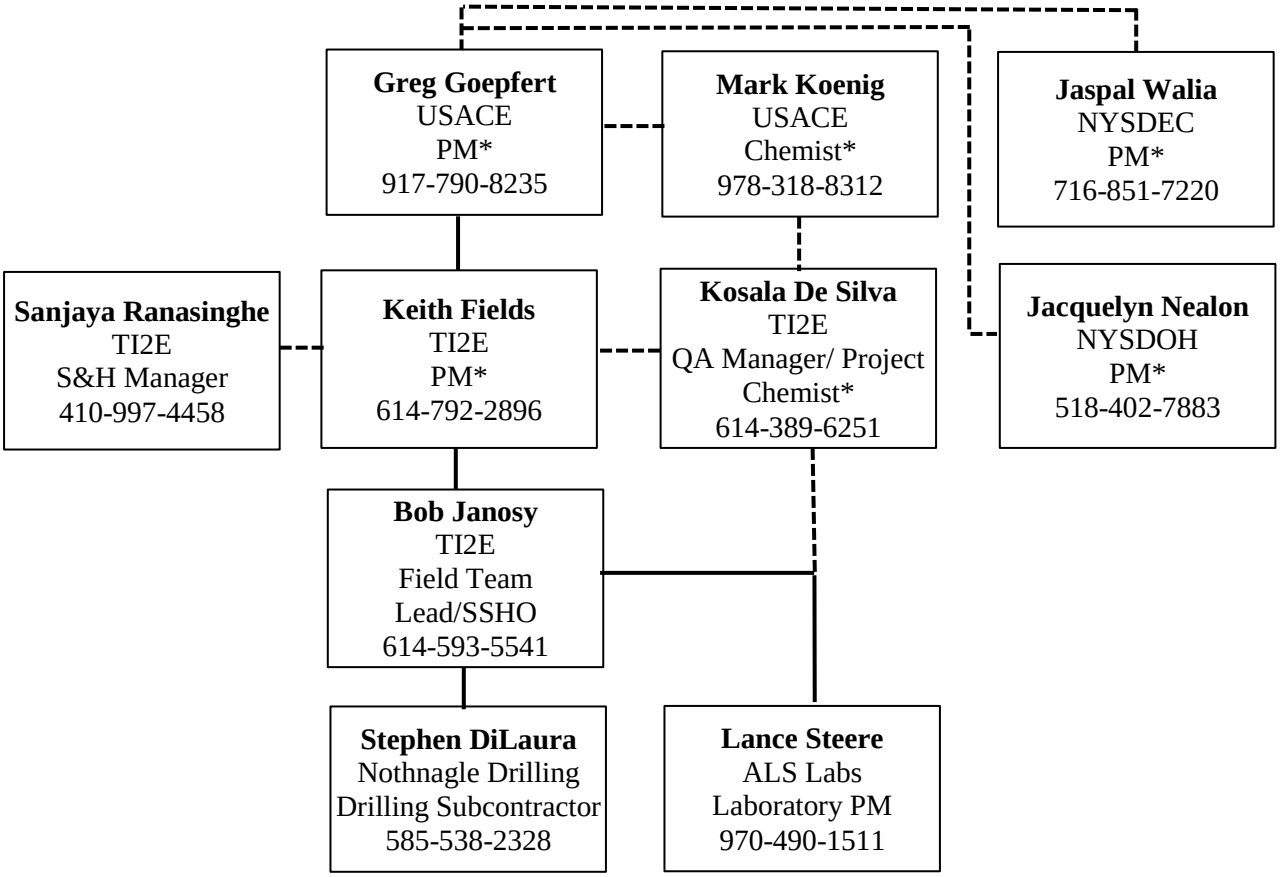
_____

3. List plans and reports from previous investigations relevant to this project

Title	Received Date
<u>Launch Area</u>	
Draft Limited Site Remedial Investigation Report (1996)	August 2015
Draft Contaminant Evaluation (1988)	

QAPP WORKSHEET #3 & 5: PROJECT ORGANIZATION AND QAPP DISTRIBUTION

(UFP-QAPP Manual Section 2.3 and 2.4)



QAPP WORKSHEET #4, 7, & 8: PROJECT PERSONNEL SIGN-OFF SHEET

(UFP-QAPP Manual Section 2.3.2)

Organization: TI2E					
Project Personnel	**Title**	**Education/Experience**	**Specialized Training/Certifications**	**Telephone Number**	**Signature/ Date**
Gary Verban,	Program Manager	BS/MS Chemical Engineering; 25 years of site characterization and remediation experience	Professional Engineer (PE), Project Management Professional (PMP)	703-288-1844	
Keith Fields	Project Manager	BS Civil Engineering; 20 years of site characterization and remedial action selection, design and implementation experience	PE, PMP	614-792-2896	
Sanjaya Ranasinghe	Safety & Health Manager	BS Biology, MS Forestry; >10 years of experience developing, implementing and monitoring H&S activities and projects	Certified Hazardous Materials Manager (CHMM), Certified Safety Professional (CSP)	410-997-4458	
Bob Janosy	Field Team Lead and Site Safety and Health Officer	BS/MS Geological Sciences; 20 years of site characterization and remediation experience	Professional Geologist (PG), HAZWOPER	614-593-5541	
Kosala De Silva	QA Manager/ Project Chemist	BS Chemistry/ MS Environmental Engineering; 5 years of site characterization and remedial action design and implementation experience	PE, CHMM	614-389-6251	
Ben Headington	Alternate Site Safety and Health Officer	10+ years of site characterization and remediation experience	PMP, HAZWOPER	614-348-8939	

Organization: ALS Laboratory (ALS), Fort Collins					
Lance Steere	Laboratory Project Manager	BS/MS Chemistry; 30 years of analytical lab management experience/ 28 years as Project Manager	Seminars in Project Management Certification in Department of Transportation (DOT) Shipping Regulations (HM126F, HM181)	970-490-1511	
Robert DiRienzo	Laboratory Quality Assurance (QA) Manager	BS Environmental Toxicology; 32 years of lab experience/ 16 years of QA experience and 15 years of NELAC support	NELAC Technical Auditor (L-A-B), ISO 17025 Lead and Technical Auditor(L-A-B and AIHA), Certified Quality Auditor (CQA) DoD QSM Auditor (L-A-B)	970-490-1511	
Organization: Laboratory Data Consultants					
Stella Cuenco	Data Reviewer, Operations Manager	B.S. Chemistry, University of the Philippines, 1991 Data Validation Experience: 18 years Laboratory Experience: 5 years	Specialized in hands-on CLP and SW-846 GC/MS methods.	760-827-1100	

NELAC - National Environmental Laboratory Accreditation

QAPP WORKSHEET #6 - COMMUNICATION PATHWAYS

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Point of Contact with USACE Project Manager (PM)	Lead Organization Project Manager	Greg Goepfert	917-790-8235	All materials and information about the project will be forwarded to the USACE PM by TI2E PM. The USACE PM is the main point of contact for TI2E for all aspects of the project.
Engineering /Design issues	Engineering Technical Lead/Design Team Lead	Penelope Reddy	978-318-8160	Responsible for coordinating with the USACE technical team and guiding TI2E in the development of all work plans, field work and reports required to complete the project.
Geological/Hydrogeological issues	USACE Geologist	Paul Young	978-318-8597	Geological or hydrogeological issues that arise will be discussed (through the USACE PM) with the USACE Geologist.
Ecological Risk Issues	USACE Ecologist	John (Mike) Penko	978-318-8139	Ecological Risk Assessment issues related to the RI will be discussed (through the USACE PM) with the USACE Ecologist.
Human Health Risk Issues	USACE Human Health Risk Assessor	Cynthia Colquitt	978-318-8042	Human Health Risk Assessment issues related to the RI will be discussed (through the USACE PM) with the USACE Human Health Risk Assessor.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Analytical data/sampling program issues	USACE Chemist	Mark Koenig	978-318-8312	Laboratory analytical issues or issues related to the sampling program will be discussed (through the USACE PM) with the USACE Chemist. Sampling/analytical issues that negatively affect the objectives of the project will be raised immediately.
Safety	USACE Safety Officer	Sheila Harvey	978-318-8504	Responsible for reviewing all Safety Plans, Accident Prevention Plans, Hazard Analyses, etc., to assure appropriate actions are taken to facilitate the safety of all project personnel during the course of this project.
Disposal of IDW/RW	USACE Regulatory Specialist	TBD		Waste streams generated for off-site disposal, other than trash and disposable personal protective equipment (PPE), will be discussed (through the USACE PM) with the USACE Environmental Scientist. Waste manifests and associated paperwork prepared by TI2E will be reviewed and signed by the USACE Environmental Scientist.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Manage all Project Phases	TI2E Project Manager	Keith Fields	614-792-2896	Overall management of the project. Maintain lines of communication between USACE, NYSDEC, NYSDOH, US Environmental Protection Agency (USEPA) Region 1, and subcontractors. Single point of contact for USACE PM. Notify Jeffery Skog of field-related problems by phone, email, or fax by COB the next business day.
QAPP Changes in the field	Field Team Lead	Bob Janosy	614-593-5541	Notify TI2E PM by phone (who is responsible to contact USACE PM) and email changes to QAPP made in the field and the reasons immediately.
Daily Field Progress Reports	Field Team Lead	Bob Janosy	614-593-5541	Email or fax daily field progress reports to the TI2E PM, who will review and distribute the progress reports to the USACE PM within 24 hours.
Reporting lab data quality issues	Laboratory Quality Assurance Officer	Robert DiRienzo	970-490-1511	All Quality Assurance (QA)/Quality Control (QC) issues with project field samples will be reported to the TI2E Project Chemist immediately. The issue and possible solutions will be identified and communicated by the TI2E Project Chemist to TI2E's Contractor Quality Control (CQC) Manager, who will contact the USACE Chemist within 24 hours.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (timing, pathways, etc.)
Field and Analytical Corrective Actions	Field Team Lead/Project Chemist	Bob Janosy	614-593-5541	The need for corrective action for field and analytical issues will be determined by the QA Manager. Any corrective actions identified will be communicated to the USACE PM and USACE Project Chemist within 24 hours.
Release of Analytical Data	QA Manager/ Project Chemist	Kosala De Silva	614-389-6251	No analytical data can be released until validation is completed and the Project Chemist has approved the release. Once the validation is complete and data approved, the TI2E Project Chemist will forward the information to the TI2E PM.
QAPP Amendments	Lead Organization Project Manager	Greg Goepfert	917-790-8235	Any major changes to the UFP-QAPP must be reviewed by U.S. Environmental Protection Agency (EPA) and NYSDEC, and approved by the USACE PM before the change can be implemented.
State of New York Regulatory Compliance issues	NYSDEC	Jaspal Walia	716-851-7220	Issues regarding compliance with state regulations will be raised by the TI2E PM to the USACE PM, who will contact NYSDEC for discussion.
State of New York Health and Safety Issues	NYSDOH	Jacquelyn Nealon	518-402-7883	Health and Safety issues regarding state regulations will be raised by the TI2E PM to the USACE PM, who will contact NYSDOH for discussion.

QAPP WORKSHEET #9 - PROJECT SCOPING SESSION PARTICIPANTS SHEET

(UFP-QAPP Manual Section 2.5.1)

Project Name: Environmental Remediation Services at the Former Niagara Falls-Buffalo Defense Nike Battery Bu-34-35 East Aurora and Orchard Park, New York **Projected Date(s) of Sampling:** April 2016 **Project Manager:** Keith Fields	**Site Name:** Former Niagara Falls-Buffalo Defense Nike Battery Bu-34-35 **Site Location:** East Aurora and Orchard Park, New York

Date of Session: 10/26/15

Scoping Session Purpose: Discuss direction of future field activities

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Greg Goepfert	Project Manager	USACE	917-790-8235	Gregory.J.Goepfert@usace.army.mil	PM
Cynthia Colquitt	Risk Assessor	USACE	978-318-8042	Cynthia.A.Colquitt@usace.army.mil	Risk Assessor
Mark Koenig	Chemist	USACE	978-318-8312	Mark.R.Koenig@usace.army.mil	Chemist
John Wyckoff	Geologist	TI2E	703-288-1844	John.wyckoff@tideh2o.net	Geologist
Julie Kramer	Env. Scientist	TI2E	614-707-4774	jkramer@ieinc.net	Dep. PM
Ben Headington	Project Manager	TI2E	614-792-2897	ben.headington@tideh2o.net	SSHO
Keith Fields	Project Manager	TI2E	614-792-2896	keith.fields@tideh2o.net	PM

Notes/Comments:
-Project activities will proceed based on CX comments to Work Plan and supporting documents
-Additional sampling is proposed to obtain representative samples rather than only define nature and extent of contamination
-Cynthia Colquitt is filling in for Penelope Reddy from USACE.
Action Items:
-A revised sampling approach will be prepared and submitted to USACE for review
-Responses to CX comments will be submitted within the week

QAPP WORKSHEET #10 – CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2)

Background Information: See Executive Summary

Sources of known Suspected Hazardous Waste: The Limited Remedial Investigation (1996) noted the presence of PCBs in soil and free product characterized as diesel range organics in Silo 4.

Primary Release Mechanism: Contaminants released from storage or containment structures into the soil and groundwater.

Secondary Contaminant Migration: Contaminants potentially migrating along underground utility pipelines.

Fate and Transport Considerations: The goal of the planned RI at the Nike sites is to adequately characterize the nature and extent of threat from hazardous substances, the fate and transport of the hazardous substances, and to gather data necessary to assess the extent to which the release poses a threat to human health, safety, or the environment. Data will be gathered to support the potential future Feasibility Study (FS) analysis and preliminary design of response actions, as warranted, by assessing the following factors [40 Code of Federal Regulations (CFR) 300.430(d)(2)]:

- Physical Characteristics of the property;
- Characteristics/classification surface soil, sediment, subsurface soil, surface water, and groundwater;
- General characteristics of potential sources/waste (e.g., quantities, concentration, toxicity, persistence, mobility, depth, nature, and extent, etc.);

Potential Receptors and Exposure Pathways: A Risk Assessment will be conducted as part of the planned RI at the Nike sites to assess:

- Actual and potential exposure pathways through environmental media;
- Actual and potential exposure routes (e.g., inhalation and ingestion); and
- Other factors such as sensitive populations that pertain to the characterization of the site or support the future analysis of potential remedial action alternatives.

Land Use Considerations:

The section describes the property parcel information, zoning information, and surrounding land-use. Information regarding the property parcels was obtained from the Erie County, New York Geographic Information System website (see, Erie County, "On Maps."). Zoning information was obtained from Town of Aurora, Erie County, New York Zoning District Map (CRA Infrastructure & Engineering, Inc., November 1996 and Revised, March 2010).

The Launch Area (fee parcels) is comprised of two parcels. The first 7.53-acre parcel (a rectangular shaped parcel) with frontage along Willardshire Road with address of 601 Willardshire Road, East Aurora, New York, is identified as Section-Block-Lot 160.00-3-36.2. The parcel type is identified as rural-residential. The second 12.4-acre parcel (an irregular shaped polygon) is identified as Section-Block-Lot as 160.00-3-36.1. This parcel formerly contained the six underground Nike missile storage magazines, acid fueling station, missile assembly building and generator building, and launchers; missile fuel service area; and four shallow two-inch diameter monitoring wells installed by M&E (1988). The current owner of both Launch Area parcels is identified as Waterhill Evergreen Holdings (Waterhill). The two parcels owned by Waterhill are surrounded on the east, south, and west by a 52.98-acre parcel identified as Section-Block-Lot 163.00-3-37 owned by Ambit Properties, LLC. This parcel type is identified as rural vacant and is situated between the two parcels that comprise the former Launch Area and Cazenovia Creek.

The Launch Area parcels are zoned Agricultural (CRA Infrastructure & Engineering, Inc., November 1996 and Revised, March 2010). The Launch Area is bounded: on the east by residential estates; on the south by forested land, an intermittent stream, and Cazenovia Creek on the west are residences, estate (large) properties; and the Elma Agricultural District; on the north by Willardshire Road and further to the north residential properties and estates. In addition to the above, the Christ the King Seminary, formerly the St. John Vianney Seminary is located 3,000 feet southeast of the Launch Area and the Craig Burn Country Club is located 1,600 feet to the north. The BU 34/35 Launch Area parcels have been designated part of the Knox Park Priority Property Grouping by the Town of Aurora Open Space Committee (Aurora, 2010). This property district consists of large-parcel properties located on Willardshire Road within close proximity of Knox Farm State Park. The Knox Park Priority Property Grouping was designated by the Aurora Open Space Committee for purposes of preserving large parcels as open space in the vicinity of the State Park (Aurora, 2010).

Currently the property is vacant (no residential inhabitants), posted, and monitored via all-terrain vehicle patrols (to prevent poachers) by the owner. The owner and its guests use the property for recreational hunting (Personal Communication, July 14, 2015, between Mr. David Novak, Waterhill Evergreen Holdings, Property Manager and Mr. Greg Goepfert, USACE).

Current interpretation of nature and extent of contamination to the extent that it will influence project-specific decision-making:

The current Conceptual Site Model (CSM) for Former Nike BU-34/35 is documented in Sections 1 and 2 of the RI Work Plan. Additional data collected as part of this RI will be used to update and refine the current CSM. Additional, data visualization methods (e.g., receptor flow chart and schematic diagrams) will be prepared as part of the RI Report.

QAPP WORKSHEET #11: PROJECT/DATA QUALITY OBJECTIVES

(UFP-QAPP Manual Section 2.6.1)

Step 1: State the Problem

USACE's overall goal for the Former Nike BU-34/35 site is to achieve site closure under the FUDS program. The purpose of this project is to conduct a Remedial Investigation to 1) determine the nature and extent of the contamination in soil and groundwater, 2) update/refine the current CSM, 3) collect data needed to evaluate the existing missile silo/pits to make future recommendations, and 4) perform a baseline human health and screening level ecological risk assessment. Following the RI, TI2E will prepare a Feasibility Study Report utilizing the data from the Remedial Investigation. Based on the Feasibility Study Report, TI2E will prepare a Proposed Plan and a Decision Document to document selection of the final remedy.

Step 2: Goals of the Study

The goal of the RI at the Former Nike BU-34/35 site is to characterize the nature and extent of threat from hazardous substances, the fate and transport of the hazardous substances, and to gather data necessary to assess the extent to which the release poses a threat to human health or the environment. Data will be gathered to support the potential future FS analysis and preliminary design of response actions, as warranted, by assessing the following factors:

- Physical Characteristics of the property;
- Characteristics/classification of surface soil, sediment, subsurface soil, surface water, and groundwater;
- General characteristics of potential sources/waste (e.g., quantities, concentration, toxicity, persistence, mobility, depth, nature, and extent, etc.);
- The extent to which the source(s) can be characterized;
- Actual and potential exposure pathways through environmental media;
- Actual and potential exposure routes (e.g., inhalation and ingestion); and
- Other factors such as sensitive populations that pertain to the characterization of the site or support the future analysis of potential remedial action alternatives.

Step 3: Information Inputs

Information inputs for the Former Nike BU-34/35 site include historical data gathered on the site (see Section 1.0, Section 2.0, and Appendix A of the RI Work Plan); analytical data collected from soil borings, test pits, groundwater monitoring wells, and silo water;

and observations made as part of the silo/pit evaluation. Tables 3-1 through 3-4 in the RI Work Plan describe the data gaps, data collection rationale, and data collection approach.

Step 4: Boundaries of the Study

The boundary of the RI is shown on Figure 3-3 in the RI Work Plan and includes the former Launch Area and adjacent background area. Target analytes are specified in Tables 4-1, and Table 4-2 in the RI Work Plan and in Table 11-1 below. The investigation area is comprised of one DU with the adjacent "background area." The DU includes the entire site investigation area, consistent with the likely future use of the site as a rural/residential property. The DU is divided into three sampling units (SUs): surface soil SU from 0 to 1 foot bgs; intermediate soil SU from 2 to 8 feet bgs comprised of the fill material at the contact between the fill and underlying native soil; and deep soil SU at 8 to 15 feet bgs comprised of soil at the contact between the glacial deposits and underlying shale bedrock. Data will be collected to assess: 1) the presence/absence of contamination; 2) representative conditions; and 3) background conditions. To address previously identified data gaps, the soil sampling design includes "biased" samples" collected from borings (12 samples from 4 borings) and test pits (9 samples from 3 test pits) to determine the presence or absence of contamination. To assess site "representative conditions," the sampling design includes collecting 42 soil samples from 14 randomly located borings. From each boring, soil will be collected from three sampling units described above. To assess background conditions, 20 background soil samples will be collected from 10 randomly located soil borings within the designated background area comprised of 5.8 acres located to the north of the access road/silo area and south of Willardshire Road. At each background soil boring location, one soil sample will be collected from 0 to 1 ft bgs and a second soil sample will be collected at approximately 8 ft bgs. In addition, near surface samples (defined as 0 to 2 inches bgs) will be collected from five soil boring locations and 5 background sampling locations.

Step 5: Analytical Approach

All soil samples in the investigation area will be analyzed for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), and metals (except surface and near surface soils will not be analyzed for VOCs). Soil samples in the background area will be analyzed for SVOC's and metals. In addition, surface soil samples collected from within drainage ditches will be analyzed for grain size and total organic carbon (TOC). All groundwater samples in the investigation area and silo water will be analyzed for VOCs, SVOCs, PCBs, anions, metals (total and dissolved), and TOC. Composite samples collected during pumping from Silo/Pit 1 will include additional analyses to evaluate IDW and remediation waste disposal options. Additional analyses include: total petroleum hydrocarbons (TPH) gasoline range (GRO) and diesel range (DRO); organochloride

pesticides; organophosphorous compounds; herbicides; sulfides; nitrogen; oil, grease, and total petroleum; hexavalent chromium; total cyanide; ignitability; and total coliform. The full list of proposed RI samples and analyses are listed in Table 11-1 below.

Table 11-1. Analytes, Number and Type of Samples, and EPA Methods for the RI at the Former Nike BU-34/35 Launch Area.

Analyte	No. and Type of Samples (incl. QA/QC)	EPA Method
28 soil borings (10 of these background), 5 new wells, 4 existing wells, 5 silo/pits (water), 1 composite silo (water for IDW disposal), 3 test pits (soil)		
Volatile Organic Compounds (VOCs)	18 RI soil borings (2 soil samples per boring); 9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW disposal; and 3 test pits (3 soil samples per test pit)	8260C
Semi-volatile Organic Compounds (SVOCs)	18 RI soil borings (3 soil samples per boring and an additional near surface sample collected from 5 of those borings); 10 background soil borings (2 samples per boring and an additional near surface sample collected from 5 of those background soil borings); 9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW disposal; and 3 test pits (3 soil samples per test pit)	8270D
Polychlorinated Biphenyls (PCBs)	18 RI soil borings (3 soil samples per boring and an additional near surface sample collected from 5 of those borings); 9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW disposal; and 3 test pits (3 soil samples per test pit)	8082

Analyte	No. and Type of Samples (incl. QA/QC)	EPA Method
Metals (filtered and unfiltered)	18 RI soil borings (3 soil samples per boring and an additional near surface sample collected from 5 of those borings); 10 background soil borings (2 samples per boring and an additional near surface sample collected from 5 of those background soil borings); 9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW; and 3 test pits (3 soil samples per test pit)	200.7/6010B/200.8/6020A
Anions (Chloride, Nitrate, Nitrite, Sulfate)	9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW disposal	300.0,
Total Organic Carbon (Aqueous)	9 groundwater monitoring wells (1 water sample per well); 5 silo/pits (2 depths of water from each silo); 1 composite silo water for IDW disposal	415.1/9060
Total Petroleum Hydrocarbons (TPH) (Diesel Range Organics [DRO] and Gasoline Range Organics [GRO])	1 composite silo water for IDW disposal	8015
Organochloride Pesticides	1 composite silo water for IDW disposal	SW 8081 A or B/EPA 608
Organophosphorus Compounds	1 composite silo water for IDW disposal	EPA 8141 A or B, and EPA 614
Herbicides	1 composite silo water for IDW disposal	SW8151A, EPA 615 and EPA 515.1

Analyte	No. and Type of Samples (incl. QA/QC)	EPA Method
Sulfides	1 composite silo water for IDW disposal	EPA 376.1 and SM4500 S2 F
Nitrogen as Nitrate and Nitrite	1 composite silo water for IDW disposal	EPA Method 353.2
Oil, Grease and Total Petroleum Hydrocarbons	1 composite silo water for IDW disposal	EPA 1664 A, AND SW9070A
Hexavalent Chromium	1 composite silo water for IDW disposal	SW3060A AND 7196A
Total Cyanide	1 composite silo water for IDW disposal	SW9010C, SW9013, EPA 335.1, EPA 335.2, CLP Inorganic SOW (ILM04.0); Determination Of Weak and Dissociable Cyanide – SM4500-CN I
Ignitability	1 composite silo water for IDW disposal	SW1010A and American Society for Testing and Materials (ASTM) 93-80
Total Coliforms	1 composite silo water for IDW disposal	SM9222
Field Duplicate Samples	10% of all samples/matrices collected	8260C; 8270D; 8015; 200.7/6010B/200.8/6020A; 8082; 300.0; 415.1/9060
Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Samples	5% of all samples/matrices collected	8260C; 8270D; 8015; 200.7/6010B/200.8/6020A; 8082; 300.0; 415.1/9060
Trip Blanks	1 per sample cooler w/VOCs	8260C

Analyte	No. and Type of Samples (incl. QA/QC)	EPA Method
Conditional Task: Install new boring(s)/ monitoring well(s) downgradient of new well and upgradient of Cazenovia Creek		
Volatile Organic Compounds (VOCs)	10 soil; 5 monitoring well groundwater	8260C
Semi-volatile Organic Compounds (SVOCs)	15 soil; 5 monitoring well groundwater	8270D
Polychlorinated Biphenyls (PCBs)	15 soil; 5 monitoring well groundwater	8082
Metals (filtered and unfiltered)	15 soil; 5 monitoring well groundwater	200.7/6010B/200.8/6020A
Field Duplicate Samples	10% of all samples/matrices collected	8260C; 8270D; 8015; 200.7/6010B/200.8/6020A; 8082; 300.0; 415.1/9060
Matrix Spike/Matrix Spike Duplicate Samples	5% of all samples/matrices collected	8260C; 8270D; 8015; 200.7/6010B/200.8/6020A; 8082; 300.0; 415.1/9060
Trip Blanks	1 per sample cooler w/VOCs	8260C

All RI characterization samples will be analyzed by ALS Environmental, TI2E's DoD Environmental Laboratory Approval Program (ELAP)-certified subcontract laboratory, in accordance with DoD Quality Systems Manual (QSM) Version 5.0.

Step 6: Performance Criteria

The RI sample design/investigation includes random boring/sample locations to collect representative samples to support baseline human health and screening level ecological risks assessment as well as targeted soil boring/test pit locations to satisfy identified data gaps. In addition, an adjacent area was identified for the purpose of collecting samples representative of ambient background conditions. The evaluation of risks to human or ecological receptors from exposure to site-related COPCs will be based on a computational approach in which hazard quotient (HQ) values (for human and ecological receptors) and lifetime excess cancer risks

(for humans) are calculated from available data. The probability of making either a false negative or a false positive decision error depends on the accuracy of all of the information used to make the calculations, including the concentration term, the exposure parameters, and the toxicity term. In general, the RA goal is to limit the risk of false negative decision errors by ensuring that all uncertain inputs into risk calculations are "conservative" (i.e., are more likely to overestimate than underestimate risk). For these reasons the sampling plan is focused on optimizing the number of samples that will be available for estimating average exposure levels in each exposure area for each environmental medium. The number of samples needed to limit uncertainty depends mainly on the nature of the underlying distribution and the degree of between-sample variability. The degree of uncertainty that can be accepted depends mainly on how close the data are to a decision criterion. That is, greater uncertainty is acceptable when the values are far removed (either below or above) the decision criterion than when the values are near the decision criterion. The maximum acceptable probability of making a false negative decision will be 5%, a value typically used by EPA.

Analytical data performance criteria/data quality indicators are specified in QAPP Worksheet #12-1 and #12-2. These data quality indicators include indicators (performance criteria) for precision, accuracy/bias, sensitivity, and completeness. With respect to data verification, validation, and usability: QAPP Worksheet #34 provides Data Verification and Validation Inputs; QAPP Worksheet #35 provides Data Verification Procedures; QAPP Worksheet #36 provides Data Validation Procedures; and QAPP Worksheet #37 provides Data Usability Assessment.

Step 7: Plan for Obtaining Data

Field documentation, equipment calibration and decontamination, and environmental sample handling and analysis will be performed in accordance with Standard Operation Procedures (SOPs) specified in Tables #18, #20 and #23 of this UFP-QAPP and are provided in Attachment B. All field activities will be documented in a field logbook and/or on field data forms (equipment calibration forms, soil-boring logs, Chain-of-Custody [C-O-C] forms, well gauging sheets, well purging and sampling sheets, etc.). TI2E will conduct all drilling and sampling activities in accordance with the above referenced SOPs.

All drilling and sampling equipment will be decontaminated between soil boring/well locations using a steam cleaner or pressure washer with non-phosphate detergent. Expendable materials will be used to the maximum extent possible to reduce decontamination requirements and to minimize the potential for cross contamination between boring/well sample locations.

TI2E will establish a data management and filing system including field sampling (e.g., soil boring, well construction and low-flow protocol stabilization parameter) logs, sample management and tracking procedures, and document control and inventory procedures for both laboratory data and field measurements to ensure the data collected are of adequate quality and quantity to support RI objectives for the Nike sites.

All sampling (soil borings, wells, surface water/sediment) will be used to determine the potential source(s), the general extent of contamination, and contaminant fate and transport mechanisms for the Former Nike Battery BU-34/35 site. All validated media sample results will be used to support a baseline HHRA and for the RI.

TI2E will conduct all sampling and analysis in accordance with ALS laboratory SOPs specified in QAPP Worksheet #23 (Analytical SOP References Table) and the USACE "Chemistry Instructions for Scope of Services for Contracted Environmental Studies" (latest version).

Environmental sampling and analysis field work is described below:

Field activities at the Former Niagara Falls – Buffalo Defense Nike Battery BU-34/35 will encompass the Launch Area only and will include the following:

1. **Mobilization and utility clearance** - TI2E will coordinate all required utility clearances with a qualified contractor and/or other responsible entities prior to the commencement of any ground-intrusive activities at the site. The locations of the proposed borings/monitoring wells and test pit excavations will be staked prior to mobilizing equipment to the site, and silo/pit doors will be located if they are not visibly identifiable through field observation.

2. **Evaluation of existing structures and installations** - Upon mobilization to the site, existing installations will be located and evaluated to determine their condition and whether they can be used for investigation purposes. These installations include monitoring wells, the sand filter, the pump house assembly, and the silo/pits. Once located, the silo/pits will need to be exposed with a backhoe and sampling ports installed.

3. **Soil boring and monitoring well installation and sampling** - Soil borings will be advanced for the purpose of collecting soil samples, obtaining lithologic data, and installing monitoring wells for groundwater sampling. A license professional geologist

will oversee drilling activities and classify and log cuttings retrieved from the drilling process according to the USCS. During drilling and sampling activities, a PID will be used to screen soil samples/cuttings for residual VOCs. Soil samples for laboratory analysis will be collected from the depths indicated in Table 11-1.

Once the borings are completed, groundwater monitoring wells will be installed to a depth of approximately 15 ft bgs and screened across the water table (approximately 5 to 15 ft bgs). If possible, soil borings will be advanced to 18 feet bgs (unless refusal is met due to bedrock). Groundwater monitoring wells will be installed as deep as possible within the boring. Monitoring wells will be constructed of 2-inch diameter Schedule 40 PVC pipe. Final construction details will depend on site conditions at the time of installation. The wells will be finished at the surface with a stickup well cover.

Groundwater samples will be collected from the 5 new monitoring wells along with 4 existing monitoring wells provided they are in satisfactory condition. Additional monitoring wells may be installed if it becomes necessary to delineate the downgradient extent of any potential contamination. Laboratory analysis will take place according to the methods listed in the table above. All drilling, lithologic logging, and sampling activities will be conducted in accordance with the USACE *"Geology Supplement to the Scope of Services"* (May 2011).

4. **Test pit excavations** - Test pits will be installed near the Generator Building/Missile Assembly Building, near the Acid Mixing and Wash Rack, and at Silo/Pit 6 to evaluate shallow subsurface soils in areas of potential chemical release. The test pits will be approximately 10 ft wide x 10 ft long and will be installed with a backhoe to a depth of approximately 5 ft. Dimensions may vary slightly depending on site specific conditions.

   Prior to initiating excavation activities, the bucket of the backhoe will be decontaminated. Excavated soils will be placed on plastic sheets adjacent to the excavation and will be examined for any visual staining or any other unusual features. A lithologic description will be noted for discrete depths.

   Field samples will be collected from the excavated soil that are representative of the shallow soils (0-1 feet bgs range), mid-excavation (approximately 3 feet bgs), and base of the pit (approximately 5 feet bgs). The soil will be placed in a jar, sealed and then after 15 minutes, the head space will be screened for organic vapors using a PID. Additional field samples for VOC analysis will be collected from any locations where odor or visual staining is observed.

Three discrete soil samples will be collected from each test pit for laboratory analysis that are representative of the shallow soils (1 feet bgs range), mid-excavation (approximately 3 feet bgs), and base of the pit (approximately 5 feet bgs). Samples will be collected from the bucket of the backhoe and placed in laboratory supplied, pre-cleaned sample jars, labeled with a unique sample identification, chilled, and shipped under chain-of-custody to an ELAP-certified laboratory. Samples will be analyzed according to the laboratory methodology listed in the table above. Upon completion of sampling activities, soil will be returned to the respective test pit excavations and, to the extent possible, the area restored to a similar condition that existed before the work commenced.

5. **Silo/pit evaluation** - The silo/pit evaluation will serve to assess the physical structures, contents, current water chemistry, and hydraulic connectivity of silo/pits. Currently, all silos/pits are covered with top soil/vegetation and cannot be accessed. Access into the silos/pits will require earthwork and footprint delineation which will include:
   - Excavation of 1-2 ft around the perimeter of Silo/Pit 1;
   - Removal of top soil, vegetation, and concrete from the surface of Silo/Pit 1 to gain access through the hatch;
   - Documentation of definable silo/pit features and measurements;
   - Removal of top soil/concrete from the access hatches at Silos/Pits 2 through 5 and establish 4-in access ports at each; and
   - Covering the access ports when field work is complete.

   A backhoe will be mobilized to the site to remove top soil, excavate the footprint of Silo/Pit 1, and remove concrete obstructions blocking access to the hatches. If practical, previously used monitoring ports will be identified and utilized. If not, access will be established by core drilling an access port measuring at least 4 inches in diameter. Access ports/openings will be secured in a manner that limits unauthorized access but allows access for further evaluation if necessary.

   Each silo/pit will be measured for current water levels and free product thickness (if present) using a Solinst 122 Interface Meter, or similar meter. Depth to bottom of the silo will also be recorded. In addition to field monitoring, water samples for laboratory analysis will be collected from the surface and the bottom of each silo/pit and analyzed according to the methods listed in the table above.

   Determining if the silos/pits are hydraulically connected is an important factor to develop future recommendations. In order to do this, the following activities will be performed:

   - Mobilize 100,000 gallons of water storage and required pump;
   - Install a data logging transducer into Silo/Pit 1 to monitor drawdown and recharge rates;

- Pump 100,000 gallons of water from Silo/it 1 (or alternative silo/pit if access and/or free product levels prohibit access or pumping);
- Monitor water levels (using a pressure transducer) in all silos/pits during pumping to determine drawdown;
- Sample purged water from storage tanks; and
- Return purged water back into Silo/Pit 1.

Extraction of the water will require the mobilization of five 21,000 gallon steel tanks, pump, and associated piping. Prior to pumping, a data logging level transducer will be placed into Silo/Pit 1 to track drawdown and potential recharge rates into the silo/pit over a 24-hour period. Pumping from Silo/Pit 1 will average approximately 750-1,500 gallons per minute (gpm) and last approximately 1-2 hours. During pumping, water levels will be collected from the silos/pits every 10 minutes to accurately determine if pumping activities at Silo/Pit 1 hydraulically impact the other silos/pits.

A composite silo water sample will be collected during pumping prior to the water entering the tanks. This sample will be collected at 4 different intervals during pumping for the purposes of collecting a representative silo/pit water sample. The water sample and analytical results will be compared to New York Class GA Groundwater Effluent Limitations and the Erie County Sanitation District Guidelines for the Discharge of Petroleum Contaminated Water and Groundwater Remediation (2006). The guidelines specify that Groundwater from remediation sites must meet District limits for discharge, have no other contaminates of concern, and be treated prior to discharge.

The silo/pits and storage tanks will be continuously monitored, visually, for free product. If measurable free product is identified within the storage tanks, it will be skimmed off with an absorbent boom and disposed of as IDW waste before the purge water is returned to Silo/Pit 1. Once empty, the temporary storage tanks will be decontaminated and removed from the site.

Following completion of the silo/pit evaluation, an accurate footprint of Silo/Pit 1 will be defined and access points to all silo/pits (except Silo/Pit 6) will be clearly identified and made available for future activities. In addition, an understanding of the current water levels, product thickness (if any), and chemical composition of the silo/pit water will be established. Hydraulic testing of the pits will determine if the silos/pits are hydraulically connected and provide an understanding of groundwater infiltration rates into the silos/pits. This site-specific information will be used in conjunction with document findings and experiences from other Nike missile sites to support a feasibility study and analysis of alternatives for future recommendations.

6. **Surveying the site** - The location of all relevant site features will be surveyed to prepare a site map. These include Silo/Pit 1 footprint and definable features, access points for Silo/Pits 1 through 5, utilities and infrastructure identified by the geophysical survey, existing wells, new borings/wells, test pits, as well as any surface water/sediment sampling locations, if any. The elevation of wells and water levels will also be accurately measured for determination of groundwater gradient and predicted flow direction.

Details on the HHRA and SLERA are provided in Section 5.0 of the RI Work Plan.

QAPP WORKSHEET #12-1 - MEASUREMENT PERFORMANCE CRITERIA TABLE – AQUEOUS

(UFP-QAPP Manual Section 2.6.2)

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	VOCs (8260C)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	Relative Percent Difference (RPD) ≤ 30% for target compounds detected in parent sample and field duplicate ≥ Limit of Quantitation (LOQ).
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Laboratory Control Sample (LCS) recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Overall Accuracy/Bias (contamination)	Equipment Blanks	< ½ LOQ for all target compounds, except common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride), which are allowable to the LOQ; or as otherwise stipulated in the applicable Laboratory

		Information Management System (LIMS) program
Sensitivity	Reporting limit Verification Sample [RVS] (spiked at LOQ).	Value should be greater than ½ LOQ
Completeness	Number of valid samples	95 percent

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	SVOCs (8270D)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for target compounds detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	LCS recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Overall Accuracy/Bias (contamination)	Equipment Blanks	< ½ LOQ for all target compounds, or as otherwise specified in applicable LIMS program specification.
Sensitivity	Reporting limit Verification Sample [RVS] (spiked at LOQ).	Value should be greater than ½ LOQ
Completeness	Number of valid samples	95 percent

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	PCBs (8082)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for target compounds detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	LCS recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Overall Accuracy/Bias (contamination)	Equipment Blanks	< ½ LOQ for all target compounds, or as otherwise specified in applicable LIMS program specification.
Sensitivity	ALS analyzes a single point for Aroclors 1221, 1232, 1242, 1254, 1262 and 1268 at the reporting limit to show instrument sensitivity for all the targets.	LOQ
Completeness	Number of valid samples	95 percent

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	Metals (200.7/6010B/200.8/6020A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 20%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Recovery limit 85-115% for each analyte.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Recovery limit 70-130% for each analyte, not calculated if analyte conc >4X the spike level. For each analyte, RPD ≤20%
Overall Accuracy/Bias (contamination)	Equipment Blanks	< LOQ/2 for all target compounds, or as otherwise specified in applicable LIMS program specification.
Sensitivity	CRI - Low concentration test solution containing analyte concentrations at the reporting limit	Analyzing the CRI solution at the beginning and end of each sequence provides assurance that the instrument sensitivity is adequate to support the reporting limit
Completeness	Number of valid samples	95 percent

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	Mercury (245.1/7470A /7471A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 20%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Recovery must be within ±15% of expected value (EPA 245.1). For SW7470A recovery for aqueous LCS must agree within +20% of expected value. For SW7471A, the recovery for the solid matrix LCS must agree within +20% of expected value
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Overall Accuracy/Bias (contamination)	Equipment Blanks	<LOQ/2: Should not contain any target compounds at or above the LOQ or per other criteria as specified in the applicable LIMS program specification

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	TPH-GRO (8015), TPH-DRO (8015), Pesticides (402/SW8081A or B), Organophosphorus Compounds (407/EPA 8141 A OR B), Herbicides (434/SW8151A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Matrix Spike Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	LCS recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	For SW7470A/7471A, recovery should agree within ±20% of expected value. For EPA 245.1, recovery must agree within ±30% of expected value.
Overall Accuracy/Bias (contamination)	Equipment Blanks	< ½ LOQ for all target compounds, or as otherwise specified in applicable LIMS program specification.

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	Anions (300.0)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Results obtained must agree within ±10% of expected (known) analyte concentration for aqueous samples; within ±15% of known analyte concentration for solid sample extracts
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	Recoveries should meet client criteria for the spiked compounds. RPD for the MSD should meet advisory limit of <20%
Overall Accuracy/Bias (contamination)	Method Blanks	Anion content of MB must not exceed analyte LOQ. Exception: Samples with analyte concentrations >10X amount found in blank may be reported and narrated.

Matrix	Groundwater; Surface Water; IDW Water	
Analytical Group	Total Organic Carbon (415.1/9060)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	For Method 415.1 and SW9060A analyses, the LCS result must be within ±15% of the expected concentration
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For Method 415.1 and SW9060A analyses, MS/MSD recoveries should meet advisory limits of ±20% (80-120% of the expected values) and RPD should be ≤ 20
Overall Accuracy/Bias (contamination)	Method Blanks	For Method 415.1 and SW9060A analyses, the MB result must not exceed LOQ (usually 1mg/L TOC)
Completeness	Number of valid samples	95 percent

Matrix	Surface Water	
Analytical Group	Sulfides (1120/EPA 376.1 and SM4500 S^2 F)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Laboratory Duplicate	RPD must be ≤ 20%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Concentration results obtained must agree between 80% and 120% of expected value
Overall Accuracy/Bias (contamination)	Method Blanks	Sulfide content of any blank must not exceed the analyte reporting limit (typically 2mg/L S^2)

Matrix	Surface Water	
Analytical Group	Nitrogen as Nitrates and Nitrites (1127/EPA 353.2)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Matrix Spike Duplicates	RPD advisory limit is <20; client-specified criteria may apply
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Results must agree within ±10% of expected values for aqueous sample analyses; within ±15% for solid matrix extract analyses
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	Recoveries should meet advisory limits of ±25% of the expected values; client-specified criteria may apply
Overall Accuracy/Bias (contamination)	Method Blanks	NO_2 -N / NO_3-N content of the blank must be <LOQ

Matrix	Surface Water	
Analytical Group	Oil, Grease and Total Petroleum Hydrocarbons (671/EPA 1664 A, and SW9070A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Matrix Spike Duplicates	Hexane Extractable Material (HEM) RPD should be <18%; Silica Gel Treated Hexane Material (SGT-HEM) RPD <34%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Results obtained must be within 79-114% of expected (known) concentration of HEM, and 64-132 % for SGT-HEM
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	Results obtained should be within 79-114% of expected concentration of HEM, and 64-132 % SGT-HEM
Overall Accuracy/Bias (contamination)	Method Blanks	MB must not yield HEM content above the 5.0 mg/L LOQ

Matrix	Surface Water	
Analytical Group	Hexavalent Chromium (1121/SW3060A and 7196A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Matrix Spike Duplicates	RPD should be ≤ 20.
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Recoveries must be within ±20% of expected values
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	Recoveries should be within ±25 % of expected values
Overall Accuracy/Bias (contamination)	Method Blanks	Cr^{+6} content of the blank must be < LOQ; LOQ usually 2.0 mg/Kg Cr^{+6}

Matrix	Surface Water	
Analytical Group	Total Cyanide (434/SW9010C, SW9013, EPA 335.1, EPA 335.2, CLP Inorganic SOW (ILM04.0); Determination of Weak and Dissociable Cyanide – SM4500-CN)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Matrix Spike Duplicates	RPD between duplicates should be <20
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Distilled LCS result must agree within ±15% of non-distilled ICV result
Analytical Accuracy/Bias (matrix interference)	Matrix Spike	Recoveries should meet control limits of 75-125 %
Overall Accuracy/Bias (contamination)	Method Blanks	Cyanide content of the blank must be less than the analyte LOQ; LOQ usually 0.01mg/L CN; 0.50mg/Kg Cyanide

Matrix	Surface Water	
Analytical Group	Total Coliforms)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicate	Presence/ absence same as original sample
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Presence for positive controls/absence for negative controls
Laboratory Precision	Laboratory Duplicate	No presence of total coliform or E. coli
Overall Accuracy/Bias (contamination)	Laboratory Blanks	No presence of total coliform or E. coli

QAPP WORKSHEET #12-2 - MEASUREMENT PERFORMANCE CRITERIA TABLE – SOIL

(UFP-QAPP Manual Section 2.6.2)

Matrix	Soil	
Analytical Group	VOCs (8260C)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	Relative Percent Difference (RPD) ≤ 30% for target compounds detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Laboratory Control Sample (LCS) recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Overall Accuracy/Bias (contamination)	Equipment Blanks	< ½ LOQ for all target compounds, except common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride), which are allowable to the LOQ; or as otherwise stipulated in the applicable LIMS program

Sensitivity	Reporting limit Verification Sample [RVS] (spiked at LOQ).	Value should be greater than ½ LOQ
Completeness	Number of valid samples	95 percent

Matrix	Soil	
Analytical Group	SVOCs (8270D)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	Relative Percent Difference (RPD) ≤ 30% for target compounds detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Laboratory Control Sample (LCS) recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Sensitivity	Reporting limit Verification Sample [RVS] (spiked at LOQ).	Value should be greater than ½ LOQ
Completeness	Number of valid samples	95 percent

Matrix	Soil	
Analytical Group	PCBs (8082)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for target compounds detected in parent sample and field duplicate ≥ LOQ
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 25%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	LCS recoveries must meet (DoD) acceptance criteria for all target compounds (QSM v5.0). In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.
Sensitivity	ALS analyzes a single point for Aroclors 1221, 1232, 1242, 1254, 1262 and 1268 at the reporting limit to show instrument sensitivity for all the targets.	LOQ
Completeness	Number of valid samples	95 percent

Matrix	Soil	
Analytical Group	Metals (200.7/6010B/200.8/6020A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 20%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Recovery limit 85-115% for each analyte.
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	Recovery limit 70-130% for each analyte, not calculated if analyte conc. >4X the spike level. For each analyte, RPD ≤20%
Sensitivity	CRI - Low concentration test solution containing analyte concentrations near the reporting limit	Analyzing the CRI solution at the beginning and end of each sequence provides assurance that the instrument sensitivity is adequate to support the reporting limit
Completeness	Number of valid samples	95 percent

Matrix	Soil	
Analytical Group	Mercury (245.1/7470A /7471A)	
Concentration Level	Low	
Data Quality Indicator	**QC Sample and / or Measurement Performance Activity**	**Measurement Performance Criteria**
Overall Precision	Field Duplicates	RPD ≤ 30% for analytes detected in parent sample and field duplicate ≥ LOQ.
Analytical Precision (Lab)	Laboratory Control Sample Duplicated	RPD ≤ 20%
Analytical Accuracy/Bias (Lab)	Laboratory Control Samples	Recovery must be within ±15% of expected value (EPA 245.1). For SW7470A recovery for aqueous LCS must agree within +20% of expected value. For SW7471A, the recovery for the solid matrix LCS must agree within +20% of expected value
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates	For matrix evaluation, use LCS acceptance criteria specified by DoD (QSM v5.0), if available. In-house limits will be used; however, for analytes falling outside the DOD QSM limits, laboratory LCS's (30-50) will be requested to determine that less than 5% fall outside the QSM limits.

QAPP WORKSHEET #13 - SECONDARY DATA CRITERIA AND LIMITATIONS

(UFP-QAPP Manual Section 2.7)

Secondary Data	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use
Meteorological	National Weather Service	Estimations of seasonal fluctuations in storm water runoff	Published data are available for past 20 years. No known limitations.
Topographic	U.S. Geological Survey	Surface water drainage pathways	
Launch Area Geology and Hydrogeology	Limited RI/FS Report, Malcolm Pirnie/URS, 1996	Groundwater flow and soil permeability data	Data in RI/FS report used data accumulated by Metcalf & Eddy (M&E) in 1988. Geologic conditions may have fluctuated since.
Launch Area Contaminant Data	Limited RI/FS Report, Malcolm Pirnie/URS, 1996	Contamination assessment of silo water, groundwater and surface soils	Natural attenuation will affect contaminant concentrations.

QAPP WORKSHEET #14/16 - PROJECT TASKS AND SCHEDULE

(UFP-QAPP Manual Section 2.8.1)

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
RI Work Plan	TI2E	07/15/2015	2/26/2016	Final RI Work Plan	2/26/2016
RI Field Investigation	TI2E	04/04/2016	05/13/2016	Final Investigation Work Plan Field Report	8/14/2016
- Perform Utility Locations	SoftDig	04/06/2016	04/08/2016		
- Install new borings/wells	Nothnagle Drilling	04/11/2016	04/29/2016		
- Sample Collection	TI2E Field Team Lead	05/02/2016	05/13/2016		
- Analysis	ALS Labs	05/16/2016	06/10/2016		
- Data Validation	LDC	06/10/2016	07/01/2016		
RI Project Team Meeting	TI2E	08/21/2016	08/21/2016	Final RI Project Team Meeting Minutes	09/12/2016
RI Report	TI2E	09/13/2016	01/29/2017	Final RI Report	05/05/2017

QAPP WORKSHEET #15-1 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - VOC (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater; Surface Water
Analytical Group: VOCs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Acetone	67-64-1	14,000	RSL	10	3	10
Benzene	71-43-2	1.6	RSL	1	0.3	1
		5.0	MCL			
Bromobenzene	108-86-1	62	RSL	1	0.3	1
Bromochloromethane	74-97-5	83	RSL	1	0.3	1
Bromodichloromethane	75-27-4	0.13[3]	RSL	1	0.3	1
		80[4]	MCL			
Bromoform	75-25-2	3.3	RSL	1	0.3	1
		80[4]	MCL			
Bromomethane	74-83-9	7.5	RSL	1	0.3	1
2-Butanone (Methyl ethyl ketone - MEK)	78-93-3	5,600	RSL	10	3	10
n-Butylbenzene	104-51-8	1,000	RSL	1	0.3	1
sec-Butylbenzene	135-98-8	2,000	RSL	1	0.3	1
tert-Butylbenzene	98-06-6	690	RSL	1	0.3	1
Carbon disulfide	75-15-0	810	RSL	1	0.3	1

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Carbon tetrachloride	56-23-5	0.45[3]	RSL	1	0.3	1
		5.0	MCL			
Chlorobenzene	108-90-7	78	RSL	1	0.3	1
		100	MCL			
Chlorodibromomethane (Dibromochloromethane)	124-48-1	0.17[3]	RSL	1	0.3	1
		80[4]	MCL			
Chloroethane	75-00-3	21,000	RSL	1	0.3	1
Chloroform	67-66-3	0.22[3]	RSL	1	0.3	1
		80[4]	MCL			
1-Chlorohexane	544-10-5	No RSL / MCL		1	0.3	1
Chloromethane	74-87-3	190	RSL	1	0.3	1
2-Chlorotoluene	95-49-8	240	RSL	1	0.3	1
4-Chlorotoluene	106-43-4	250	RSL	1	0.3	1
1,2-Dibromo-3-chloropropane	96-12-8	0.0003[5]	RSL	2	0.3	2
		0.2[5]	MCL			
		2	LOQ			
1,2-Dibromoethane	106-93-4	0.008[5]	RSL	1	0.3	1
		0.05[5]	MCL			
		1	LOQ			
Dibromomethane	74-95-3	8	RSL	1	0.3	1
1,2-Dichlorobenzene	95-50-1	300	RSL	1	0.3	1
		600	MCL			

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
1,3-Dichlorobenzene	541-73-1	No RSL / MCL		1	0.3	1
1,4-Dichlorobenzene	106-46-7	0.48[3]	RSL	1	0.3	1
		75	MCL			
Dichlorodifluoromethane	75-71-8	200	RSL	1	0.3	1
1,1-Dichloroethane	75-34-3	2.7	RSL	1	0.3	1
1,2-Dichloroethane	107-06-2	0.17[3]	RSL	1	0.3	1
		5.0	MCL			
cis-1,2-Dichloroethene	156-59-2	36	RSL	1	0.3	1
		70	MCL			
trans-1,2-Dichloroethene	156-60-5	360	RSL	1	0.3	1
		100	MCL			
1,1-Dichloroethene	75-35-4	280	RSL	1	0.3	1
		7.0	MCL			
1,2-Dichloropropane	78-87-5	0.44[3]	RSL	1	0.3	1
		5.0	MCL			
1,3-Dichloropropane	142-28-9	370	RSL	1	0.3	1
2,2-Dichloropropane	594-20-7	No RSL / MCL		1	0.3	1
cis-1,3-Dichloropropene	10061-01-5	No RSL / MCL		1	0.3	1
trans-1,3-Dichloropropene	10061-02-6	No RSL / MCL		1	0.3	1
1,1-Dichloropropene	563-58-6	No RSL / MCL		1	0.3	1

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Ethylbenzene	100-41-4	1.5	RSL	1	0.3	1
		700	MCL			
Hexachlorobutadiene	87-68-3	0.14[5]	RSL	1	0.3	1
		1	LOQ			
2-Hexanone (Methyl butyl ketone)	591-78-6	38	RSL	10	3	10
Iodomethane	74-88-4	No RSL / MCL		1	0.3	1
Isopropylbenzene (Cumene)	98-82-8	450	RSL	1	0.3	1
p-Isopropyltoluene	99-87-6	No RSL / MCL		1	0.3	1
Methylene chloride (Dichloromethane or DCM)	75-09-2	11.4	RSL	1	0.44	1
		5.0	MCL			
4-Methyl-2-pentanone (MIBK)	108-10-1	1,200	RSL	10	3	10
Methyl tert-butyl ether (MTBE)	1634-04-4	14	RSL	1	0.3	1
Naphthalene	91-20-3	0.17[3]	RSL	1	0.3	1
		1	LOQ			
n-Propylbenzene	103-65-1	660	RSL	1	0.3	1
Styrene	100-42-5	1,200	RSL	1	0.3	1
		100	MCL			
1,1,1,2-Tetrachloroethane	630-20-6	0.57[5]	RSL	1	0.3	1
		1	LOQ			

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
1,1,2,2-Tetrachloroethane	79-34-5	0.076[5]	RSL	1	0.3	1
		1	LOQ			
Tetrachloroethene	127-18-4	11	RSL	1	0.2	1
		5.0	MCL			
Toluene	108-88-3	1,100	RSL	1	0.3	1
		1,000	MCL			
1,2,3-Trichlorobenzene	87-61-6	7.0	RSL	1	0.3	1
1,2,4-Trichlorobenzene	120-82-1	1.1	RSL	1	0.3	1
		70	MCL			
1,1,1-Trichloroethane	71-55-6	8,000	RSL	1	0.3	1
		200	MCL			
1,1,2-Trichloroethane	79-00-5	0.28[3]	RSL	1	0.3	1
		5.0	MCL			
Trichloroethene	79-01-6	0.49[3]	RSL	1	0.3	1
		5.0	MCL			
Trichlorofluoromethane	75-69-4	1,100	RSL	1	0.3	1
1,2,3-Trichloropropane	96-18-4	0.00075[5]	RSL	1	0.3	1
		1	LOQ			
1,2,4-Trimethylbenzene	95-63-6	15	RSL	1	0.3	1
1,3,5-Trimethylbenzene	108-67-8	120	RSL	1	0.3	1
Vinyl acetate	108-05-4	410	RSL	2	0.52	2
Vinyl chloride	75-01-4	0.019[3]	RSL	1	0.3	1

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
		2.0	MCL			
m-Xylene & p-Xylene	179601-23-1	190	RSL	1	0.3	1
o-Xylene	95-47-6	190	RSL	1	0.3	1

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include both EPA Regional Screening Levels (June 2015) and Maximum Contaminant Levels (MCLs) (May 2009); tables for both are included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the MCL.
[4]MCL of 80 µg/L applies to total trihalomethanes (chloroform, bromodichloromethane, dibromochloromethane, and bromoform).
[5]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Tap Water Level.

QAPP WORKSHEET #15-2 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - SVOC (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater; Surface Water
Analytical Group: SVOCs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Acenaphthene	83-32-9	530	RSL	10	3.0	10
Acenaphthylene	208-96-8	No RSL / MCL		10	3.0	10
Aniline	62-53-3	13	RSL	10	3.0	10
Anthracene	120-12-7	1,800	RSL	10	3.0	10
Azobenzene	103-33-3	0.12[4]	RSL	10	3.0	10
		10	LOQ			
Benzo(a)anthracene	56-55-3	0.012[4]	RSL	10	3.0	10
		10	LOQ			
Benzo(b)fluoranthene	205-99-2	0.034[4]	RSL	10	3.0	10
		10	LOQ			
Benzo(k)fluoranthene	207-08-9	0.34[4]	RSL	10	3.0	10
		10	LOQ			
Benzoic acid	65-85-0	75,000	RSL	50	20	50.0
Benzo(g,h,i)perylene	191-24-2	No RSL / MCL		10	3.0	10
Benzo(a)pyrene	50-32-8	0.0034[4]	RSL	10	3.0	10
		0.20[4]	MCL			

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
		10	LOQ			
Benzyl alcohol	100-51-6	2,000	RSL	10	3.0	10
bis(2-Chloroethoxy) methane	111-91-1	59	RSL	10	3.0	10
bis(2-Chloroethyl) ether	111-44-4	0.014[4]	RSL	10	3.0	10
		10	LOQ			
Bis (2-chloroisopropyl) ether	108-60-1	0.36[4]	RSL	10	3.0	10
		10	LOQ			
bis(2-Ethylhexyl) phthalate	117-81-7	5.6[4]	RSL	10	3.0	10
		6.0[4]	MCL			
		10	LOQ			
4-Bromophenyl phenyl ether	101-55-3	No RSL / MCL		10	3.0	10
Butyl benzyl phthalate	85-68-7	16	RSL	10	3.0	10
Carbazole	86-74-8	No RSL / MCL		10	3.0	10
4-Chloroaniline	106-47-8	0.36[4]	RSL	10	3.0	10
		10	LOQ			
4-Chloro-3-methylphenol	59-50-7	1,400	RSL	10	3.0	10
2-Chloronaphthalene	91-58-7	750	RSL	10	3.0	10
2-Chlorophenol	95-57-8	91	RSL	10	3.0	10
4-Chlorophenyl phenyl ether	7005-72-3	No RSL / MCL		10	3.0	10
Chrysene	218-01-9	3.4[4]	RSL	10	3.0	10

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
		10	LOQ			
Dibenzo(a,h)anthracene	53-70-3	0.0034[4]	RSL	10	3.0	10
		10	LOQ			
Dibenzofuran	132-64-9	7.9[4]	RSL	10	3.0	10
		10	LOQ			
Di-n-butyl phthalate (butyl phthalate)	84-74-2	900	RSL	10	3.0	10
1,2-Dichlorobenzene	95-50-1	300	RSL	10	3.0	10
		600	MCL			
1,3-Dichlorobenzene	541-73-1	No RSL / MCL		10	3.0	10
1,4-Dichlorobenzene	106-46-7	0.48[3]	RSL	10	3.0	10
		75	MCL			
3,3'-Dichlorobenzidine	91-94-1	0.12[4]	RSL	10	3.0	10
		10	LOQ			
2,4-Dichlorophenol	120-83-2	46	RSL	10	3.0	10
Diethyl phthalate	84-66-2	15,000	RSL	10	3.0	10
2,4-Dimethylphenol	105-67-9	360	RSL	10	3.0	10
Dimethyl phthalate	131-11-3	No RSL / MCL		10	3.0	10
4,6-Dinitro-2-methylphenol	534-52-1	1.50[4]	RSL	20	4.5	20
		20	LOQ			
2,4-Dinitrophenol	51-28-5	39	RSL	20	6.2	20
2,4-Dinitrotoluene	121-14-2	0.24[4]	RSL	10	3.0	10

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
		10	LOQ			
2,6-Dinitrotoluene	606-20-2	0.048[4]	RSL	10	3.0	10
		10	LOQ			
Di-n-octyl phthalate	117-84-0	200	RSL	10	3.0	10
Fluoranthene	206-44-0	800	RSL	10	3.0	10
Fluorene	86-73-7	290	RSL	10	3.0	10
Hexachlorobenzene	118-74-1	0.0098[4]	RSL	10	3.0	10
		1.0[4]	MCL			
		10	LOQ			
Hexachlorobutadiene	87-68-3	0.14[4]	RSL	10	3.0	10
		10	LOQ			
Hexachlorocyclopentadiene	77-47-4	0.41[3]	RSL	10	4.5	10
		50	MCL			
Hexachloroethane	67-72-1	0.33[4]	RSL	10	3.0	10
		10	LOQ			
Indeno(1,2,3-cd)pyrene	193-39-5	0.034[4]	RSL	10	3.0	10
		10	LOQ			
Isophorone	78-59-1	78	RSL	10	3.0	10
2-Methylnaphthalene	91-57-6	36	RSL	10	3.0	10
2-Methylphenol (o-cresol)	95-48-7	930	RSL	10	3.0	10
3-Methylphenol & 4-Methylphenol (p-cresol)	106-44-5	1,900	RSL	10	3.0	10

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Naphthalene	91-20-3	0.17[4]	RSL	10	3.0	10
		10	LOQ			
2-Nitroaniline	88-74-4	190	RSL	20	3.0	20
3-Nitroaniline	99-09-2	No RSL / MCL		20	3.0	20
4-Nitroaniline	100-01-6	3.8[4]	RSL	20	3.0	20.0
		20	LOQ			
Nitrobenzene	98-95-3	0.14[4]	RSL	10	3.0	10
		10	LOQ			
2-Nitrophenol	88-75-5	No RSL / MCL		10	3.0	10
4-Nitrophenol	100-02-7	No RSL / MCL		20	3.1	20.0
N-Nitrosodimethylamine	62-75-9	0.000112[4]	RSL	10	3.0	10
		10	LOQ			
N-Nitrosodiphenylamine	86-30-6	12	RSL	10	3.0	10
N-Nitrosodi-n-propylamine	621-64-7	0.011[4]	RSL	10	3.0	10
		10	LOQ			
Pentachlorophenol	87-86-5	0.04[4]	RSL	20	5.3	20
		1.0[4]	MCL			
		20	LOQ			
Phenanthrene	85-01-8	No RSL / MCL		10	3.0	10
Phenol	108-95-2	5,800	RSL	10	3.0	10
Pyrene	129-00-0	120	RSL	10	3.0	10
Pyridine	110-86-1	20	RSL	10	3.7	10

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
2,3,4,6-Tetrachlorophenol	58-90-2	240	RSL	10	3.0	10
2,4,6-Tribromophenol	118-79-6	No RSL/MCL		10	3.0	10
1,2,4-Trichlorobenzene	120-82-1	1.1[3]	RSL	10	3.0	10
		70	MCL			
2,4,5-Trichlorophenol	95-95-4	1,200	RSL	10	3.0	10
2,4,6-Trichlorophenol	88-06-2	4.0[4]	RSL	10	3.0	10
		10	LOQ			
1-Methyl naphthalene	90-12-0	1.1[4]	RSL	10	3.0	10
		10	LOQ			

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include both EPA Regional Screening Levels (June 2015) and Maximum Contaminant Levels (MCLs) (May 2009); tables for both are included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the MCL.
[4]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Tap Water Level.

QAPP WORKSHEET #15-3 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -METALS (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater; Surface Water
Analytical Group: Metals
Concentration Level: Low

Analyte	CAS	Project Screening Level[3] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Aluminum	7429-90-5	20,000	RSL	5	1.89	5
Antimony	7440-36-0	7.8	RSL	0.03	0.0227	0.03
		6.0	MCL			
Arsenic	7440-38-2	0.052[3]	RSL	0.2	0.358	0.2
		10	MCL			
Barium	7440-39-3	3,800	RSL	0.1	0.094	0.1
		2,000	MCL			
Beryllium	7440-41-7	25	RSL	0.05	0.0145	0.05
		4.0	MCL			
Cadmium	7440-43-9	9.2	RSL	0.03	0.0127	0.03
		5.0	MCL			
Calcium	7440-70-2	No RSL / MCL		100	9.4	100
Chromium (Total)	7440-47-3	100	MCL	1	0.0736	1
Cobalt	7440-48-4	6.0	RSL	0.1	0.021	0.1
Copper	7440-50-8	800	RSL	1	0.204	1
		1,300	MCL			

Analyte	CAS	Project Screening Level[3] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Iron	7439-89-6	14,000	RSL	10	1.27	10
Lead	7439-92-1	15	RSL	0.05	0.0198	0.05
		15	MCL			
Magnesium	7439-95-4	No RSL / MCL		10	3.86	10
Manganese	7439-96-5	430	RSL	0.2	0.0735	0.2
Nickel	7440-02-0	390	RSL	0.5	0.234	0.5
Potassium	7449-09-7	No RSL / MCL		100	20	100
Selenium	7782-49-2	100	RSL	0.1	0.0425	0.1
		50	MCL			
Silver	7440-22-4	94	RSL	0.01	0.00409	0.01
Sodium	7440-23-5	No RSL / MCL		100	83.5	100
Thallium	7440-28-0	0.2	RSL	0.02	0.00342	0.02
		2.0	MCL			
Vanadium	7440-62-2	86	RSL	0.1	0.0272	0.1
Zinc	7440-66-6	6,000	RSL	2	0.711	2
Mercury	7439-97-6	0.63	RSL	0.2	0.06	0.2

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include both EPA Regional Screening Levels (June 2015) and Maximum Contaminant Levels (MCLs) (May 2009); tables for both are included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the MCL.
CAS = Chemical Abstract Service DL = Method Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Tap Water Level.

QAPP WORKSHEET #15-4 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -PCBS (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater; Surface Water
Analytical Group: PCBs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Aroclor 1016	12674-11-2	0.22[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1221	11104-28-2	0.0046[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1232	11141-16-5	0.0046[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1242	53469-21-9	0.0078[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1248	12672-29-6	0.0078[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1254	11097-69-1	0.0078[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			
Aroclor 1260	11096-82-5	0.0078[3]	RSL	0.5	0.3	0.5
		0.5	LOQ			

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs are the laboratory LOQs. EPA Regional Screening Levels (June 2015) are included for informational purposes; a table is included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the LOQ.

CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Tap Water Level.

QAPP WORKSHEET #15-5 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -TPH GRO/ DRO (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: TPH GRO/ DRO
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
TPH - GRO	8006-61-9	No RSL/MCL		100	10	100
TPH - DRO	68334-30-5	No RSL/MCL		500	150	500

[1]Achievable MDLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]Analysis for water disposal purposes.
CAS = Chemical Abstract Service MDL = Method Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-6 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -ORGANOCHLORIDE PESTICIDES (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Organochloride Pesticides
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
4,4'-DDD	72-54-8	0.3	MAC	0.05	0.03	0.05
4,4'-DDE	72-55-9	0.2	MAC	0.05	0.03	0.05
4,4'-DDT	50-29-3	0.2	MAC	0.05	0.03	0.05
Aldrin	309-00-2	Not detectable	MAC	0.05	0.03	0.05
Alpha-BHC	319-84-6	0.01[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Alpha-chlordane	5103-71-9	NA		0.05	0.03	0.05
Beta-BHC	319-85-7	0.04[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Delta-BHC	319-86-8	0.04[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Dieldrin	60-57-1	0.004[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Endosulfan I	959-98-8	NA		0.05	0.03	0.05
Endosulfan II	33213-65-9	NA		0.05	0.03	0.05
Endosulfan sulfate	1031-07-8	NA		0.05	0.03	0.05
Endrin	72-20-8	Not detectable	MAC	0.05	0.043	0.05

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Endrin aldehyde	7421-93-4	NA		0.05	0.03	0.05
Endrin ketone	53494-70-5	NA		0.05	0.03	0.05
Gamma-BHC (Lindane)	58-89-9	0.05	MAC	0.05	0.03	0.05
Gamma-chlordane	5566-34-7	NA		0.05	0.03	0.05
Heptachlor	76-44-8	0.04[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Heptachlor epoxide	1024-57-3	0.03[3]	MAC	0.05	0.03	0.05
		0.05	LOQ			
Methoxychlor	72-43-5	35.0	MAC	0.25	0.03	0.25
Toxaphene	8001-35-2	0.06[3]	MAC	2.5	0.75	2.5
		2.5	LOQ			

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
[3]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-7 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - ORGANOPHOSPHOROUS COMPOUNDS (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Organophosphorous Compounds
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Chlorpyrifos	2921-88-2	NA		1.0	0.6	1.0
Coumaphos	56-72-4	NA		2.0	0.6	2.0
Demeton O+S	8065-48-3	NA		1.0	0.6	1.0
Diazinon	333-41-5	0.7[3]	MAC	1.0	0.6	1.0
		1.0	LOQ			
Dichlorvos	62-73-7	NA		1.0	0.6	1.0
Disulfoton	298-04-4	Not detectable	MAC	4.0	0.6	4.0
Ethoprophos	13194-48-4	NA		1.0	0.6	1.0
Fensulfothion	115-90-2	NA		1.0	0.9	1.0
Fenthion	55-38-9	NA		1.0	0.6	1.0
Merphos A+B		NA		2.0	0.6	2.0
Methyl azinphos	86-50-0	NA		2.0	0.6	2.0
Methyl parathion	298-00-0	1.5	MAC	1.0	0.6	1.0
Mevinphos	7786-34-7	NA		1.0	0.6	1.0
Naled	300-76-5	NA		3.0	0.6	3.0

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.

[3]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-8 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -HERBICIDES (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Herbicides
Concentration Level: Low

Analyte	CAS	Project Screening Level[3] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
2,4,5-Trichlorophenoxyacetic acid	93-76-5	35	MAC	0.1	0.06	0.1
2,4-Dichlorophenoxyacetic acid (2,4-D)	94–75–7	50	MAC	1.0	0.26	1.0
4-(2,4-dichlorophenoxy)butanoic acid (2,4-DB)	94-82-6	NA		1.0	0.6	1.0
Dalapon	75-99-0	NA		4.0	2.1	4.0
Dicamba	1918-00-9	0.44	MAC	0.2	0.06	0.2
Dichloroprop	120-36-5	NA		1.0	0.6	1.0
Dinoseb	88-85-7	NA		1.0	0.3	1.0
MCPA	94-74-6	NA		100	60	100
MCPP	93-65-2	NA		100	60	100
Silvex	1746-01-6	NA		0.1	0.032	0.1

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP.Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-9 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -SULFIDES (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Sulfides
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Sulfide	NA	1,000	MAC	2,000	520	2,000

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-10 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -NITRATE/NITRITE AS N (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Nitrate/Nitrite as N
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Nitrate/Nitrite as N	NA	20,000	MAC	10	3.0	10
Nitrate as N	NA	20,000	MAC	10	3.0	10
Nitrite as N	NA	2,000	MAC	10	3.0	10

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-11 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -OIL AND GREASE (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Oil and Grease
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Oil and Grease	NA	15,000	MAC	5,000	0.636	5,000

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-12 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -HEXAVALENT CHROMIUM (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Hexavalent Chromium
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Hexavalent Chromium	NA	1,000	MAC	10	3.0	10

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-13 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS ‑TOTAL CYANIDE (AQ)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Total Cyanide
Concentration Level: Low

Analyte	CAS	Project Screening Levels[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Total Cyanide	NA	400	MAC	10	3.0	10

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]PSLs include NYSDEC Groundwater effluent limitations for discharges to Class GA waters - Maximum Allowable Concentrations [MAC] (Aug 2015); tables are included in Attachment A to this UFP-QAPP. Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation

QAPP WORKSHEET #15-14 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - TOTAL COLIFORMS

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water
Analytical Group: Total Coliforms
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (µg/L)	PSL Reference	Project Quantitation Limit [PQL] Goal (µg/L)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (µg/L)	Level of Quantitation [LOQ] Goal (µg/L)
Total Coliform and E. Coli	NA	NA	NA	NA	<1 CFU/ ml to 300 CFU/ 0.1 ml	NA

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method (09/09/2015).
[2]Analysis for water disposal purposes.
CAS = Chemical Abstract Service DL = Detection Limit NA = not applicable LOQ = Level of Quantitation

QAPP WORKSHEET #15-15 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - VOCS(S)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil; Sediment
Analytical Group: VOCs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Acetone	67-64-1	61,000	RSL	0.02	0.00846	0.02
		0.05	SCO			
Benzene	71-43-2	1.2	RSL	0.005	0.00116	0.005
		0.06	SCO			
Bromobenzene	108-86-1	290	RSL	0.005	0.001	0.005
Bromochloromethane	74-97-5	150	RSL	0.005	0.00103	0.005
Bromodichloromethane	75-27-4	0.29	RSL	0.005	0.00112	0.005
Bromoform	75-25-2	19.0	RSL	0.005	0.00106	0.005
Bromomethane	74-83-9	6.8	RSL	0.005	0.00114	0.005
2-Butanone (Methyl ethyl ketone - MEK)	78-93-3	27,000	RSL	0.02	0.00554	0.02
		0.12	SCO			
n-Butylbenzene	104-51-8	3,900	RSL	0.005	0.00105	0.005
		12	SCO			
sec-Butylbenzene	135-98-8	7,800	RSL	0.005	0.00113	0.005
		11	SCO			
tert-Butylbenzene	98-06-6	7,800	RSL	0.005	0.00112	0.005

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
		5.9	SCO			
Carbon disulfide	75-15-0	770	RSL	0.005	0.00107	0.005
Carbon tetrachloride	56-23-5	0.65	RSL	0.005	0.00143	0.005
		0.76	SCO			
Chlorobenzene	108-90-7	280	RSL	0.005	0.000915	0.005
		1.1	SCO			
Chlorodibromomethane (Dibromochloromethane)	124-48-1	0.75	RSL	0.005	0.00103	0.005
Chloroethane	75-00-3	14,000	RSL	0.005	0.00141	0.005
Chloroform	67-66-3	0.32	RSL	0.005	0.00126	0.005
		0.37	SCO			
1-Chlorohexane	544-10-5	No RSL / SCO		0.005	0.00106	0.005
Chloromethane	74-87-3	110	RSL	0.005	0.0013	0.005
2-Chlorotoluene	95-49-8	1,600	RSL	0.005	0.00111	0.005
4-Chlorotoluene	106-43-4	1,600	RSL	0.005	0.00103	0.005
1,2-Dibromo-3-chloropropane	96-12-8	0.0053[3]	RSL	0.01	0.00222	0.01
		0.01	LOQ			
1,2-Dibromoethane	106-93-4	0.036	RSL	0.005	0.00107	0.005
Dibromomethane	74-95-3	23	RSL	0.005	0.00128	0.005
1,2-Dichlorobenzene	95-50-1	1,800	RSL	0.005	0.00108	0.005
		1.1	SCO			
1,3-Dichlorobenzene	541-73-1	No RSL		0.005	0.000966	0.005

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
		2.4	SCO			
1,4-Dichlorobenzene	106-46-7	2.6	RSL	0.005	0.0011	0.005
		1.8	SCO			
Dichlorodifluoromethane	75-71-8	87	RSL	0.005	0.00129	0.005
1,1-Dichloroethane	75-34-3	3.6	RSL	0.005	0.00118	0.005
		0.27	SCO			
1,2-Dichloroethane	107-06-2	0.46	RSL	0.005	0.00129	0.005
		0.02	SCO			
cis-1,2-Dichloroethene	156-59-2	160	RSL	0.005	0.00109	0.005
		0.25	SCO			
trans-1,2-Dichloroethene	156-60-5	1,600	RSL	0.005	0.00116	0.005
		0.19	SCO			
1,1-Dichloroethene	75-35-4	230	RSL	0.005	0.00123	0.005
		0.33	SCO			
1,2-Dichloropropane	78-87-5	1.00	RSL	0.005	0.00112	0.005
1,3-Dichloropropane	142-28-9	1,600	RSL	0.005	0.00111	0.005
2,2-Dichloropropane	594-20-7	No RSL / MCL		0.005	0.00113	0.005
cis-1,3-Dichloropropene	10061-01-5	No RSL / MCL		0.005	0.00108	0.005
trans-1,3-Dichloropropene	10061-02-6	No RSL / MCL		0.005	0.00119	0.005
1,1-Dichloropropene	563-58-6	No RSL / MCL		0.005	0.00115	0.005
Ethylbenzene	100-41-4	5.8	RSL	0.005	0.00102	0.005

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
		1.0	SCO			
Hexachlorobutadiene	87-68-3	1.2	RSL	0.005	0.00109	0.005
2-Hexanone (Methyl butyl ketone)	591-78-6	200	RSL	0.02	0.00611	0.02
Iodomethane	74-88-4	No RSL / MCL		0.005	0.000972	0.005
Isopropylbenzene (Cumene)	98-82-8	1,900	RSL	0.005	0.00116	0.005
p-Isopropyltoluene	99-87-6	No RSL / MCL		0.005	0.00113	0.005
Methylene chloride (Dichloromethane or DCM)	75-09-2	57	RSL	0.005	0.00137	0.005
		0.05	SCO			
4-Methyl-2-pentanone (MIBK)	108-10-1	5,300	RSL	0.02	0.00651	0.02
Methyl tert-butyl ether (MTBE)	1634-04-4	47	RSL	0.005	0.00132	0.005
		0.93	SCO			
Naphthalene	91-20-3	3.80	RSL	0.005	0.00107	0.005
n-Propylbenzene	103-65-1	3,800	RSL	0.005	0.00111	0.005
		3.9	SCO			
Styrene	100-42-5	6,000	RSL	0.005	0.000957	0.005
1,1,1,2-Tetrachloroethane	630-20-6	2.0	RSL	0.005	0.000994	0.005
1,1,2,2-Tetrachloroethane	79-34-5	0.6	RSL	0.005	0.0011	0.005
Tetrachloroethene	127-18-4	24	RSL	0.005	0.00109	0.005
		1.3	SCO			

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Toluene	108-88-3	4,900	RSL	0.005	0.00101	0.005
		0.7	SCO			
1,2,3-Trichlorobenzene	87-61-6	63	RSL	0.005	0.0011	0.005
1,2,4-Trichlorobenzene	120-82-1	24	RSL	0.005	0.00108	0.005
1,1,1-Trichloroethane	71-55-6	8,100	RSL	0.005	0.00132	0.005
		0.68	SCO			
1,1,2-Trichloroethane	79-00-5	1.1	RSL	0.005	0.00123	0.005
Trichloroethene	79-01-6	0.94	RSL	0.005	0.00109	0.005
		0.47	SCO			
Trichlorofluoromethane	75-69-4	730	RSL	0.005	0.00136	0.005
1,2,3-Trichloropropane	96-18-4	0.005	RSL	0.005	0.00127	0.005
1,2,4-Trimethylbenzene	95-63-6	58	RSL	0.005	0.0011	0.005
		3.6	SCO			
1,3,5-Trimethylbenzene	108-67-8	780	RSL	0.005	0.00113	0.005
		8.4	SCO			
Vinyl acetate	108-05-4	910	RSL	0.02	0.0015	0.02
Vinyl chloride	75-01-4	0.059	RSL	0.005	0.00131	0.005
		0.02	SCO			
Xylenes (Mixed)	1330-20-7	650	RSL	0.005 (M+P-Xylene)	0.00207(M+P-Xylene)	0.005 (M+P-Xylene)
		0.26	SCO	0.005 (O-Xylene)	0.00109 (O-Xylene)	0.005 (O-Xylene)

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method.
[2]PSLs include both EPA Regional Screening Levels (June 2015) and NYSDEC Division of Environmental Remediation soil cleanup objectives (SCOs) for contaminants; tables are included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Residential Soil Level.

QAPP WORKSHEET #15-16 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS - SVOCS(S)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil; Sediment
Analytical Group: SVOCs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Acenaphthene	83-32-9	3,600	RSL	0.333	0.1	0.333
		20	SCO			
Acenaphthylene	208-96-8	100	SCO	0.333	0.1	0.333
Aniline	62-53-3	95	RSL	0.333	0.1	0.333
Anthracene	120-12-7	18,000	RSL	0.333	0.1	0.333
		100	SCO			
Azobenzene	103-33-3	5.6	RSL	0.333	0.1	0.333
Benzo(a)anthracene	56-55-3	0.16	RSL	0.333	0.1	0.333
		1	SCO			
Benzo(b)fluoranthene	205-99-2	0.16	RSL	0.333	0.1	0.333
		1	SCO			
Benzo(k)fluoranthene	207-08-9	1.60	RSL	0.333	0.1	0.333
		0.8	SCO			
Benzoic acid	65-85-0	250,000	RSL	1.670	0.666	1.670
Benzo(g,h,i)perylene	191-24-2	100	SCO	0.333	0.1	0.333
Benzo(a)pyrene	50-32-8	0.016[3]	RSL	0.333	0.1	0.333

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
		1	SCO			
Benzyl alcohol	100-51-6	6,300	RSL	0.333	0.1	0.333
bis(2-Chloroethoxy)methane	111-91-1	190	RSL	0.333	0.1	0.333
bis(2-Chloroethyl) ether	111-44-4	0.23[4]	RSL	0.333	0.1	0.333
		0.333	LOQ			
bis(2-Ethylhexyl) phthalate	117-81-7	39	RSL	0.333	0.1	0.333
4-Bromophenyl phenyl ether	101-55-3	No RSL/SCO		0.333	0.1	0.333
Butyl benzyl phthalate	85-68-7	290	RSL	0.333	0.1	0.333
Carbazole	86-74-8	No RSL/SCO		0.333	0.1	0.333
4-Chloroaniline	106-47-8	2.70	RSL	0.333	0.1	0.333
4-Chloro-3-methylphenol	59-50-7	6,300	RSL	0.333	0.1	0.333
2-Chloronaphthalene	91-58-7	4,800	RSL	0.333	0.1	0.333
2-Chlorophenol	95-57-8	390	RSL	0.333	0.1	0.333
4-Chlorophenyl phenyl ether	7005-72-3	No RSL / SCO		0.333	0.1	0.333
Chrysene	218-01-9	16	RSL	0.333	0.1	0.333
		1	SCO			
Dibenzo(a,h)anthracene	53-70-3	0.016	RSL	0.333	0.1	0.333
		0.33	SCO			
Dibenzofuran	132-64-9	73	RSL	0.333	0.1	0.333

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Di-n-butyl phthalate (butyl phthalate)	84-74-2	6,300	RSL	0.333	0.1	0.333
1,2-Dichlorobenzene	95-50-1	600	RSL	0.333	0.1	0.333
1,3-Dichlorobenzene	541-73-1	No RSL/SCO		0.333	0.1	0.333
1,4-Dichlorobenzene	106-46-7	2.60	RSL	0.333	0.1	0.333
3,3'-Dichlorobenzidine	91-94-1	1.20	RSL	0.333	0.1	0.333
2,4-Dichlorophenol	120-83-2	190	RSL	0.333	0.1	0.333
Diethyl phthalate	84-66-2	51,000	RSL	0.333	0.1	0.333
2,4-Dimethylphenol	105-67-9	1,300	RSL	0.333	0.1	0.333
Dimethyl phthalate	131-11-3	No RSL/SCO		0.333	0.1	0.333
4,6-Dinitro-2-methylphenol	534-52-1	5.10	RSL	0.667	0.24	0.667
2,4-Dinitrophenol	51-28-5	130	RSL	0.667	0.15	0.667
2,4-Dinitrotoluene	121-14-2	1.70	RSL	0.333	0.1	0.333
2,6-Dinitrotoluene	606-20-2	0.36	RSL	0.333	0.1	0.333
Di-n-octyl phthalate	117-84-0	630	RSL	0.333	0.1	0.333
Fluoranthene	206-44-0	2,400	RSL	0.333	0.1	0.333
		100	SCO			
Fluorene	86-73-7	2,400	RSL	0.333	0.1	0.333
		30	SCO			
Hexachlorobenzene	118-74-1	0.21[3]	RSL	0.333	0.1	0.333
		0.33	SCO			
Hexachlorobutadiene	87-68-3	1.20	RSL	0.333	0.1	0.333

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Hexachlorocyclopentadiene	77-47-4	1.80	RSL	0.333	0.15	0.333
Hexachloroethane	67-72-1	1.80	RSL	0.333	0.1	0.333
Indeno(1,2,3-cd)pyrene	193-39-5	0.16[3]	RSL	0.333	0.1	0.333
		0.5	SCO			
Isophorone	78-59-1	570	RSL	0.333	0.1	0.333
2-Methylnaphthalene	91-57-6	240	RSL	0.333	0.1	0.333
2-Methylphenol (o-cresol)	95-48-7	3,200	RSL	0.333	0.1	0.333
		0.33	SCO			
3-Methylphenol (m-cresol)	106-44-5	6,300	RSL	0.333	0.1	0.333
		0.33	SCO			
4-Methylphenol (p-cresol)	106-44-5	6,300	RSL	0.333	0.1	0.333
		0.33	SCO			
Naphthalene	91-20-3	3.80	RSL	0.333	0.1	0.333
		12	SCO			
2-Nitroaniline	88-74-4	630	RSL	0.667	0.1	0.667
3-Nitroaniline	99-09-2	No RSL/SCO		0.667	0.1	0.667
4-Nitroaniline	100-01-6	27.0	RSL	0.667	0.1	0.667
Nitrobenzene	98-95-3	5.10	RSL	0.333	0.1	0.333
2-Nitrophenol	88-75-5	No RSL/SCO		0.333	0.1	0.333
4-Nitrophenol	100-02-7	No RSL/SCO		0.667	0.1	0.667
N-Nitrosodimethylamine	62-75-9	0.002[4]	RSL	0.333	0.1	0.333

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
		0.333	LOQ			
N-Nitrosodiphenylamine	86-30-6	110	RSL	0.333	0.1	0.333
N-Nitrosodi-n-propylamine	621-64-7	0.078[4]	RSL	0.333	0.1	0.333
		0.333	LOQ			
Pentachlorophenol	87-86-5	1.00	RSL	0.667	0.2	0.667
		0.8	SCO			
Phenanthrene	85-01-8	100	SCO	0.333	0.1	0.333
Phenol	108-95-2	19,000	RSL	0.333	0.1	0.333
		0.33	SCO			
Pyrene	129-00-0	1,800	RSL	0.333	0.1	0.333
		100	SCO			
Pyridine	110-86-1	78	RSL	0.333	0.17	0.333
2,3,4,6-Tetrachlorophenol	58-90-2	1,900	RSL	0.333	0.13	0.333
1,2,4-Trichlorobenzene	120-82-1	24	RSL	0.333	0.1	0.333
2,4,5-Trichlorophenol	95-95-4	6,300	RSL	0.333	0.1	0.333
2,4,6-Trichlorophenol	88-06-2	49	RSL	0.333	0.1	0.333
1-Methyl naphthalene	90-12-0	18.0	RSL	0.333	0.1	0.333

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method.
[2]PSLs include both EPA Regional Screening Levels (June 2015) and NYSDEC Division of Environmental Remediation soil cleanup objectives (SCOs) for contaminants; tables are included in Attachment A to this UFP-QAPP.
[3]Level is presented for informational purposes only. Screening will be based on the SCO.
[4]Level is presented for informational purposes only. Screening will be based on the LOQ.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Residential Soil Level.

QAPP WORKSHEET #15-17 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -METALS (S)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil; Sediment
Analytical Group: Metals
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Aluminum	7429-90-5	77,000	RSL	0.50	0.1560	0.50
Antimony	7440-36-0	31	RSL	0.003	0.0017	0.003
Arsenic	7440-38-2	0.68	RSL	0.02	0.0033	0.02
		13	SCO			
Barium	7440-39-3	15,000	RSL	0.01	0.0058	0.01
		350	SCO			
Beryllium	7440-41-7	160	RSL	0.005	0.0014	0.005
		7.2	SCO			
Cadmium	7440-43-9	71	RSL	0.003	0.0017	0.003
		2.5	SCO			
Calcium	7440-70-2	No RSL /SCO		10	0.911	10
Chromium (Total)	7440-47-3	No RSL/SCO		0.10	0.0072	0.10
Cobalt	7440-48-4	23	RSL	0.01	0.0052	0.01
Copper	7440-50-8	3,100	RSL	0.10	0.0245	0.10
		50	SCO			
Iron	7439-89-6	55,000	RSL	1.0	0.337	1.0

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Lead	7439-92-1	400	RSL	0.005	0.0019	0.005
		63	SCO			
Magnesium	7439-95-4	No RSL / SCO		1.0	0.366	1.0
Manganese	7439-96-5	1,800	RSL	0.02	0.0062	0.02
		1,600	SCO			
Nickel	7440-02-0	1,500	RSL	0.05	0.0254	0.05
		30	SCO			
Potassium	7449-09-7	No RSL / SCO		10	1.760	10
Selenium	7782-49-2	390	RSL	0.01	0.0037	0.01
		3.9	SCO			
Silver	7440-22-4	390	RSL	0.001	0.0005	0.001
		2.0	SCO			
Sodium	7440-23-5	No RSL / SCO		10	1.66	10
Thallium	7440-28-0	No RSL / SCO		0.002	0.0004	0.002
Vanadium	7440-62-2	No RSL / SCO		0.01	0.0044	0.01
Zinc	7440-66-6	109	SCO	0.20	0.0421	0.20
Mercury	7439-97-6	9.4	RSL	0.033	0.0036	0.033
		0.18	SCO			

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method.
[2]PSLs include both EPA Regional Screening Levels (June 2015) and NYSDEC Division of Environmental Remediation soil cleanup objectives (SCOs) for contaminants; tables are included in Attachment A to this UFP-QAPP.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Residential Soil Level.

QAPP WORKSHEET #15-18 – PROJECT SCREENING LEVELS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS -PCBS (S)

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil; Sediment
Analytical Group: PCBs
Concentration Level: Low

Analyte	CAS	Project Screening Level[2] [PSL] (mg/kg)	PSL Reference	Project Quantitation Limit [PQL] Goal (mg/kg)	Achievable Laboratory Limits[1]	
					Method Detection Limit [DL] (mg/kg)	Level of Quantitation [LOQ] Goal (mg/kg)
Aroclor 1016	12674-11-2	4.10	RSL	0.0166	0.01	0.0166
Aroclor 1221	11104-28-2	0.17	RSL	0.0166	0.01	0.0166
Aroclor 1232	11141-16-5	0.17	RSL	0.0166	0.01	0.0166
Aroclor 1242	53469-21-9	0.23	RSL	0.0166	0.01	0.0166
Aroclor 1248	12672-29-6	0.23	RSL	0.0166	0.01	0.0166
Aroclor 1254	11097-69-1	0.24	RSL	0.0166	0.01	0.0166
Aroclor 1260	11096-82-5	0.24	RSL	0.0166	0.01	0.0166

[1]Achievable DLs and LOQs are limits that ALS can achieve when performing the specific analytical method.
[2]PSLs based on EPA Regional Screening Levels (June 2015); tables are included in Attachment A to this UFP-QAPP.
CAS = Chemical Abstract Service DL = Detection Limit NA = not available LOQ = Level of Quantitation
RSL = USEPA Regional Screening Level Residential Soil Level.

QAPP WORKSHEET #17 - SAMPLING DESIGN AND RATIONALE

(UFP-QAPP Manual Section 3.1.1)

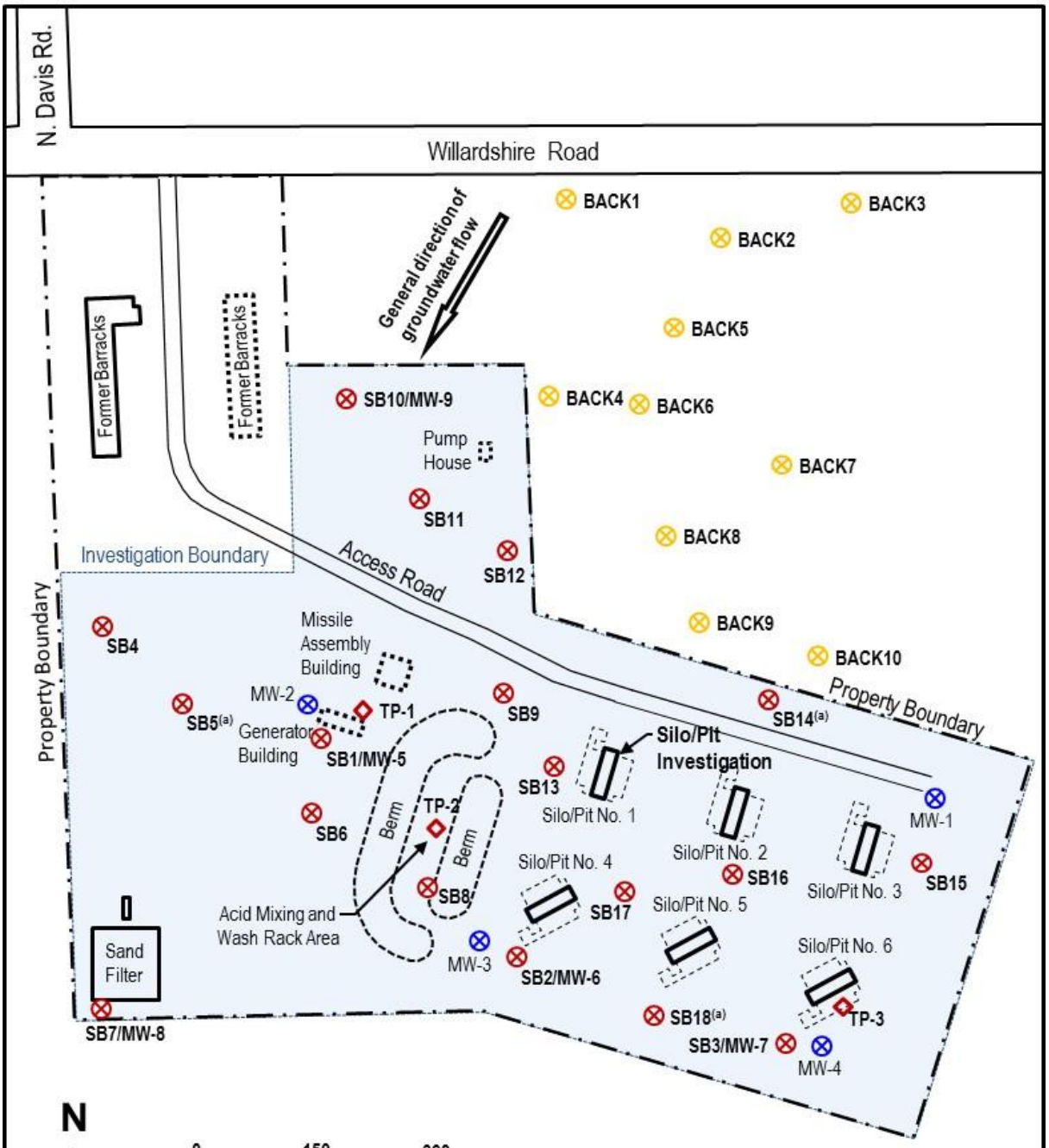
Physical boundaries for the area under the study:

The Launch Area (Figure 1) formerly occupied 19.84 acres (fee parcels) and 26 acres (easement parcels) located at 601 Willardshire Road in East Aurora, New York (USDOA, 1958). Most, if not all, of the former Nike Missile operations were located on the fee parcels except for wastewater structures including the Launch Area sanitary sewer line and outfall to Cazenovia Creek.

Sampling Design

Sampling will take place in the Launch Area as designated in the site layout map provided in Figure 2.

Soil and groundwater data presented in the 1996 RI/FS Report for the Launch Area are over 19 years old and are limited in vertical and horizontal extent. Twenty eight new soil borings (SB1 through SB18 and BACK1 through BACK10) will be installed, ten of which are located in the background area. Five of these soil borings will be converted into monitoring wells, one of which will be designated as a background well. Three test pits will be installed and soil samples collected from 3 depth intervals from each test pit (1 ft bgs, 3 ft bgs, and 5 ft bgs). Soil samples will be collected from three depth intervals for the 18 investigation soil borings (representing the 0-1 ft, 2-8 ft, and 8-15 ft bgs depth ranges) and from two depth intervals for background soil borings (0-1 ft and approximately 2-15 ft bgs). In addition, near surface samples (defined as 0 to 2 inches bgs) will be collected from five soil boring locations and 5 background sampling locations. The rationale for decision units and sampling depth intervals are presented in Worksheet 11. Groundwater samples from the nine monitoring wells (including 4 existing monitoring wells) will be collected and analyzed for the suite of analytes listed in Worksheet 11.



Note: Soil samples representative of near surface (0-2 in bgs), surface (0-1 ft bgs), fill (2-8 ft bgs), and native material (8-15 ft bgs) will be collected from soil borings consistent with Table 3-2.

LEGEND
- Existing Monitoring Well
- Proposed Boring/Monitoring Well
- Proposed Background Boring
- Proposed Test Pit

(a) Locate boring within drainage ditch based on field observations

February 2016

Prepared by: K. DeSilva

Approved by: K. Fields

TI2E, A Joint Venture Between Tidewater and IEI

Figure 2. Proposed Locations for Borings/Monitoring Wells and Test Pits Launch Area, Nike BU 34/35 East Aurora, NY

Silo Water Samples

Water samples will be collected from Silo/Pits 1 through 5 in the Launch Area. A surface and a deep water sample will be collected from each of the silo/pits and analyzed for the analytes listed in Worksheet 11. In addition, a composite water sample will be collected during pumping from Silo 1 to evaluate if contents meet NYDPES discharge criteria.

Field QC Samples

Field QC will consist of field-designated MS/MSDs, field duplicates, and trip blanks. Field-designated MS/MSDs will be collected at the standard collection frequency of 5 percent (i.e., one MS/MSD per 20 samples). Blind field duplicates will be collected at a frequency of 10 percent. One trip blank will accompany each cooler shipped to the analytical laboratory that contains samples for VOC analysis. All samples will be collected in appropriate sample containers with appropriate preservatives, and stored on ice at 4 degrees centigrade (^{o}C) ± 2^{o}C. Refer to QAPP Worksheets 11, 18, 19, and 20 for sample locations, sample identifications (IDs), specific QC samples, and sample container and preservative requirements.

QAPP WORKSHEET #18 - SAMPLING LOCATIONS AND METHODS

(UFP-QAPP Manual Section 3.1.1 and 3.1.2)

Sampling Location / ID Number	Matrix[3]	Depth[2] (Feet)	Analytical Group	Number of Samples[4]	Sampling SOP Reference[1]	Comments
Launch Area						
5 New Monitoring Wells (MW-5, MW-6, MW-7, MW-8, MW-9); 4 Existing Monitoring Wells (MW-1, MW-2, MW-3, and MW-4)	GW	15-18	VOCs, SVOCs, PCBs, Metals, Anions, and TOC	9+1 Duplicate	SOP No. 2 – Groundwater Sampling (**Attachment C**)	Determine presence or absence of VOCs, SVOCs PCBs, Metals, Anions, and TOC in groundwater; obtain representative samples for risk assessment purposes; establish background concentrations (at 2 of the 9 wells)
5 Silos (Silo1-SW, Silo1-Deep, Silo2-SW, Silo2-Deep, Silo3-SW, Silo3-Deep, Silo4-SW, Silo4-Deep, Silo5-SW, Silo5-Deep,)	SW	Surface and Bottom of each Silo	VOCs, SVOCs, PCBs, Metals, Anions, and TOC	10+1 Duplicate	SOP No. 10 – Surface water Sampling (**Attachment C**)	Determine presence or absence of VOCs, SVOCs PCBs, Metals, Anions, and TOC in silo water; obtain representative samples for assessing risk

Sampling Location / ID Number	Matrix[3]	Depth[2] (Feet)	Analytical Group	Number of Samples[4]	Sampling SOP Reference[1]	Comments
1 Composite Surface Water from Silo Pumping	SW	NA	VOCs, SVOCs, PCBs, Metals, Anions, TOC, TPH-DRO/GRO, Organochloride Pesticides, Organophosphorous Compounds, Herbicides, Sulfides, Nitrogen, Oil and Grease, Hexavalent Chromium, Total Cyanide, Ignitability, and Total coliforms	1	SOP No. 10 – Surface water Sampling (**Attachment C**)	Determine if water quality meets NYPDES discharge criteria
18 Soil Borings (SB1 through SB18) (3 samples/boring except for VOCs; an additional near surface sample collected at 0 to 2 inches bgs at 5 locations)	Soil	0-1, 2-8, 8-15	VOCs, SVOCs, PCBs, and Metals (TOC and Grain Size at SB5, SB14, and SB18 only)	59+6 Duplicate	SOP No. 5 – Soil Sampling (**Attachment C**)	Determine presence or absence of VOCs, SVOCs PCBs, and Metals in soil; obtain representative samples for risk assessment purposes. VOC samples will be collected from subsurface samples only.

Sampling Location / ID Number	Matrix[3]	Depth[2] (Feet)	Analytical Group	Number of Samples[4]	Sampling SOP Reference[1]	Comments
10 Soil Borings (BACK1 through BACK10) (2 samples/boring except an additional near surface sample collected at 0 to 2 inches bgs at 5 locations)	Soil	0-1, 8	SVOCs and Metals	25+3 Duplicate	SOP No. 5 – Soil Sampling (**Attachment C**)	Determine background levels of SVOCs and Metals in soil; obtain representative samples for risk assessment purposes
3 Test pit (3 samples/test pit)	Soil	1, 3, 5	VOCs, SVOCs, PCBs, and Metals	9+1 Duplicate	SOP No. 5 – Soil Sampling (**Attachment C**)	Determine presence or absence of VOCs, SVOCs, PCBs, and Metals in soil; obtain representative samples for risk assessment purposes

1. Specify the appropriate letter or number from the Project Sampling SOP References table (Table #21).
2. Anticipated depth of soil sample collection/estimated depth of monitoring wells.
3. GW = groundwater; SW = Surface Water
4. Random duplicate samples will be collected for 10% of soil and groundwater samples to evaluate field reproducibility. MS/MSD samples will also be collected for 5% of samples to evaluate laboratory method precision and accuracy.

QAPP WORKSHEETS #19 & #30 -SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

(UFP-QAPP Manual Section 3.1.2.2)

Analytical Group	**Matrix**	**Analytical Method /SOP**	**Accreditation Expiration Date**	**Containers** (number, size, and type)	**Preservation Requirements**	**Preparation Holding Time**	**Analytical Holding Time**	**Data Package Turnaround Time**
VOCs	Water	EPA Method 8260B, ALS SOP 525	June 01, 2016	3 x 40 mL VOA vial	HCl, 4ºC ± 2ºC, zero headspace	14 days from collection to analysis if preserved with HCl	7 days from collection to analysis if unpreserved.	2-14 days
VOCs	Soil	EPA Method 8260B, ALS SOP 525	June 01, 2016	Terracore® kit (3 x 40 mL VOA vials)	2 x 40 mL VOAs w/ 5 mL sodium bisulfate 1 x 40 mL VOA w/ 5 mL methanol, 4ºC ± 2ºC	14 days	14 days	2-14 days
SVOCs	Water	EPA Method 8270D, ALS SOP 506	June 01, 2016	1 x 1L amber	4ºC ± 2ºC	7 days	40 days	7-14 days
SVOCs	Soil	EPA Method 8270D, ALS SOP 506	June 01, 2016	1 x 4oz WM glass	4ºC ± 2ºC	14 days	40 days	7-14 days
PCBs	Water	EPA Method 8082, ALS SOP 409	June 01, 2016	1 x 1L amber	4ºC ± 2ºC	None, per SW846, 4th Edition	40 days	7-14 days

Analytical Group	Matrix	Analytical Method /SOP	Accreditation Expiration Date	Containers (number, size, and type)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
PCBs	Soil	EPA Method 8082, ALS SOP 409	June 01, 2016	1 x 4oz WM glass	4ºC ± 2ºC	None, per SW846, 4th Edition	40 days	7-14 days
TPH-DRO	Water	EPA Method 8015Mod, ALS SOP 406	June 01, 2016	1 x 1L amber	4ºC ± 2ºC	7 days	40 days	7-14 days
TPH-GRO	Water	EPA Method 8015B, ALS SOP 425	June 01, 2016	3 x 40 mL VOA vial	HCl, 4ºC ± 2ºC, zero headspace	14 days from collection to analysis if preserved with HCl	7 days from collection to analysis if unpreserved.	2-14 days
TAL Metals	Water	EPA Method 6020A, ALS SOP 806/827	June 01, 2016	1 x 500 mL, HDPE	HNO_3, pH < 2; 4ºC ± 2ºC	180 days	180 days	7-14 days
TAL Metals	Soil	EPA Method 6020A, ALS SOP 806/827	June 01, 2016	1 x 4oz WM glass	4ºC ± 2ºC	180 days	180 days	7-14 days
Mercury	Water	EPA Method 7470A, ALS SOP 812	June 01, 2016	1 x 500 mL, HDPE	HNO_3, pH < 2; 4ºC ± 2ºC	28 days	28 days	7-14 days
Mercury	Soil	EPA Method 7471A, ALS SOP 812	June 01, 2016	1 x 4oz WM glass	4ºC ± 2ºC	28 days	28 days	7-14 days
Inorganic Anions	Water	EPA 300.0 and SW9056,	June 01, 2016	1 x 500 mL, glass/HDPE	4ºC ± 2ºC	48 hours	28 days	7-14 days

Analytical Group	Matrix	Analytical Method /SOP	Accreditation Expiration Date	Containers (number, size, and type)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
		ALS SOP 1113						
TOC	Water	EPA 415.1, SW9060 A, SM5310 C,ALS SOP 670	June 01, 2016	1 x 1000 mL, amber glass	HCl, pH < 2;4ºC ± 2ºC		28 days	7-14 days
Organochloride pesticides	Water	SW 8081 A or B, ALS SOP 402	June 01, 2016	1 x 1000 mL, amber glass	4ºC ± 2ºC, Sodium thiosulfate may be used to dechlorinate liquid samples that contain residual chlorine	7 days	40 days	7-14 days
Organophosphorous Compounds	Water	EPA 8141 A or B, and EPA 614, ALS SOP 407	June 01, 2016	1 x 1000 mL, amber glass	4ºC ± 2ºC	7 days	40 days	7-14 days
Herbicides	Water	SW8151A, EPA 615 and EPA 515.1 ALS SOP 434	June 01, 2016	1 x 1000 mL, amber glass	4ºC ± 2ºC, Sodium thiosulfate may be used to dechlorinate liquid samples that contain residual chlorine	7 days	40 days	7-14 days
Sulfide	Water	EPA 376.1 and SM4500 S^2 F, ALS SOP 1120	June 01, 2016	1 x 500 mL, HDPE	2.0mL of 2N Zinc Acetate Solution and 1.5 mL of 20% Sodium Hydroxide Solution, 4ºC ± 2ºC	7 days	40 days	7-14 days

Analytical Group	Matrix	Analytical Method /SOP	Accreditation Expiration Date	Containers (number, size, and type)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
Nitrogen as Nitrate plus Nitrite	Water	EPA 353.2, SM 4500, ALS SOP 1127	June 01, 2016	1 x 500 mL, HDPE	HCl, pH < 2;4ºC ± 2ºC	7 days For analysis of NO_2^--N only, aqueous samples must be analyzed within 48 hours after collection.	28 days	7-14 days
Oil and Grease	Water	EPA 1664 A, and SW9070A,ALS SOP 671	June 01, 2016	1 x 1000 mL, glass	4ºC ± 2ºC		28 days	7-14 days
Hexavalent Chromium	Water	SW3060A and 7196A, ALS SOP 1121	June 01, 2016	1 x 500 mL, glass/HDPE	4ºC ± 2ºC	7 days	30 days	7-14 days
Total Cyanide	Water	SW9010C, SW9013, EPA 335.1, EPA 335.2, ALS SOP 1110	June 01, 2016	1 x 500 mL, glass/HDPE	Sodium Hydroxide; pH>12, 4ºC ± 2ºC	14 days	14 days	7-14 days
Ignitability	Water	EPA Method 1010A ALS SOP 629	June 01, 2016	1 x 250 mL, glass	4ºC ± 2ºC			7-14 days
Total Coliforms	Water	SM 9222		1 x 500 mL, glass/HDPE	4ºC ± 2ºC	6 hours (collection to lab receipt);		

Analytical Group	Matrix	Analytical Method /SOP	Accreditation Expiration Date	Containers (number, size, and type)	Preservation Requirements	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround Time
						2 hours (lab receipt to analysis)		

ALS SOPs are provided in Attachment D

Free Chlorine must be removed by the appropriate addition of Na2S2O3 at the time of collection. This preservation is not necessary if free chlorine is not present in the groundwater.

HCl = hydrochloric acid

HDPE = high density polyethylene

HNO_3 = nitric acid

mL = milliliter

^{o}C = degrees Centigrade

QAPP WORKSHEET #20 - FIELD QUALITY CONTROL SAMPLE SUMMARY

(UFP-QAPP Manual Section 3.1.1 and 3.1.2)

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSDs (5%)	No. of Trip Blanks[3]	No. of Equip. Blanks[4]	Other	Total No. of Samples to Lab
Launch Area										
GW	VOCs	Low	SW8260B ALS SOP 525	9	1	1	1/cooler	0	0	11 (+TB)
SW	VOCs	Low	SW8260B ALS SOP 525	11	1	1	1/cooler	0	0	13 (+TB)
Soil	VOCs	Low	SW8260B ALS SOP 525	42	5	1	1/cooler	2	0	50 (+TB)
GW	SVOCs	Low	SW8270C ALS SOP 506	9	1	1	0	0	0	11
SW	SVOCs	Low	SW8270C ALS SOP 506	11	1	1	0	0	0	13
Soil	SVOCs	Low	SW8270C ALS SOP 506	93	10	5	0	0	0	108
GW	PCBs	Low	SW8082 ALS SOP 409	9	1	1	0	0	0	11
SW	PCBs	Low	SW8082 ALS SOP 409	11	1	1	0	0	0	13
Soil	PCBs	Low	SW8082 ALS SOP 409	68	7	3	0	0	0	78
GW	Metals & Mercury	Low	SW6020A, 7470A ALS SOP 806/827/812	9	1	1	0	0	0	11
SW	Metals & Mercury	Low	SW6020A, 7470A ALS SOP 806/827/812	11	1	1	0	0	0	13
Soil	Metals & Mercury	Low	SW6020A, 7471A ALS SOP 806/827/812	93	10	5	0	0	0	108

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSDs (5%)	No. of Trip Blanks[3]	No. of Equip. Blanks[4]	Other	Total No. of Samples to Lab
GW	Anions	Low	300 & SW9056, ALS SOP 1113	9	1	1	0	0	0	11
SW	Anions	Low	300 & SW9056, ALS SOP 1113	11	1	1	0	0	0	13
GW	TOC	Low	415.1, SW9060 A, SM5310 C ALS SOP 670	9	1	1	0	0	0	11
SW	TOC	Low	415.1, SW9060 A, SM5310 C ALS SOP 670	11	1	1	0	0	0	13
SW	TPH-GRO/DRO	Low	SW8015Mod, ALS SOP 406	1	0	0	0	0	0	1
SW	Organochloride pesticides	Low	SW 8081 A or B, ALS SOP 402	1	0	0	0	0	0	1
SW	Organophosphorous Compounds	Low	EPA 8141 A or B, and EPA 614 ALS SOP 407	1	0	0	0	0	0	1
SW	Herbicides	Low	SW8151A, EPA 615 and EPA 515.1 ALS SOP 434	1	0	0	0	0	0	1
SW	Sulfide	Low	EPA 376.1 and SM4500 S^2 F ALS SOP 1120	1	0	0	0	0	0	1
SW	Nitrogen as Nitrate plus Nitrite	Low	EPA 353.2, SM 4500 ALS SOP 1127	1	0	0	0	0	0	1

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSDs (5%)	No. of Trip Blanks[3]	No. of Equip. Blanks[4]	Other	Total No. of Samples to Lab
SW	Oil and Grease	Low	EPA 1664 A, and SW9070A ALS SOP 671	1	0	0	0	0	0	1
SW	Hexavalent Chromium	Low	SW3060A and 7196A, ALS SOP 1121	1	0	0	0	0	0	1
SW	Total Cyanide	Low	SW9010C, SW9013, EPA 335.1, EPA 335.2, ALS SOP 1110	1	0	0	0	0	0	1
SW	Total Coliforms		SM9222, Lab SOP 5-20-8	1	0	0	0	0	0	1

[1] Refers to independent grab samples with the exception of one composite surface water sample collected for water disposal purposes and analyzed for all of the surface water analytes listed above.
ALS SOPs are provided in Attachment D

QAPP WORKSHEET #21 - FIELD SOP REFERENCES

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 1	Water Level Measurement (**Attachment C**)	TI2E	Water Level Probe	N	
SOP No. 2	Groundwater Sampling (**Attachment C**)	TI2E	Low-Flow pump with Controller, Bailer	N	
SOP No. 3	Sample Handling and Management (**Attachment C**)	TI2E	NA	N	
SOP No. 4	Sampling Equipment Decontamination (**Attachment C**)	TI2E	Decontamination of Sampling Equipment, Field Monitoring Equipment and Personnel	N	
SOP No. 5	Soil Sampling for Chemical Analysis (**Attachment C**)	TI2E	Pre-cleaned stainless steel sampling utensils, Terra Core® kits	N	
SOP No. 6	IDW Management (**Attachment C**)	TI2E	NA	N	
SOP No. 7	Drilling and Lithologic Logging (**Attachment C**)	TI2E	Direct Push Technology (DPT) and Hollow Stem Auger (HSA) rig, air/fluid rotary rig Munsell Soil Color Chart Soil Boring Log Form, drums for cuttings	N	

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 8	Piezometer, Monitoring Well and Injection Point Construction (**Attachment C**)	TI2E	DPT, HSA rig, air/fluid rotary rig, hollow-stem auger drill rig, monitoring well construction diagram, PVC rise pipe and screen, protective casing, lock, concrete form for pads, bollards	N	
SOP No. 9	Monitoring Well Development (**Attachment C**)	TI2E	Submersible pump, well development forms	N	
SOP No. 10	Surface Water Sampling for Chemical Analysis (**Attachment C**)	TI2E	Pre-cleaned stainless steel sampling utensils	N	
SOP No. 11	Well and Borehole Abandonment (**Attachment C**)	TI2E	Grout machine, tremie pipe	N	
N/A	HoribaU-10 Water Quality Checker Manufacturers Instruction Manual (**Attachment C**)	Horiba	HoribaU-10 Water Quality Checker	N	
N/A	Volatiles Monitoring with a photoionization detector (PID) Manufacturers' Instruction Manual (**Attachment C**)	MiniRAE	PID	N	
SOP No. 12	ASTM D 4044-96R08 Slug Test SOP	ASTM	Slug, water level meter, data logger, AquiferTest software	No	

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP No. 13	Geology Scope of Services (May 2011), Section 7 SURVEYS (GENERAL) (soil boring and monitoring well survey requirements)	USACE	To be provided by a New York licensed surveyor	No	

QAPP WORKSHEET #22 - FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	SOP Reference[1]	Resp. Person	Frequency	Acceptance Criteria	Corrective Action
Horiba U-10	Calibration	Manufacturers Instruction Manual (Attachment C)	Field Team leader	Daily before use	Auto calibration successful	Re-calibrate if "Er3" or "Er4" appears in display
Horiba U-10	Maintenance	Manufacturers Instruction Manual (Attachment C)	Equipment Coordinator	Yearly	Inspection certification	N/A
PID	Calibration	Manufacturers Instruction Manual (Attachment C)	Field Team leader	Daily before use	Calibration successful	Re-calibrate if as necessary
PID	Maintenance	Manufacturers Instruction Manual (Attachment C)	Equipment Coordinator	Yearly	Inspection certification	N/A

QAPP WORKSHEET #23 - ANALYTICAL SOP REFERENCES TABLE

(UFP-QAPP Manual Section 3.2.1)

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP 525	Determination of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry - Methods SW8260 C and EPA 624	Definitive	VOCs	Gas Chromatograph/ Mass Spectrometer (GC/MS)	ALS Environmental – Fort Collins	N
SOP 807	Determination of Metals by Inductively Coupled Plasma Emission Spectroscopy – Methods 200.7	Definitive	Metals	Inductively Coupled Plasma (ICP) or Inductively Coupled Plasma Mass Spectrometer (ICPMS)	ALS Environmental – Fort Collins	N
SOP 506	Semi-volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry - Method SW8270D	Definitive	SVOCs	Gas Chromatograph/ Mass Spectrometer (GC/MS)	ALS Environmental – Fort Collins	N
SOP 812	Preparation and Determination of mercury by Cold Vapor Atomic Absorption Spectroscopy- methods SW7470A, SW7471A and EPA 245.1	Definitive	Mercury	Cold Vapor Atomic Absorption (CVAA)	ALS Environmental – Fort Collins	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP 827	Determination of Elements by Inductively Coupled Plasma Mass Spectroscopy- Methods EPA 200.8 and SW6020A	Definitive	Metals	Inductively Coupled Plasma Mass Spectrometer (ICPMS)	ALS Environmental – Fort Collins	N
SOP 629	Determination of Ignitability by the Pensky-Marten Closed Cup Tester, Methods SW1010A and ASTM93-80	Definitive	Ignitability	Pensky-Marten	ALS Environmental – Fort Collins	N
SOP 1113	Determination of Inorganic Anions by Ion Chromatography - Methods EPA 300.0 and SW9056	Definitive	Anions	Ion Chromatography	ALS Environmental – Fort Collins	N
SOP 670	Analysis of Total Organic Carbon - Methods EPA 415.1, SW9060 A, SM5310 C	Definitive	Total Organic Carbon	Automated TOC analyzer.	ALS Environmental – Fort Collins	N
SOP 409	Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography- Methods SW8082 and EPA 608	Definitive	PCBs	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N
SOP 425	Analysis of Total Volatile Petroleum Hydrocarbons (TVPH) Gasoline Range Organics (GRO) by Gas Chromatography - Methods SW8015B or D and CAL-	Definitive	GRO	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	LUFT					
SOP 406	Extractable Petroleum Hydrocarbons Analysis by Gas Chromatography (TEPH, DRO)	Definitive	DRO	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N
SOP 1126	Determination of pH by Electrometric measurement, Methods EPA 150.1, SW9040C, SW9045D and SM4500-H+B	Definitive	pH	pH Meter	ALS Environmental – Fort Collins	N
SOP 402	Determination of Organochloride Pesticides by Gas Chromatography - SW8081 A or B; and EPA 608	Definitive	Organochloride pesticides	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N
SOP 407	Organophosphorous compounds by Gas Chromatography - EPA 8141 A or B, and EPA 614	Definitive	Organophosphorous compounds	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N
SOP 434	Analysis of Chlorinated Herbicides by Gas Chromatography - SW8151A, EPA 615 and EPA 515.1	Definitive	Herbicides	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP 1120	Determination of Total Sulfides in water - EPA 376.1 AND SM4500 S^2 F	Definitive	Total Sulfides	Gas Chromatograph (GC)	ALS Environmental – Fort Collins	N
SOP 1127	Determination of Nitrogen as Nitrate plus Nitrite (NO_3^--N + NO_2^--N), Nitrite (NO_2^--N), And Nitrate (NO_3^--N) in environmental water and soil samples using a Colorimetric, Automated, Cadmium Reduction procedure - EPA 353.2, SM 4500	Definitive	Nitrogen as Nitrate and Nitrite	Colorimetry	ALS Environmental – Fort Collins	N
SOP 671	Determination of Hexane Extractable Material (HEM) and Silica Gel Treated Hexane Material (SGT-HEM) by Extraction and Gravimetry for aqueous samples - EPA 1664 A, and SW9070A	Definitive	Oil and Grease	Extraction and Gravimetry	ALS Environmental – Fort Collins	N
SOP 1121	Determination and analysis of Hexavalent Chromium in Solid Matrices using Alkaline Digestion - SW3060A And 7196A	Definitive	Hexavalent Chromium	UV/VIS Spectrophotometer	ALS Environmental – Fort Collins	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP 1110	Determination of Total and amendable Cyanide (Distillation) - SW9010C, SW9013, EPA 335.1, EPA 335.2, CLP Inorganic SOW (Ilm04.0); Determination of Weak and Dissociable Cyanide – SM4500	Definitive	Total Cyanide	Cyanide Distillation System, Spectrophotometer,	ALS Environmental – Fort Collins	N
SOP 5-20-08	Total Coliform Detection in Potable and Non-potable Water – SM9222	Definitive	Total Coliform	Petri plates/HPC media	Testamerica Labs	No

GC = gas chromatograph MS = mass spectrometer ICP = inductively coupled plasma ICAP = inductively coupled argon plasma
NA = not applicable ZHE = zero headspace extraction

QAPP WORKSHEET #24 - ANALYTICAL INSTRUMENT CALIBRATION

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
GC/MS - 8260	Tuning Criteria	Every 12 hour period	Balanced Failure Biasing (BFB) abundance criteria (Table 1) must be met	Re-tune. Do not proceed with analysis until tune meets criteria.	Analyst	525
	Initial Calibration (ICAL)	Prior to sample analysis.	Ave Relative Frequency (RF) may be used if: analytes are <20% Relative Standard Distribution (RSD) r^2 for regression (or quadratic) curve fit must be ≥0.99; a quadratic curve may be used if 6 or more data points are used	When client or method criteria are not met, reanalyze ICAL. Evaluate/correct instrument malfunction if required	Analyst	525
	Initial Calibration Verification (ICV): different source than that of ICAL standards	Following every ICAL	Measured concentrations of all analytes should be within 20% of true value. Poor performers identified by the laboratory (Acetone and Iodomethane) will be recovered to within 30% of true value. Sporadic	Re-analyze ICV. If still out, evaluate/correct instrument malfunction as needed; perform a new ICAL	Analyst	525

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
			failures allowed for up to two analytes. Internal standard (IS) retention times <30 seconds drift from mid-point in most recent ICAL. IS areas –50 to +100% of corresponding internal standard area in the mid-point of the most recent ICAL			
	Continuing Calibration Verification (CCV); at or near mid-point	Every 12-hour period following tune, if ICAL not performed. Required for quantitating all samples analyzed during the 12 hour sequence. NOTE: The ICV of the next run will be substituted for the ending CCV.	Analytes should be within 20% of expected concentrations. • See section 8.7.1. IS retention times <30 seconds drift from mid-point in most recent ICAL. IS areas –50 to +100% of corresponding internal standard areas in the mid-point of the most recent ICAL	Re-analyze the daily standard. If failure repeats, evaluate/correct instrument malfunction; perform a new ICAL. If the ICV that is being substituted for the ending CCV fails, samples between the starting CCV and ICV (at the end of the sequence) will be reanalyzed. NOTE: Recoveries that are high and outside of the stated acceptance criteria may be acceptable in some programs if the analyte that is high was not detected in the associated	Analyst	525

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
				samples.		
ICP - 6010	Initial Calibration using 4 standards and a blank	Daily	Correlation coefficient (r2) for all analytes >0.995	Correct problem and repeat initial calibration.	Analyst	834
	Reanalysis of Mix-A, Mix-B and Mix C cal standards as samples	Immediately after calibration	All analytes within 5% of expected value	Correct problem then repeat initial calibration	Analyst	834
	ICV (Initial Calibration Verification) check std (second source); at or below midpoint	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration	Analyst	834
	ICB (Initial Calibration Blank)	Immediately following ICV	Absolute value of result for each analyte < reporting limit, or as specified in applicable Laboratory Information Management System (LIMS) program specification	Correct problem then re-analyze ICB	Analyst	834
	CCV (Continuing	After every 10 samples and at	All analytes within 10% of expected value	Repeat calibration and reanalyze all samples since	Analyst	834

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Calibration Verification) check std; concentration of analytes must be different from ICV	the end of the analytical sequence		last successful CCV		
	CCB (Continuing Calibration Blank)	Immediately after every CCV	Absolute value of result for each analyte < reporting limit, or as specified in applicable LIMS program specification	Correct problem then analyze CCB and previous 10 samples	Analyst	834
	ICSA (Interference Check Solution A) and ICSB (Interference Check Solution B)	At the beginning and the end of an analytical run or once every 12 hours, whichever is more frequent	All analytes within 20% of expected value. Concentrations of non-spiked analytes should not exceed twice the RL or as otherwise specified in the applicable LIMS program specification.	Terminate analysis, correct problem, reanalyze ICSA, reanalyze all affected samples	Analyst	834
pH 150.1, 9040C, 9045D	Calibration: Standardization requires the use of at least three	Daily; each day of use	NA	The pH meter must be successfully standardized before sample analyses can proceed	Analyst	1126

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
Sm4500-H[+] B	certified buffer solutions					
	ICV	Run following the initial standardization, before any samples are analyzed	ICV results must agree within ± 0.05 pH units of the known value	If ICV fails, check the integrity of the second-source buffer solution. Replace and reanalyze. If ICV still fails, problem must be identified and corrected and pH meter re-standardized before analyses can proceed.	Analyst	1126
	CCV	Run following the analyses of every ten samples set and to close-out the run sequence	CCV results must agree within ± 0.1 pH units of the expected value.	If CCV fails, pH meter must be re-standardized and all samples run since the last acceptable CCV must be reanalyzed	Analyst	1126
	Laboratory Duplicate (DUP)	One per batch of ≤20 field samples	Aqueous duplicate values should agree within ± 0.2 pH units of each other; solid matrix duplicate values should agree within ± 0.5 pH units of each other	If criteria are not met and CCV analyses are within QC limits, narrate client data package report.	Analyst	1126
Reactive Cyanide,	Initial Calibration-	As needed (i.e., at on-set of analyses	Correlation coefficient (r^2) for linear regression	Check that the calibration standards were prepared	Analyst	1112

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
Reactive Sulfide USEPA SW846 Chapter 7	minimum 5-point plus blank (cyanide)	or repeated when continuing calibration does not meet criteria)	must be ≤ 0.995	properly. Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.		
	Independent Calibration Verification-ICV (cyanide)	Once after each initial calibration	Response must agree within ± 15% of initial calibration	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.	Analyst	1112
	Method Blank (MB) Initial Calibration Blank-ICB and Continuing Calibration Blank (CCB cyanide)	The MB may be run initially as part of the calibration curve (as applicable. (cyanide) The ICB is run following the calibration curve. CCBs are run following the CCV (after every ten samples), and to close an analytical run sequence.	No cyanide detected >LOD.	Check all calibrations. If no computation errors are found prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	1112
	Continuing Calibration	Run after every ten samples and	Response must agree within ± 10% of initial	Check the calculations and preparations are correct,	Analyst	1112

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Verification-CCV (cyanide)	to end any run sequence (must be followed by a CCB analysis)	calibration	evaluate/correct instrument malfunction, reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.		
	Laboratory Control Sample (LCS)	Once LCS must be run per 20 environmental samples processed	Laboratory limits have been established as 5-30 percent recovery (%R) for Reactive cyanide (CN), and 10-60%R for Reactive Sulfide	Check calculations and preparation for documentable errors. Of no errors are found reanalyze. Associated samples must also be reanalyzed.	Analyst	1112
	Laboratory Duplicate (DUP)	One sample DUP must be run per batch of 20 or less environmental samples processed.	RPD between the sample and its duplicate should be ≤35, for both Reactive CN and sulfide	For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department Project QA Managers.	Analyst	1112

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
GC/MS - 8270	Tuning Criteria	Every 12 hour period	DFTPP ion ratio criteria PCP Tailing <2 Benzidine Tailing <2 DDT degradation <20%	Retune. Do not proceed with analysis until tune meets criteria. Perform injection port and column maintenance. Do not proceed until tune meets criteria	Analyst	506
	Initial Calibration (ICAL)	Following major instrument maintenance; when the daily calibration do not meet criteria	All analytes: <20% RSD Linear regression, r2 ≥ 0.990 Quadratic fit, COD ≥ 0.990	Up to 10% of compounds may be noncompliant. Any noncompliant compounds may be reported as estimated values	Analyst	506
	Initial Calibration Verification (ICV): different source than that of ICAL standards	Following every ICAL	Measured concentrations of all analytes should be within 20% of true value. Poor performers identified by the laboratory (Benzoic Acid, Hexachloro cyclopentadiene, 2,4-Dinitrophenol, 4-Nitroanaline, 4,6-Dinitro-2-methylphenol, Pentachlorophenol, 3,3'-Dichlorobenzidine) will be recovered to within 30% of true value.	Up to 10% of the compounds may exceed 30% of expected concentration but the samples must be reported as estimated values.	Analyst	506

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Continuing Calibration Verification (CCV); at or near mid-point	Every 12-hour period following tune, if ICAL not performed. NOTE: The ICV of the next run will be substituted for the ending CCV.	All analytes: <20 %D	Re-analyze the daily standard. If failure repeats, evaluate/correct instrument malfunction; perform a new ICAL Up to 20% of the analytes may exceed the 20% criteria but the compounds must be reported as estimated values. If the ICV that is being substituted for the ending CCV fails, samples between the starting CCV and ICV (at the end of the sequence) will be reanalyzed. NOTE: Recoveries that are high and outside of the stated acceptance criteria may be acceptable in some programs if the analyte that is high was not detected in the associated samples.	Analyst	506
ICPMS - 6020	Tune Standard; analyzed at least 5 times consecutively	Daily before the initial calibration	Peak width <0.9amu at 10% peak height, mass calibration within 0.1amu and %RSD of	Correct problem and repeat tune standard routine.	Analyst	827

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
			replicates <5% (unless otherwise noted in Program Specification)			
	Initial Calibration; uses at least 3 standards and a blank	Daily	Correlation coefficient (r2) for all analytes >0.998	Correct problem and repeat initial calibration.	Analyst	827
	Initial Calibration. Verification (ICV); second source	Daily after initial calibration	All analytes within 10% of expected value	Correct problem then repeat initial calibration	Analyst	827
	ICB (Initial Calibration Blank)	Immediately following ICV	No analytes detected >LOQ.	Correct problem then repeat initial calibration.	Analyst	827
	Continuing Calibration Verification (CCV); conc. of analytes must be different from the ICV	After every 10 samples and at the end of the analytical sequence	All analytes within 10% of expected value	Correct problem and reanalyze all samples since last successful CCV.	Analyst	827
	CCB (Continuing	Immediately after every CCV	Absolute value of result for each analyte <	Correct problem then analyze CCB and previous	Analyst	827

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Calibration Blank)		reporting limit, or as specified in applicable LIMS program specification	10 samples		
	CRI (reporting limit check) Standard	Run immediately following the ICV/ICB	ALS does not typically control on CRI Standard response. However, if so specified by LIMS program specification, ALS will observe the following performance criteria: recovery within 50-150% for Sb, Pb and Tl; recovery within 70-130% for all other analytes.	Reanalyze failed analytes. If still outside of control limits, halt analyses, correct problem and recalibrate. Analyses may not proceed until an acceptable CRI Standard has been analyzed.	Analyst	827
	ICSA (Interference Check Solution A) and ICSAB (Interference Check Solution B)	At the beginning of an analytical run or every 12 hours whichever is more frequent	ICSA should not contain non-spiked analytes at concentrations above twice the analyte LOQ, or as otherwise specified in the applicable LIMS program specification. ISCAB: all analytes of	No directives are given in the referenced methods for this QC check. The limits indicated in this Table are used as ALS guidelines; no corrective actions are taken on an analytical batch basis.	Analyst	827

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
			interest should be within ±20% of expected value.			
CVAA-7470, 7471	Initial Calibration: Minimum 5-point plus blank	Daily at on-set of analyses or when corrective action for CCV failure does not resolve calibration verification non-compliance.	Correlation coefficient (r^2) for all analytes >0.995	Check that the calibration standards were prepared properly. Evaluate/correct instrument malfunction and reanalyze calibration standards. If quality control acceptance criterion still not met, analyses cannot proceed; a new suite of calibration standards must be prepared and analyzed. Analyses cannot proceed until an acceptable initial calibration curve is generated.	Analyst	812
	Independent Calibration Verification (ICV); second source; run at a	Once after each initial calibration	Response must agree within 10% of initial calibration	If QC criterion not met, analyze again. If ICV still fails, ICV and initial calibration standards must be re-digested and reanalyzed	Analyst	812

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	concentration at or below the midpoint of the calibration curve					
	Blanks: Preparation (Method), Initial and Continuing Calibration Blank (ICB and CCB)	ICB run following the ICV One method blank per matrix type processed and analyzed per batch of 20 or less environmental samples processed. CCB run following the CCV to bracket a set of 10 analyses and to close a run sequence.	Blank value must be less than the LOQ, or as otherwise specified in applicable LIMS program specification	If QC criterion not met for ICB, locate and correct problem; repeat initial calibration. If QC criterion not met for the method blank, the method blank and all associated samples must be re-digested and reanalyzed. If QC criterion not met for CCB, locate and correct problem; all samples analyzed since last acceptable CCB must be reanalyzed.	Analyst	812
	CRA Standard – Low- Level	Run immediately following the	No acceptance criteria applicable	No corrective actions required	Analyst	812

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Reporting Limit Standard	ICB				
	Continuing Calibration Verification (CCV) may be first or second source; run at or below the midpoint of the calibration curve	Run to bracket a set of 10 analyses, and to end any run sequence (must be followed by a CCB analysis)	Response must agree within 20% of expected value	Check for calculation errors. If no calculation errors are found, analyze again. If CCV still fails, evaluate/correct instrument malfunctions; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after last acceptable CCV must be reanalyzed.	Analyst	812
GRO-SW8015B or D	Initial Calibration (ICAL); minimum 5-points, all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, may use mean RF to quantitate, If RSD ≥20%, calculate linear regression (not forced through origin); use for quantitation if correlation coefficient (r) ≥0.995 or calculate quadratic regression (minimum of six points required); use for quantitation if COD (r2)	Evaluate/correct instrument malfunction and reanalyze ICAL to obtain acceptable curve	Analyst	425

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
			≥0.99			
	Initial Calibration Verification (ICV); conc. not equal to midpoint of calibration curve; second source	After each ICAL	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.	Analyst	425
	Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Run at start of sequence if ICAL not performed; brackets each set of 20 field sample analyses; more frequent analysis recommended.	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g., rinse or change liner, remove some of the guard column); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. Samples analyzed before and after a failed CCV must be reanalyzed. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV (bracketed by acceptable CCVs) will be	Analyst	425

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
				reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.		
	Retention Time Window (RTW); The retention time range for GRO includes 2-methyl pentane through 1,2,4 trimethlybenzene, window is checked against CCV for each batch.	Whenever a new column is installed; and checked with each batch	Note that the ICV and CCV analyses are also used to monitor RT drift	Width of GRO window should include 2-methyl pentane and 1,2,4- trimethylbenzene of bracketing CCVs.	Analyst	425
	Retention Time Shift; RT of analytes in CCV are evaluated against the	Each CCV	Column and compound specific	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate Evaluate data based on	Analyst	425

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	midpoint of the ICAL			comparison with other standards run during sequence, consider RTs for the surrogates and spiked compounds analyzed before and after the sample in question.		
DRO-GC/FID	Initial Calibration (ICAL); minimum 5-point, all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, may use mean RF to quantitate Calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r2) ≥0.99 or Calculate quadratic regression (minimum of six points required); use for quantitation if COD ≥0.99	Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.	Analyst	406
	Initial Calibration Verification (ICV);	After each ICAL conc. not equal conc. To midpoint of calibration curve; second source	≤20%D of each compound	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.	Analyst	406

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
	Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Run at start of sequence if ICAL not performed; brackets each set of 20 sample analyses; more frequent analyses recommended; standard practice is CCV every 10 samples.	≤20%D of each compound NOTE: Cal LUFT method requirements state that analyses may continue only when CCV is ±10%D. Check the LIMS Project Specification to ensure that appropriate criteria are applied.	Evaluate/correct instrument malfunction as needed (e.g., rinse or change liner, remove some of the guard column); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. - Samples analyzed before and after a failed CCV (bracketing with acceptable calibration fails) must be reanalyzed. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV (bracketed by acceptable CCVs) will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR)	Analyst	406

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
				and notify the PM for sample disposition.		
	Retention Time Window (RTW); set to include the designated extractable petroleum hydrocarbon reference standard.	Whenever a new column is installed or if a new extractable petroleum hydrocarbon range is required.	Brackets appropriate hydrocarbon elution range. Note that the ICV and CCV analyses are also used to monitor RT drift.	Perform system maintenance to correct drift. Experience of analyst weighs heavily in interpretation of chromatograms.	Analyst	406
	Retention Time Shift; RT of pattern in CCV is evaluated against the midpoint of the ICAL or the preceding CCV	Each CCV; RT of analytes evaluated against the ICAL or preceding CCV to ensure that the designated extractable petroleum hydrocarbon range has not significantly shifted	Instrument performance supports accurate quantitation of TEPH	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate Evaluate data based on comparison with other standards run during sequence, consider RTs for the surrogates and spiked compounds analyzed before and after the sample in question.	Analyst	406
Pensky-Marten	Instrument Verification	Prior to sample analysis, after	Corrected flashpoint for p-xylene must be 27°C	Clean and dry the apparatus and re-check. Criteria must	Analyst	629

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action (CA)	Person Responsible for CA	SOP Reference[2]
Closed Cup Tester		every 10 samples and at the end of the analytical sequence	±1°C.	be met before analysis may proceed or be reported from bracketing checks.		

QAPP WORKSHEET #25 - ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Ignition Port Maintenance	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	525, 506
GC/MS	Mass Selective Detector Maintenance	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	525, 506
GC/MS	Mass Selective Detector Maintenance	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	525, 506
GC/MS	Capillary Column Replacement	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	525, 506
GC/MS	Concentrator Trap Replacement	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	525
GC/MS	Purge and Trap Autosampler Maintenance	Physical Check	Physical Check	As needed	C	Service as needed.	Analyst	525
ICP, ICPMS	Check peristaltic pump tubing	Physical Check	Physical Check	Daily	NA	Change as needed.	Analyst	834, 827
ICP, ICPMS	Check torch for deposits	Physical Check	Physical Check	As needed	NA	Clean as needed.	Analyst	834, 827
ICP, ICPMS	Check drain water	Physical Check	Physical Check	Daily	NA	Empty as needed.	Analyst	834, 827

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP, ICPMS	Check nebulizer and spray chamber	Physical Check	Physical Check	Daily	NA	Clean as needed.	Analyst	834, 827
ICP, ICPMS	Clean air filters	Physical Check	Physical Check	Monthly	NA	Clean monthly.	Analyst	834, 827
ICP, ICPMS	Check entrance slit	Physical Check	Physical Check	As needed	NA	Clean as needed.	Analyst	834, 827
ICP, ICPMS	Fill water recirculating reservoir	Physical Check	Physical Check	Monthly	NA	Fill as needed.	Analyst	834, 827
GC	Ignition Port Maintenance	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	402, 409, 406, 425
GC	FID, ECD Detector Maintenance	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	402, 409, 406, 425
GC	Capillary Column Replacement	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	402, 409, 406, 425
CVAA	Pump tubing	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	812
CVAA	Check gas liquid separator	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	812
CVAA	Check rinse water and stannous chloride reservoirs	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	812
CVAA	Clean sample and	Physical	Physical Check	As needed	Acceptable	Service as	Analyst	812

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
	reference cells	Check			method performance	needed.		
CVAA	Change nafion cartridge	Physical Check	Physical Check	As needed	Acceptable method performance	Service as needed.	Analyst	812

QAPP WORKSHEET #26& 27 - SAMPLE HANDLING, CUSTODY, AND DISPOSAL

(UFP-QAPP Manual Attachment A)

Sampling Organization: TI2E
Laboratory: ALS Environmental
Method of sample delivery: Laboratory courier (Overnight delivery to laboratory or laboratory courier)
Number of Days from Reporting until sample disposal: 30 days after the final report is issued

Activity	Organization and Title of person responsible for activity	SOP Reference
Sample Collection	Field Sampling Team: TI2E	SOP No. 3 - Sample Handling and Management (Attachment C)
Sample Labeling	Field Sampling Team: TI2E	SOP No. 3 - Sample Handling and Management (Attachment C)
Chain-of-Custody form completion	Field Sampling Team: TI2E	SOP No. 3 - Sample Handling and Management (Attachment C)
Packaging	Field Sampling Team: TI2E	SOP No. 3 - Sample Handling and Management (Attachment C)
Shipping coordination	Field Sampling Team: TI2E/ laboratory courier	
Sample Receipt	Laboratory sample custodian/ALS Environmental	
Sample Custody and Storage	Laboratory sample custodian/ALS Environmental	
Sample Preparation	Laboratory sample preparation technician/ALS Environmental	
Sample Determinative Analysis	Laboratory analytical chemist/ALS Environmental	
Sample Disposal	Laboratory sample custodian/ALS Environmental	

QAPP WORKSHEET #28-1 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – VOCS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: VOCs
Analytical Method/SOP Reference[1]: SW 8260B / 525

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	Every 12-hour period; after each calibration/check and 1 per batch of 20 samples of like matrix	Re-analyze to determine if instrument contamination was the cause. If MB is still non-compliant, correct the problem and obtain a successful MB analysis before resuming analysis of samples. NOTE: Reporting of samples associated with MBs that yield contaminants may be permitted by some program specifications or at the client's discretion. Example: Toluene in MB at LOQ but not detected in any sample above the DL. In this case, document occurrence and resolution using a Nonconformance Report (NCR), SOP 928.	Analyst	< ½ LOQ for all target compounds, except common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride), which are allowable to the LOQ; or as otherwise stipulated in the applicable LIMS program specification.
Surrogates (SS)	Every standard, client sample and QC sample	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are	Analyst	See laboratory or other applicable limits; recoveries should be within these limits Laboratory limits for waters:

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		found, sample may be reanalyzed once (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If still non-compliant, report results and narrate. If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon content contributes to SS areas), reanalysis is not required. NOTE: Per program specifications, surrogate recovery that is high and outside of acceptance criteria, with no associated target compounds detected, may not require reanalysis.		4-Bromofluorobenzene 85-115 Dibromofluoromethane 84-118 Toluene-D8 85-115 Laboratory limits for soils: 4-Bromofluorobenzene 52-151 Dibromofluoromethane 61-134 Toluene-D8 57-135
Internal Standard (IS)	Every standard, client sample and QC sample	Inspect instrument for malfunction, correct. Sample may be reanalyzed (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon	Analyst	Average area within -50% to +100% window of corresponding daily calibration verification standard area RT shift <30 seconds compared to daily standard (STD50); relative retention time (RRT) of sample must be ± 0.06 RRT

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td>content contributes to IS areas), reanalysis is not required. Narrate.</td><td></td><td>units of standard</td></tr>
<tr><td>Matrix Spike (MS)</td><td>1 per batch of 20 samples of like matrix</td><td>If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause. Narrate.</td><td>Analyst</td><td>See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits.
Laboratory limits for waters:<table><tr><td>1,1-Dichloroethene</td><td>77-119</td></tr><tr><td>Benzene</td><td>83-117</td></tr><tr><td>Chlorobenzene</td><td>81-113</td></tr><tr><td>Toluene</td><td>82-113</td></tr><tr><td>Trichloroethene</td><td>83-117</td></tr></table>Laboratory limits for soils:<table><tr><td>1,1-Dichloroethene</td><td>65-136</td></tr><tr><td>Benzene</td><td>73-126</td></tr><tr><td>Chlorobenzene</td><td>75-123</td></tr><tr><td>Toluene</td><td>71-127</td></tr><tr><td>Trichloroethene</td><td>77-124</td></tr></table></td></tr>
<tr><td>Matrix Spike Duplicate (MSD) or Duplicate</td><td>1 per batch of 20 samples of like matrix</td><td>If non-compliant, check calculations for errors. If significant differences exist between the duplicate results,</td><td>Analyst</td><td>See laboratory or other applicable limits; recoveries for the spiked compounds should be within these</td></tr>
</table>

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td>consult with Department Manager (reanalysis of the sample and spikes may be necessary, or sample inhomogeneity may be the likely cause).</td><td></td><td>advisory limits. RPD’s for the spiked compounds should also be within advisory limits.
Laboratory limits for waters:<table><tr><td>1,1-Dichloroethene</td><td>77-119</td></tr><tr><td>Benzene</td><td>83-117</td></tr><tr><td>Chlorobenzene</td><td>81-113</td></tr><tr><td>Toluene</td><td>82-113</td></tr><tr><td>Trichloroethene</td><td>83-117</td></tr></table>Laboratory limits for soils:<table><tr><td>1,1-Dichloroethene</td><td>65-136</td></tr><tr><td>Benzene</td><td>73-126</td></tr><tr><td>Chlorobenzene</td><td>75-123</td></tr><tr><td>Toluene</td><td>71-127</td></tr><tr><td>Trichloroethene</td><td>77-124</td></tr></table></td></tr>
</table>

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.
TBD = to be determined

QAPP WORKSHEET #28-2 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – SVOCS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: SVOCs
Analytical Method/SOP Reference[1]: SW 8270D / 506

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Instrument Blank	Every 12-hour period; after each calibration An extraction method blank can be used. The extraction method blank should be analyzed with the associated samples	Re-analyze to determine if instrument contamination was the cause. If the instrument blank is till non-compliant, correct the problem before analysis of samples.	Analyst	< ½ LOQ for all target compounds, or as otherwise specified in applicable LIMS program specification.
Extraction Method Blank	One per extraction batch of ≤ 20 samples of similar matrix	Re-analyze to determine if instrument contamination was the cause. If MB is still non-compliant, correct the problem and obtain a successful MB analysis before resuming analysis of samples. NOTE: If problem is isolated to the method blank (Associated samples meet all IS and	Analyst	< ½ LOQ for all target compounds, or as otherwise specified in applicable program specifications

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td>SS criteria and no target compounds are detected above limits), report and complete a non-conformance report (SOP 928).</td><td></td><td></td></tr>
<tr><td>Surrogates (SS)</td><td>Every standard, client sample and QC sample</td><td>If non-compliant, check calculations and spike preparation for documentable errors. Reanalyze sample once (re-analysis requirements may be fulfilled by existing multiple extractions, e.g., MS, MSD, REP). If still out, report results and note in narrative.
<u>NOTE:</u> Because of the number of surrogates used by this method, the laboratory will allow for samples to have one acid and one base/neutral surrogate outside limits if the remaining surrogates suggest the problem is matrix related and that there were no problems</td><td>Analyst</td><td>See laboratory or other applicable limits; recoveries should be within these limits
Laboratory limits for waters:
<table>
<tr><td>2-Fluorphenol</td><td>46-105</td></tr>
<tr><td>Phenol-D5</td><td>50-109</td></tr>
<tr><td>Nitrobenzene-D5</td><td>53-111</td></tr>
<tr><td>2-Fluorobiphenyl</td><td>55-108</td></tr>
<tr><td>2,4,6-Tribromophenol</td><td>42-117</td></tr>
<tr><td>Terphenyl-D14</td><td>34-139</td></tr>
</table>
Laboratory limits for soils:
<table>
<tr><td>2-Fluorphenol</td><td>16-106</td></tr>
<tr><td>Phenol-D5</td><td>31-105</td></tr>
<tr><td>Nitrobenzene-D5</td><td>31-110</td></tr>
<tr><td>2-Fluorobiphenol</td><td>41-111</td></tr>
<tr><td>2,4,6-Tribromophenol</td><td>19-119</td></tr>
<tr><td>Terphenyl-D14</td><td>23-159</td></tr>
</table></td></tr>
</table>

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		with laboratory performance during the extraction and analysis. At the client's discretion, the sample may be submitted for re-extraction.		Surrogates will be considered diluted out, if the dilution of the extract is ≥ 10X
Internal Standard (IS)	Every standard, client sample and QC sample	Inspect instrument for malfunction, correct identified malfunctions, then reanalyze samples. If no instrument malfunction is identified, reanalyze. If analysis of sample extract is still out, report results and note in narrative.	Analyst	EICP area within -50% to +100% of previous daily calibration check standard.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	1 per extraction batch of ≤20 samples of like matrix	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause. Narrate.	Analyst	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits. Laboratory limits for waters: Phenol 60-102 2-Chlorophenol 64-100 1,4-Dichlorobenzene 54-94

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
				N-Nitroso-Di-N-propylamine 62-113 1,2,4-Trichlorobenzene 47-92 4-Chloro-3-Methylphenol 61-105 Acenaphthene 60-108 4-Nitrophenol 24-128 Pentachlorophenol 40-114 Pyrene 60-113 2,4-Dinitrotoluene 46-114 Laboratory limits for soils: Phenol 39-109 2-Chlorophenol 40-107 1,4-Dichlorobenzene 41-107 N-Nitroso-Di-N-Propylamine 42-118 1,2,4-Trichlorobenzene 39-101 4-Chloro-3-Methylphenol 50-111 Acenaphthene 47-110 4-Nitrophenol 24-126 2,4-Dinitrotoluene 47-117

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td></td><td></td><td><table><tr><td>Pentachlorophenol</td><td>32-116</td></tr><tr><td>Pyrene</td><td>48-118</td></tr></table>
RPDs for the spike compounds should also be within advisory limits</td></tr>
<tr><td>Laboratory Control Sample (LCS) or Duplicate (LCSD)</td><td>1 per extraction batch of 20 samples of like matrix; typically the LCSD is analyzed when matrix spikes are not performed</td><td>If non-compliant, check calculations and spike preparation for documentable errors; correct as needed. If no errors are found, then re-analyze to determine if instrumental conditions was the cause. Notify the Supervisor and initiate corrective action (NCR). Re-analyze associated samples, if appropriate. Note that recoveries that are high and outside of acceptance criteria may be acceptable, when the same target compound is not detected in any sample in the batch. Narrate.</td><td>Analyst</td><td>See laboratory or other applicable limits; recoveries for the spiked compounds should be within these limits NOTE: When the full list of compounds is spiked, the laboratory will accept a small number of sporadic marginal exceedances, based on the probability that a certain number of compounds will exceed their control limits. Exceedances must be sporadic and marginal, systematic or gross failures shall not be accepted.
Laboratory limits for waters:<table><tr><td>Phenol</td><td>60-102</td></tr><tr><td>2-Chlorophenol</td><td>64-100</td></tr><tr><td>1,4-Dichlorobenzene</td><td>54-94</td></tr><tr><td>N-Nitroso-Di-N-Propylamine</td><td>62-113</td></tr></table></td></tr>
</table>

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td></td><td></td><td>
<table>
<tr><td>1,2,4-Trichlorobenzene</td><td>35-105</td></tr>
<tr><td>4-Chloro-3-Methylphenol</td><td>45-110</td></tr>
<tr><td>Acenaphthene</td><td>60-108</td></tr>
<tr><td>4-Nitrophenol</td><td>24-128</td></tr>
<tr><td>2,4-Dinitrotoluene</td><td>46-114</td></tr>
<tr><td>Pentachlorophenol</td><td>40-114</td></tr>
<tr><td>Pyrene</td><td>60-113</td></tr>
</table>
Laboratory limits for soils:
<table>
<tr><td>Phenol</td><td>39-109</td></tr>
<tr><td>2-Chlorophenol</td><td>40-107</td></tr>
<tr><td>1,4-Dichlorobenzene</td><td>35-105</td></tr>
<tr><td>N-Nitroso-Di-N-Propylamine</td><td>42-118</td></tr>
<tr><td>1,2,4-Trichlorobenzene</td><td>45-110</td></tr>
<tr><td>4-Chloro-3-methylphenol</td><td>45-115</td></tr>
<tr><td>Acenaphthene</td><td>47-110</td></tr>
<tr><td>4-Nitrophenol</td><td>24-128</td></tr>
<tr><td>2,4-Dinitrotoluene</td><td>47-117</td></tr>
<tr><td>Pentachlorophen</td><td>32-116</td></tr>
</table>
</td></tr>
</table>

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
				ol: Pyrene: 48-118
Retention Time Shift (RT)	Every sample, standard, and blank	Inspect chromatographic system for malfunction; correct identified malfunctions, then reanalyze sample.	Analyst	RT shift <30 seconds compared to daily standard Relative retention time (RRT) of sample must be ±0.06 RRT units of daily calibration check

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.
TBD = to be determined

QAPP WORKSHEET #28-3 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – PCBS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: PCBs
Analytical Method/SOP Reference[1]: SW 8082 / 409

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	1 per each preparation batch of ≤20 samples of like matrix	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise	Analyst	<LOQ: MB should not contain any target compounds at or above the LOQ or per other criteria as specified in the applicable LIMS program specification
Laboratory Control Sample (LCS)	1 per batch of ≤20 samples of like matrix	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant,	Analyst	See laboratory limits; recoveries for the spiked compounds must be within the laboratory limits or other limits as specified in the LIMS program specification. Laboratory control limits: water Arochlor-1016 68-125

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria.		Arochlor-1260 66-130 Laboratory control limits: soil Arochlor-1016 64-126 Arochlor-1260 60-130
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	1 per batch of samples, not to exceed 20 samples of a given matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	See laboratory limits; recoveries for the spiked compounds should be within advisory limits Laboratory control limits: water Arochlor-1016 68-125 Arochlor-1260 66-130 Laboratory control limits: soil Arochlor-1016 64-126 Arochlor-1260 60-130
Surrogate Spike	All extractions including field and laboratory QC samples.	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely	Analyst	See laboratory limits; recoveries should be within current limits for one or both surrogates; alternative criteria as defined in the LIMS program specifications may apply. Laboratory limits for waters:

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td>cause. However, any samples with both surrogate recoveries outside the recovery limits, with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery.

- if both surrogate recoveries in the associated MB are not within limits, , then re-extract and reanalyze all associated samples.

-</td><td></td><td><table><tr><td>Tetrachloro-M-Xylene</td><td>53-137</td></tr><tr><td>Decachlorobiphenyl</td><td>14-115</td></tr></table>
Laboratory limits for soils:
<table><tr><td>Tetrachloro-M-Xylene</td><td>61-120</td></tr><tr><td>Decachlorobiphenyl</td><td>56-130</td></tr></table></td></tr>
</table>

QAPP WORKSHEET #28-4 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – GROS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: GROs
Analytical Method/SOP Reference[1]: SW 8015B/425

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix Note: Methanol extracts additionally require a methanol MB.	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample	Analyst	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		disposition		disposition
Blank Spike (BS); Laboratory Control Sample (LCS)	1 per preparation batch of ≤20 samples of like matrix	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration	Analyst	See laboratory limits; recoveries for spiked compounds must be within laboratory limits or other limits as specified in the LIMS program specification Laboratory limits for waters: Gasoline Range Organics 79-118 Laboratory limits for soils: Gasoline Range Organics 79-118
Matrix Spike (MS	1 per preparation batch of ≤20 samples of like matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	See laboratory limits; recoveries for spiked compounds should be within advisory limits Laboratory limits for waters: Gasoline Range Organics 79-118 Laboratory limits for soils: Gasoline Range 79-118

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
				Organics
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per preparation batch of ≤20 samples of like matrix	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers. Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and	Analyst	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within advisory limits. Laboratory limits for waters: Gasoline Range Organics: 79-118 Laboratory limits for soils: Gasoline Range Organics: 79-118

<table>
<tr><th>QC Sample</th><th>Frequency / Number</th><th>Corrective Action</th><th>Person(s) Responsible for Corrective Action</th><th>Measurement Performance Criteria</th></tr>
<tr><td></td><td></td><td>the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed)</td><td></td><td></td></tr>
<tr><td>Surrogate Spike</td><td>All extractions including field and laboratory QC samples</td><td>Check calculations and spike preparation for documentable errors.
- if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery.
- if surrogate recovery in the associated MB is not within limits and the samples are within the holding time, then re-extract and reanalyze all associated samples
- if samples are beyond the holding time, then contact the PM via an NCR. Unless</td><td>Analyst</td><td>See laboratory limits; recoveries should be within current limits alternative criteria as defined in the LIMS program specifications may apply
Laboratory limits for waters:
<table><tr><td>2,3,4-Trifluorotoluene</td><td>76-126</td></tr></table>
Laboratory limits for soils:
<table><tr><td>2,3,4-Trifluorotoluene</td><td>76-126</td></tr></table></td></tr>
</table>

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.		

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.
TBD = to be determined

QAPP WORKSHEET #28-5 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – DROS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: DROs
Analytical Method/SOP Reference[1]: SW 8015/406

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix Note: Methanol extracts additionally require a methanol MB.	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition.	Analyst	<LOQ: MB should not contain any target compounds at or above the LOQ or per other criteria as specified in the applicable LIMS program specification
Blank Spike	1 per preparation	Check calculations and spike	Analyst	See laboratory limits;

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
(BS); Laboratory Control Sample (LCS)	batch of ≤20 samples of like matrix	preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed) - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration		recoveries for spiked compounds must be within laboratory limits or other limits as specified in the LIMS program specification Laboratory limits for waters: Diesel Range Organics 36-150 Laboratory limits for soils: Diesel Range Organics 87-124
Matrix Spike (MS	1 per preparation batch of ≤20 samples of like matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	See laboratory limits; recoveries for spiked compounds should be within advisory limits Laboratory limits for waters: Gasoline Range Organics 36-150 Laboratory limits for soils:

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
				Gasoline Range Organics: 87-124
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per preparation batch of ≤20 samples of like matrix	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.	Analyst	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within advisory limits. Laboratory limits for waters: Diesel Range Organics: 36-150 Laboratory limits for soils: Diesel Range Organics: 87-124
Surrogate Spike	All extractions including field and laboratory QC samples	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause.	Analyst	See laboratory limits; recoveries should be within current limits alternative criteria as defined in the LIMS program specifications may apply Laboratory limits for waters:

QC Sample	Frequency / Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		However, any samples with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery. - if surrogate recovery in the associated MB is not within limits and the samples are within the holding time, then re-extract and reanalyze all associated samples - if samples are beyond the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.		O-Terphenyl 54-123 Laboratory limits for soils: 2,3,4-Trifluorotoluene 53-116

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.
TBD = to be determined

QAPP WORKSHEET #28-6 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – METALS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: Metals
Analytical Method/SOP Reference[1]: SW 6010/807

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
MB (Method blank)	One MB per batch of 20 or fewer field samples	Correct problem then re-prep and analyze MB and all samples processed with the contaminated blank	Analyst	For non-drinking water matrix sample analyses, absolute values in MB<analyte LOQ, or as specified in applicable LIMS program specification
LCS / LCSD (Laboratory Control Sample and Laboratory Control Sample Duplicate)	One LCS per batch of 20 or fewer field samples	Correct problem then reanalyze. If still out re-prep and reanalyze the LCS and all samples in the affected batch	Analyst	For non-drinking water matrix sample analyses, analyte recoveries must be within 80-120% of expected values for each analyte.
Sample Duplicate	One sample duplicate per batch of 20 or fewer field samples	Flag results if RPD > 20%	Analyst	For each analyte RPD ≤20%, or as otherwise specified in the applicable LIMS program specification

1.

Post digestion analytical spike	Performed when MS/MSD recovery is outside ±25% (unless analyte concentration > 4X the spike level)	Flag results if post spike recovery or precision results are outside control limits	Analyst	Recovery limit 75-125% for each analyte, or as otherwise specified in the applicable LIMs program specification
Serial dilution	Performed on one sample per batch of 20 or fewer field samples	Flag results if outside criteria	Analyst	Results should agree within ±10% of undiluted results if analyte concentrations are sufficiently high (at least 5x LOQ
Internal standard responses	Monitored throughout the analytical run	Dilute samples until internal standard response is within criteria.	Analyst	For non-drinking water matrix sample analyses, response of internal standard for samples must be <70% of the original response in the calibration blank, or as specified in the applicable LIMS program specification.

QAPP WORKSHEET #28-7 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – MERCURY

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater/Soil
Analytical Group: Mercury
Analytical Method/SOP Reference[1]: SW7471A, EPA 245/812

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Blanks: Preparation (Method), Initial and Continuing Calibration Blank (ICB and CCB)	ICB run following the ICV One method blank per matrix type processed and analyzed per batch of twenty or less environmental samples processed. CCB run following the CCV to bracket and to close a run sequence.	If QC criterion not met for ICB, locate and correct problem; repeat initial calibration. If QC criterion not met for method blank, the method blank and all associated samples must be re-digested and reanalyzed. If QC criterion not met for CCB, locate and correct the problem all samples analyzed since the last acceptable CCB must be reanalyzed.	Analyst	If QC criterion not met for ICB, locate and correct problem; repeat initial calibration. If QC criterion not met for method blank, the method blank and all associated samples must be re-digested and reanalyzed. If QC criterion not met for CCB, locate and correct the problem all samples analyzed since the last acceptable CCB must be reanalyzed.
LCS / LCSD (Laboratory Control Sample and Laboratory Control Sample	One prepared and analyzed per matrix type per batch of 20 or fewer field	Check for documentable errors (e.g., calculations and spike preparation). If no computation errors are found, all associated field and quality control	Analyst	Recovery must be within ± 15% of expected value (EPA 245.1) For SW7470A recovery for aqueous LCS must

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Duplicate)	samples	samples must be re-digested and analyzed.		agree within ± 20% of expected value. For SW7471A, the recovery for the solid matrix LCS must agree ± 20% of expected value. Other client specific criteria may apply, consult applicable LIMS program specifications.
Laboratory Duplicate	One prepared and analyzed per batch of 20 or fewer field samples for SW 7470A/7471A. One prepared and analyzed per matrix type per batch of 10 or less field samples for EPA 245.1	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample heterogeneity is the most likely cause. Note in the narrative and flag results appropriately.	Analyst	RPD should be less than or equal to 20. Other client-specified criteria may apply, consult applicable LIMS program specification.
Matrix Spike (MS)	One prepared and analyzed per matrix type per batch of 20 or less field samples for SW 7471A/7470A	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the	Analyst	For SW7470A/7471A, recovery should agree ± 20% of expected value. Other client specific criteria may apply,

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		most likely cause. Note in the narrative and flag results appropriately.		consult applicable LIMS program specifications.
Matrix Spike Duplicate (MSD)	One prepared and analyzed per matrix type per batch of 20 or less field samples for SW 7471A/7470A	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in the narrative and flag results appropriately.	Analyst	For SW7470A/7471A, recovery should agree ± 20% of expected value. Other client specific criteria may apply, consult applicable LIMS program specifications.
Serial Dilution Test (1:5 dilution), analyzed to assist in the assessment of possible matrix interferences.	Prepared and analyzed per matrix batch of 20 or less field samples.	Sample analyte results failing this test should be flagged indicating the existence of matrix interferences.	Analyst	If analyte concentrations are sufficiently high (at least 50XLOQ), the results of the dilution test should agree within ±10% of the undiluted results, or as otherwise specified in the applicable LIMS program specification

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Continuing Calibration Verification (CCV); may be first or second source; run at a concentration at or below the midpoint of the calibration curve	Run to bracket a set of 10 analyses, and to end any run sequence (must be followed by a CCB analysis)	Check for calculation errors. If no calculation errors are found, analyze again. If CCV still fails, evaluate/correct instrument malfunctions; reanalyze. If CCV still fails, recalibrate system, All samples analyzed after last acceptable CCV must be reanalyzed.	Analyst	Response must agree within ± 20% of expected value (SW7470A/7471A); response must agree within ± 10% (EPA 245.1).

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-8 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – ANIONS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater
Analytical Group: Anions
Analytical Method/SOP Reference[1]: EPA 300.0 and SW9056/1113

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per each batch of <20 field samples of like matrix; one each time a reagent is changed	Prepare a fresh MB and analyze to confirm whether or not system contamination is present. If contamination in the MB is still present above the LOQ, perform system maintenance, analyze a CCV/CCB set, and re-analyze all samples associated with the failed MB that were not reportable.	Analyst	Anion content of MB must not exceed analyte LOQ.
Laboratory Control Sample (LCS)	One per batch of <20 field samples.	Check calculations and preparation for documentable errors. If no errors are found, reanalyze. Associated samples must also be reanalyzed.	Analyst	Results obtained must agree within ±10% of expected (known) analyte concentration for aqueous samples; within ±15% of known analyte concentration for solid sample extracts
Continuing Calibration	Brackets each set of 10 field	Rerun CCV. If CCV still not compliant, evaluate/correct	Analyst	Analyte concentration must agree within +5%

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Verification (CCV) at or below midpoint	sample/QC sample analyses	instrument malfunction and recalibrate. Samples analyzed after a failed CCV must be reanalyzed. If holding times are an issue, complete		Method SW9056 and within ±10 % Method 300.0; analyte retention time must agree within ±10%
Continuing Calibration Blank (CCB)	Analyzed immediately following each CCV.	Prepare a fresh CCB and analyze. If contamination in the CCB is still present above the LOQ, perform system maintenance, analyze a CCV/CCB set, and re-analyze all samples associated with the failed CCB that were not reportable.	Analyst	Anion content of CCB must not exceed analyte LOQ. Exception: Samples with analyte concentrations >10X amount found in blank may be reported and narrated.
Matrix Spike (MS) and/or Matrix Spike (Laboratory) Duplicate	One MS/MSD set per batch of 20 field samples (this provides an average frequency of one MS per ten samples per Method 300.0 requirements, and one MS/MSD per batch as required by Method SW9056 and EPA Chapter 1).	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated LCS is within control limits, then sample matrix effects are the most likely cause. Note in narrative. For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers	Analyst	RPD for the MS DUP should meet advisory limit of <20%

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-9 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – TOC

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Groundwater
Analytical Group: Total Organic Carbon
Analytical Method/SOP Reference[1]: EPA 415.1, SW9060 A, SM5310 C and SW9056/670

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One MB per every QC batch of 20 or fewer samples	Prepare another MB and analyze. If MB still fails, samples in QC batch must be reanalyzed.	Analyst	For Method 415.1 and SW9060A analyses, the MB result must not exceed LOQ (usually 1mg/L TOC)
Laboratory Control Sample (LCS), second source standard run near mid-point of calibration curve (The ICV can also serve as the LCS for the initial QC batch of samples analyzed)	One LCS in every QC batch of 20 or fewer samples	Check calculations, spike preparation, and freshness of the standard used for spiking. Prepare another LCS and analyze. If LCS still fails, samples in QC batch must be reanalyzed.	Analyst	For Method 415.1 and SW9060A analyses, the LCS result must be within ±15% of the expected concentration
Laboratory Duplicate (DUP)	For Method 415.1 and SW9060A, the LCSD & MSD both can serve as a laboratory duplicate analysis	For RPDs outside of QC limits, check all calculations for errors. Narrate.	Analyst	For both Method 415.1 and SW9060A, the RPD between the duplicate pair should be <20%

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Matrix Spike and Matrix Spike Duplicate (MS/MSD	Volume permitting, one MS/MSD pair per batch of ≤ 20 field samples	Check for documentable errors (e.g., calculations and spike preparation). For Method 415.1 and SW9060A analyses, sample matrix effects are the most likely cause if no errors are found. Document and note in case	Analyst	MS/MSD recoveries should meet advisory limits of ±20% (80-120% of the expected values) and RPD should be < 20

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-10 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – ORGANOCHLORIDE PESTICIDES

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Organochloride Pesticides
Analytical Method/SOP Reference[1]: SW 8081 A or B; and EPA 608

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at >10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact	Analyst	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		PM for sample disposition.		
Laboratory Control Sample (LCS)	One per preparatory batch.	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria. - if the samples are outside the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.	Analyst	QC acceptance criteria specified by DoD, if available. Otherwise, use in- house control limits. In -house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.
Continuing Calibration Verification	Prior to sample analysis, after every 10 field	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard	Analyst	All project analytes within established retention time windows.

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
(CCV) at or below midpoint	samples, and at the end of the analysis sequence	column of the GC, prepare a new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re- analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.		GC methods: All project analytes within ± 20% of expected value from the ICAL
Matrix Spike (MS) and/or Matrix Spike (Laboratory) Duplicate	One per preparatory batch per matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then	Analyst	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use In-house

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		sample matrix effects are the most likely cause. Note in narrative.		LCS control limits. MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate)

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-11 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – ORGANOPHOSPHOROUS COMPOUNDS

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Organophosphorous Compounds
Analytical Method/SOP Reference[1]: EPA 8141 A or B, and EPA 614

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at >10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact	Analyst	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		PM for sample disposition.		
Laboratory Control Sample (LCS)	One per preparatory batch.	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria. - if the samples are outside the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.	Analyst	QC acceptance criteria specified by DoD, if available. Otherwise, use in- house control limits. In -house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.
Continuing Calibration Verification	Prior to sample analysis, after every 10 field	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard	Analyst	All project analytes within established retention time windows.

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
(CCV) at or below midpoint	samples, and at the end of the analysis sequence	column of the GC, prepare a new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re- analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.		GC methods: All project analytes within ± 20% of expected value from the ICAL
Matrix Spike (MS) and/or Matrix Spike (Laboratory) Duplicate	One per preparatory batch per matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then	Analyst	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use In-house

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		sample matrix effects are the most likely cause. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.		LCS control limits. MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate)

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-12 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – HERBICIDES

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Herbicides
Analytical Method/SOP Reference[1]: SW8151A, EPA 615 and EPA 515.1

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at >10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact PM for sample disposition.	Analyst	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One per preparatory batch.	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria. - if the samples are outside the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.	Analyst	QC acceptance criteria specified by DoD, if available. Otherwise, use in- house control limits. In -house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.
Continuing Calibration Verification (CCV) at or below	Prior to sample analysis, after every 10 field samples, and at	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a	Analyst	All project analytes within established retention time windows. GC methods: All project

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
midpoint	the end of the analysis sequence	new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re- analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.		analytes within ± 20% of expected value from the ICAL
Matrix Spike (MS) and/or Matrix Spike (Laboratory) Duplicate	One per preparatory batch per matrix	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the	Analyst	For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use In-house LCS control limits.

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		most likely cause. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.		MSD: For matrix evaluation, use LCS acceptance criteria specified by DoD, if available. Otherwise, use in-house LCS control limits. MSD or sample duplicate: RPD ≤ 30% (between MS and MSD or sample and sample duplicate)

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-13 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – TOTAL SULFIDES

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Total Sulfides
Analytical Method/SOP Reference[1]: EPA 376.1 and SM4500 S^2 F

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch.	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	Sulfide content of any blank must not exceed the analyte reporting limit (typically 2mg/L S^2)
Laboratory Control Sample (LCS)	One per preparatory batch.	Check calculations and preparation for documentable errors. If no errors are found, reanalyze. Associated samples must also be reanalyzed.	Analyst	Concentration results obtained must agree between 80% and 120% of expected value
Laboratory Duplicate (DUP)	One per batch of ≤20 samples	Check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers.	Analyst	RPD must be <20%

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-14 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – NITROGEN AS NITRATE PLUS NITRITE

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Nitrogen as Nitrate plus Nitrite
Analytical Method/SOP Reference[1]: EPA 353.2, SM 4500

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	The MB may be run initially as part of the calibration curve, the ICB is run following the calibration curve. CCBs are run following the CCV (after every ten samples), and to close an analytical run sequence	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	Nitrogen as Nitrate plus Nitrite content of the blank must be <LOQ
Laboratory Control Sample (LCS)	One LCS must be run per 20 environmental samples of similar matrix	Check all calculations. If no computation errors are found, prepare a fresh LCS and analyze. If criteria are still not met, system must be recalibrated and all samples run since the last acceptable LCS	Analyst	Results must agree within ±10% of expected values for aqueous sample analyses; within ±15% for solid matrix extract analyses

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		must be reanalyzed.		
Continuing Calibration Verification (CCV); at or below midpoint; first source	Run after every ten samples and to end any run sequence (must be followed by a CCB analysis)	Check that calculations and preparation are correct, evaluate/ correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.	Analyst	Response must agree within ±10% of initial calibration
Matrix Spike (MS)	One per batch of <20 field samples of similar matrix	Compare with LCS results and check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	Recoveries should meet advisory limits of ±25% of the expected values; client-specified criteria may apply
Matrix Spike (laboratory) Duplicate	One per batch of < 20 field samples of similar matrix	For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers.	Analyst	RPD advisory limit is <20; client-specified criteria may apply

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-15 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – OIL AND GREASE

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Oil and Grease
Analytical Method/SOP Reference[1]: EPA 1664 A, and SW9070A

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per each batch of ≤20 field samples; one each time a reagent is changed	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	MB must not yield HEM content above the 5.0 mg/L LOQ
Laboratory Control Sample (LCS)	One per batch of <20 field samples	Check calculations and preparation for documentable errors. If no errors are found, reanalyze OPR, associated samples must also be re-extracted and re-analyzed (if possible). If samples cannot be re-extracted, narrate.	Analyst	Results obtained must be within 79-114% of expected (known) concentration of HEM, and 64-132 % for SGT-HEM
Matrix Spike (MS)	One per batch of <20 field samples	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated OPR is within control limits, then sample matrix effects are the most likely cause. Note in	Analyst	Results obtained must be within 79-114% of expected concentration of HEM, and 64-132 % for SGT-HEM

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
		narrative.		
Matrix Spike (laboratory) Duplicate	One per batch of <20 field samples	For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers.	Analyst	HEM RPD should be <18%; SGT-HEM RPD <34%

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-16 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – HEXAVALENT CHROMIUM

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Hexavalent Chromium
Analytical Method/SOP Reference[1]: SW3060A and 7196A

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Continuing calibration verification (CCV)	Run after every ten samples and to end any run sequence (must be followed by a CCB analysis)	Check that calculations and preparation are correct, evaluate/correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.	Analyst	Value of CCV within ± 10% of true value.
Method Blank (MB)	One per each batch of ≤20 field samples	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results
Laboratory	One per batch of	Check for documentable errors	Analyst	QC acceptance criteria

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Control Sample (LCS)	<20 field samples	(e.g., calculations and spike preparation). If no computation errors are found, prepare a fresh spike and analyze. If quality criteria still not met, all field and quality control samples must be re-prepared and analyzed.		specified by DoD
Matrix Spike (MS)	One per batch of <20 field samples	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	Spike recovery within 85-115%.
Matrix Spike (laboratory) Duplicate	Matrix-specific; one prepared for each sample batch of <20 field samples	For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers.	Analyst	RPD ≤ 20% (between MS and MSD or sample and sample duplicate).

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #28-17 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – TOTAL CYANIDE

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)

Matrix: Surface Water
Analytical Group: Total Cyanide
Analytical Method/SOP Reference[1]: SW9010C, SW9013, EPA 335.1, EPA 335.2, CLP INORGANIC SOW (ILM04.0); SM4500

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
Continuing calibration verification (CCV)	Run to bracket a set of 10 analyses, and to end any run sequence (must be followed by a CCB analysis)	Check that calculations and preparation are correct, evaluate/correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.	Analyst	Value of CCV within ± 15% of true value.
Method Blank (MB)	One per each batch of ≤20 field samples	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.	Analyst	No analyte detected > 1/2 the reporting limit and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no

QC Sample	Frequency/ Number	Corrective Action	Person(s) Responsible for Corrective Action	Measurement Performance Criteria
				analytes detected > LOQ
Distilled Laboratory Control Sample (LCS)	One low and one high prepared and analyzed per batch of ≤20 field samples	Check for documentable errors (e.g., calculations and preparation). If no errors are found, the distillation of these standards and all associated samples in the batch must be repeated.	Analyst	QC acceptance criteria specified by DoD
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per batch of <20 field samples	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.	Analyst	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.

1. SOPs are reviewed/ revised on an annual schedule. The current version will be followed at the time of sample receipt.

TBD = to be determined

QAPP WORKSHEET #29 - PROJECT DOCUMENTS AND RECORDS

(UFP-QAPP Manual Section 3.5.1)

Documents and Records	On-Site Analysis Documents and Records	Generation	Verification	Storage Location/Archival
Sample Collection				
Field data collection sheets	Equipment calibration logs	Field Team Lead	Project Manager	Project File
Chain-of-Custody (C-o-C) Records	Equipment maintenance	Field Team Lead	Project Manager	Project File
Air Bills	Testing and Inspection logs	Field Team Lead	Project Manager	Project File
Custody Seals	Corrective Action Forms	Field Team Lead	Project Manager	
Telephone Logs	Telephone Logs	Field Team Lead	Project Manager	Project File
Corrective Action Forms	Daily Quality Control Reports (DQCRs)	Field Team Lead	Project Manager	Project File
Documentation of deviations from methods/SOPs	Test kit analytical data forms	Field Team Lead	Project Manager	Project File
Identification of QC samples	Soil boring logs	Field Team Lead	Project Manager	Project File
Photographs	Well/soil boring construction diagrams	Field Team Lead		Project File
Sampling instrument calibration logs	Well purging/ water quality forms	Field Team Lead	Project Manager	Project File
Sampling locations and sampling plan	PID monitoring forms	Field Team Lead	Project Manager	Project File
Sampling notes and drilling logs		Project Geologist	Project Manager	Project File

Documents and Records	On-Site Analysis Documents and Records	Generation	Verification	Storage Location/Archival
Project Assessments				
Equipment maintenance, testing, and inspection logs		Field Team Lead	Project Manager	Project File
Corrective Action Forms		Field Team Lead	Project Manager	Project File
Reported results for QC checks, instrument tunes, and QC samples		Field Team Lead, Laboratory	Project Manager	Project File
Data Validation Report		LDC	Project Manager	Project File
Laboratory Reports				
Analytical Report		Laboratory Project Manager	Project Chemists	Project File

QAPP WORKSHEETS #31, 32, & 33: ASSESSMENTS AND CORRECTIVE ACTION

(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Chemistry Field Sampling Technical Systems Audit	Project Chemist TI2E	At start of sampling and regularly thereafter	5 days after audit	Written Field Audit	10 days after receiving notification
Fixed Laboratory Technical Systems Audit	Project Chemist TI2E	Prior to sampling. As needed afterwards.	5 days after audit	Written Audit Report	14 days after receiving notification
DoD Environmental Laboratory Accreditation Program	Validation Coordinator DoD Laboratory	Every 2 years	30 days after audit	Written Audit Report	30 days after receiving notification
Chemistry Data Audit	Project Chemist TI2E	Periodically	5 days after audit	Written Audit Report	14 days after receiving notification
Data Validation System Audit	Project Manager TI2E	Periodically	5 days after audit	Written Audit Report	14 days after receiving notification

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Chemistry Field Sampling Technical Systems Audit	Field Team Lead TI2E	Corrective Action Plan	10 days after receiving notification	Field Team Lead TI2E	Project Chemist TI2E
Fixed Laboratory Technical Systems Audit	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan	30 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	Project Chemist TI2E
DoD Environmental Laboratory Accreditation Program	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan	30 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	DoD ELAP Coordinator
Chemistry Data Audit	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan, Re-submission of data	30 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	Project Chemist TI2E
Data Validation System Audit	QA Coordinator or Technical Operations Manager Contract Laboratory	Corrective Action Plan, Re-submission of data	30 days after receiving notification	QA Coordinator or Technical Operations Manager Contract Laboratory	Project Manager TI2E

Management Reports:

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)
Verbal Status Reports	Daily/Weekly	Daily/weekly conference calls	TI2E field personnel, technical team members	TI2E field personnel, technical team members
Technical Meetings or Teleconferences	Monthly during Field Work, or as needed	Monthly during Field Work, or as needed	Contractor and regulatory attendees. Meeting minutes prepared by TI2E and sent to project members via email.	Contractor and regulatory agency team members
Email status Reports during Field Work	Weekly	Weekly	Project Manager, TI2E	USACE, and NYSDEC
DQCR	Daily	Ongoing	Field Team Lead TI2E	TI2E PM, USACE PM
Data Validation Reports	With each analytical batch reported by the laboratory	Ongoing	Project Chemist, TI2E	TI2E PM, USACE PM
Automated Data Validation	With each analytical batch reported by the laboratory	Ongoing	Project Chemist, TI2E	TI2E PM, USACE PM

QAPP WORKSHEET #34 – DATA VERIFICATION AND VALIDATION INPUTS

(UFP-QAPP Manual Section 5.2.1 and Table 9)

Item	Description	Verification (completeness)	Validation (conformance to specifications)
	Planning Documents/Records		
1	Approved QAPP	X	
2	Approved Accident Prevention Plan/ Site Safety and Health Plan (APP/SSHP)	X	
3	Field SOPs	X	
4	Laboratory SOPs	X	
	Field Records		
5	Field Logbooks	X	X
6	Equipment Calibration Logs	X	X
7	Chain-of-Custody records	X	X
8	Sampling diagrams/notes	X	X
9	Drilling logs	X	X
10	Monitoring well construction forms	X	X
11	Groundwater sampling forms	X	X
12	DQCRs	X	X
	Analytical Data Package		
13	Cover Sheet	X	X
14	Case narrative	X	X
15	Internal laboratory Chain-of-custody	X	X
16	Sample Receipt records	X	X
17	Sample chronology	X	X
18	Sample results	X	X
19	LOD/LOQ Establishment and verification	X	X

Item	Description	Verification (completeness)	Validation (conformance to specifications)
20	Standards traceability	X	X
21	Instrument calibration records	X	X
22	Definition of laboratory qualifiers	X	X
23	QC sample results	X	X
24	Corrective Action reports	X	X
25	Raw data	X	X
26	Electronic data deliverable	X	X
27	Data Validation Report	X	X

QAPP WORKSHEET #35 – DATA VERIFICATION PROCEDURES

(UFP-QAPP Manual Section 5.2.2)

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
Field Logbook	QAPP, Field SOPs	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed and results are documented.	Daily – Field Team Lead At conclusion of field activities - Project Manager
Chain-of-custody forms	QAPP, Field SOPs	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily - Field Team Lead At conclusion of field activities - Project Chemist
Laboratory Deliverable	QAPP, Lab SOPs	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the chain-of-custody forms to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary	Before release - Laboratory Project Manager Upon receipt - Project Chemist

Records Reviewed	Required Documents	Process Description	Responsible Person, Organization
		signatures and dates are present.	
Data Validation Reports	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that corrective action was implemented according to plan. Establish that all QAPP required QC samples were run and met the required limits for precision and accuracy. Verify that sample results met the project quantitation limits specified in the QAPP. Conduct 10% review of raw data to confirm laboratory calculations	LDC – Project Chemist

QAPP WORKSHEET #36 – DATA VALIDATION PROCEDURES

(UFP-QAPP Manual Section 5.2.2)

All laboratory data will undergo data validation by LDC, utilizing the ADR.net software. For data validation elements not provided in the laboratory EDD, LDC will manually validate. These elements are, but not limited to, instrument tuning and calibration, initial and continuing calibration blanks, and internal standards. All validation criteria will be in accordance with DoD QSM v5.0, CLPNFGs (2008 and 2010), EPA SW846 Methods (listed below), and Laboratory SOPs (listed below).

Analytical Group	Data Deliverable requirements	Analytical Specifications	Measurement Performance Criteria	% of data packages to be validated	% of raw data received	% of results to be calculated	Validation Procedure	Validation Code
VOCs SW-846 8260B	SEDD Stage 4	ALS SOP 525 WS #28-1	WS #12	100%	100%	100%	Level 4	S4VEM
SVOCs SW-846 8270D	SEDD Stage 4	ALS SOP 506	WS #12	100%	100%	100%	Level 4	S4VEM
PCBs SW-846 8082	SEDD Stage 4	ALS SOP 409	WS #12	100%	100%	100%	Level 4	S4VEM
Metals SW-846 6020A	SEDD Stage 4	ALS SOPs 807/827	WS #12	100%	100%	100%	Level 4	S4VEM
Mercury SW-846 7470A	SEDD Stage 4	ALS SOP 812	WS #12	100%	100%	100%	Level 4	S4VEM
Organochloride Pesticides SW-846 SW8081	SEDD Stage 4	ALS SOP 402	WS #12	100%	100%	100%	Level 4	S4VEM
Organophosphorus Compounds EPA 8141	SEDD Stage 4	ALS SOP 407	WS #12	100%	100%	100%	Level 4	S4VEM

Analytical Group	Data Deliverable requirements	Analytical Specifications	Measurement Performance Criteria	% of data packages to be validated	% of raw data received	% of results to be calculated	Validation Procedure	Validation Code
Herbicides EPA 8151	SEDD Stage 4	ALS SOP 434	WS #12	100%	100%	100%	Level 4	S4VEM
Anions SW-846 9056	SEDD Stage 4	ALS SOP 1113	WS #12	100%	100%	100%	Level 4	S4VEM
TOC 9060A	SEDD Stage 4	ALS SOP 670	WS #12	100%	100%	100%	Level 4	S4VEM
TPH-GRO SW-846 8015B	SEDD Stage 4	ALS SOP 425	WS #12	100%	100%	100%	Level 4	S4VEM
TPH-DRO SW-846 8015MOD	SEDD Stage 4	ALS SOP 406	WS #12	100%	100%	100%	Level 4	S4VEM
Nitrogen as Nitrate and Nitrite EPA 353.2	SEDD Stage 4	ALS SOP 1112	WS #12	100%	100%	100%	Level 4	S4VEM
Total sulfides EPA376.1 & SM4500	SEDD Stage 4	ALS SOP 1120	WS #12	100%	100%	100%	Level 4	S4VEM
Oil and Grease SW9070 EPA 1664A	SEDD Stage 4	ALS SOP 671	WS #12	100%	100%	100%	Level 4	S4VEM
Hexavalent Chromium SW3060A & 7196A	SEDD Stage 4	ALS SOP 1121	WS #12	100%	100%	100%	Level 4	S4VEM
Total Cyanide SW9010C, SW9013, EPA335.1, 335.2,	SEDD Stage 4	ALS SOP 1110	WS #12	100%	100%	100%	Level 4	S4VEM

Analytical Group	Data Deliverable requirements	Analytical Specifications	Measurement Performance Criteria	% of data packages to be validated	% of raw data received	% of results to be calculated	Validation Procedure	Validation Code
SM4500								

ALS SOPs are provided in Attachment D

Validation Code and Label Identifier Table (Reference: EPA 540-R-08-005)

Validation Code	Validation Label
S1VE	Stage 1 Validation Electronic
S1VM	Stage 1 Validation Manual
S1VEM	Stage 1 Validation Electronic and Manual
S2aVE	Stage 2a Validation Electronic
S2aVM	Stage 2a Validation Manual
S2aVEM	Stage 2a Validation Electronic and Manual
S2bVE	Stage 2b Validation Electronic
S2bVM	Stage 2b Validation Manual
S2bVEM	Stage 2b Validation Electronic and Manual
S3VE	Stage 3 Validation Electronic
S3VM	Stage 3 Validation Manual
S3VEM	Stage 3 Validation Electronic and Manual
S4VE	Stage 4 Validation Electronic
S4VM	Stage 4 Validation Manual
S4VEM	Stage 4 Validation Electronic and Manual

QAPP WORKSHEET #37 – DATA USABILITY ASSESSMENT

(UFP-QAPP Manual Section 5.2.3)

The Data Usability Assessment will be performed by the Project Chemist, of TI2E. After the Data Usability Assessment has been performed, data deemed appropriate for use will then be used for its various purposes, i.e., to evaluate baseline and periodic groundwater conditions at the site, determine appropriate injection point locations, and define the extent of potential contamination. Data Usability Assessments will be presented in the Remedial Investigation Report. The following items will be assessed and conclusions drawn based on their results:

Precision – Precision is measured through analysis of field and laboratory QC samples, namely field duplicates and MSDs. Field sampling precision is evaluated by calculating the RPD for target compounds detected in the parent sample and it's duplicate. Target compounds detected in both the parent sample and its field duplicate equal to or above the quantitation limit will be presented in tabular format and the RPD calculated. The RPDs will be checked against the measurement performance criteria presented on Worksheet #12, and RPDs exceeding criteria will be identified on the tables.

Laboratory precision is evaluated through calculation of RPDs for all target compounds spiked into a field-designated MS and MSD sample. Any outliers identified (i.e., those compounds with RPDs outside control limits) will be reviewed to determine the source of the error. Sources of error include matrix interference, instrument error, and/or sampling error. Any conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias Contamination – Results for all laboratory method blanks, equipment blanks, and trip blanks will be evaluated. The results for of any target compound detected will be checked against the measurement performance criteria presented on Worksheet #12. Results for target compounds that exceed criteria will be tabulated. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described.

Analytical Accuracy/Bias – Analytical accuracy and bias will be evaluated through matrix spikes, surrogate recoveries, laboratory control samples (LCSs), and second source calibration verification. Outliers will be evaluated and tabulated, using acceptance criteria presented on Worksheet #12. A discussion will follow summarizing analytical accuracy/bias and whether the source of the error is instrument, matrix, or sampling related. Conclusions about the analytical accuracy/bias of the analyses will be drawn, and any limitations on the use of the data will be described.

Sensitivity – The results for target compounds will be checked against the measurement performance criteria presented on Worksheet #12 and cross-checked against the quantitation limits presented on Worksheet #15. Results for target compounds with elevated quantitation limits will be identified and noted. A discussion will follow summarizing the results of the laboratory sensitivity. Any conclusions about the sensitivity of the analyses will be drawn and any limitations on the use of the data will be described.

Representativeness – The soil and groundwater samples required for this project will be collected using standardized procedures and at carefully selected locations. The sampling design includes the collection of 42 "representative" soil samples from 14 randomly located borings in addition to "biased" samples collected at specific locations to fill in data gaps. From each "representative" soil boring, soil will be collected from three sampling units (surface from 0-1 ft bgs, fill/soil contact at 2-8 ft bgs, and soil/bedrock contact from 8-15 ft bgs). Standardized, accepted analytical methods will be used to ensure that accurate, reproducible data are generated. To verify sample representativeness, field sample collection procedures, sample containers, and holding times will be reviewed for SOP conformance. Non-representative samples will be identified, narrated, and the impact on data quality objectives discussed.

Comparability – The site-wide groundwater data set will be comparable to historical groundwater data through use of the same sampling points (monitoring wells), and sample collection, and analytical SOPs. Any deviations will be noted, and conclusions drawn on the usability of the data.

Completeness – A completeness check will be done on all of the data generated by the laboratory. Completeness criteria are presented on Worksheet #12. Completeness will be calculated for each target compound for the site DU as follows. For each target compound, completeness will be calculated as the number of data points for each compound that meets the measurement performance criteria for precision, accuracy/bias, and sensitivity, divided by the total number of data points for each compound. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each analyte will be drawn and any limitations on the use of the data will be described.

Reconciliation – Each of the Project Quality Objectives (PQOs) presented on Worksheet #12 will be examined to determine if the objective was met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will first be evaluated separately in terms of the major impacts observed from the Data Validation, Data Quality Indicators, and measurement performance criteria assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality determined, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined if the PQO was met and whether project screening levels were exceeded. The final report will include a summary of all the points

that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described.

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ATTACHMENT A

EPA Regional Screening Levels (RSLs) (June 2015), Maximum Concentration Levels (MCLs) (May 2009) and NYSDEC Soil Cleanup Objectives (September 2006)

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels										Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
1.8E-02	C	5.1E-06	C	1.5E-01	I					1	0.1		ALAR	1596-84-5	3.0E+01	c	1.3E+02	c	5.5E-01	c	2.4E+00	c	4.3E+00	c		9.5E-04	c	
8.7E-03	I			4.0E-03	I					1	0.1		Acephate	30560-19-1	6.2E+01	c**	2.6E+02	c*					8.9E+00	c**		2.0E-03	c**	
		2.2E-06	I			9.0E-03	I	V		1		1.1E+05	Acetaldehyde	75-07-0	1.1E+01	c**	4.9E+01	c**	1.3E+00	c**	5.6E+00	c**	2.6E+00	c**		5.2E-04	c**	
				2.0E-02	I					1	0.1		Acetochlor	34256-82-1	1.3E+03	n	1.6E+04	n					3.5E+02	n		2.8E-01	n	
				9.0E-01	I	3.1E+01	A	V		1		1.1E+05	Acetone	67-64-1	6.1E+04	n	6.7E+05	nms	3.2E+04	n	1.4E+05	n	1.4E+04	n		2.9E+00	n	
						2.0E-03	X	V		1		1.1E+05	Acetone Cyanohydrin	75-86-5	5.0E+01	n	2.1E+02	n	2.1E+00	n	8.8E+00	n	4.2E+00	n		8.4E-04	n	
						6.0E-02	I	V		1		1.3E+05	Acetonitrile	75-05-8	8.1E+02	n	3.4E+03	n	6.3E+01	n	2.6E+02	n	1.3E+02	n		2.6E-02	n	
				1.0E-01	I			V		1		2.5E+03	Acetophenone	98-86-2	7.8E+03	ns	1.2E+05	nms					1.9E+03	n		5.8E-01	n	
3.8E+00	C	1.3E-03	C							1	0.1		Acetylaminofluorene, 2-	53-96-3	1.4E-01	c	6.0E-01	c	2.2E-03	c	9.4E-03	c	1.6E-02	c		7.2E-05	c	
				5.0E-04	I	2.0E-05	I	V		1		2.3E+04	Acrolein	107-02-8	1.4E-01	n	6.0E-01	n	2.1E-02	n	8.8E-02	n	4.2E-02	n		8.4E-06	n	
5.0E-01	I	1.0E-04	I	2.0E-03	I	6.0E-03	I		M	1	0.1		Acrylamide	79-06-1	2.4E-01	c	4.6E+00	c	1.0E-02	c	1.2E-01	c	5.0E-02	c		1.1E-05	c	
				5.0E-01	I	1.0E-03	I	V		1		1.1E+05	Acrylic Acid	79-10-7	9.9E+01	n	4.2E+02	n	1.0E+00	n	4.4E+00	n	2.1E+00	n		4.2E-04	n	
5.4E-01	I	6.8E-05	I	4.0E-02	A	2.0E-03	I	V		1		1.1E+04	Acrylonitrile	107-13-1	2.5E-01	c*	1.1E+00	c*	4.1E-02	c*	1.8E-01	c*	5.2E-02	c*		1.1E-05	c*	
						6.0E-03	P			1	0.1		Adiponitrile	111-69-3	8.5E+06	nm	3.6E+07	nm	6.3E+00	n	2.6E+01	n						
5.6E-02	C			1.0E-02	I					1	0.1		Alachlor	15972-60-8	9.7E+00	c*	4.1E+01	c					1.0E+00	c	2.0E+00	8.6E-04	c	1.6E-03
				1.0E-03	I					1	0.1		Aldicarb	116-06-3	6.3E+01	n	8.2E+02	n					2.0E+01	n	3.0E+00	4.9E-03	n	7.5E-04
				1.0E-03	I					1	0.1		Aldicarb Sulfone	1646-88-4	6.3E+01	n	8.2E+02	n					2.0E+01	n	2.0E+00	4.4E-03	n	4.4E-04
										1	0.1		Aldicarb sulfoxide	1646-87-3											4.0E+00			8.8E-04
1.7E+01	I	4.9E-03	I	3.0E-05	I			V		1			Aldrin	309-00-2	3.9E-02	c*	1.8E-01	c	5.7E-04	c	2.5E-03	c	9.2E-04	c		1.5E-04	c	
				2.5E-01	I					1	0.1		Ally	74223-64-6	1.6E+04	n	2.1E+05	nm					4.9E+03	n		1.9E+00	n	
				5.0E-03	I	1.0E-04	X	V		1		1.1E+05	Allyl Alcohol	107-18-6	3.5E+00	n	1.5E+01	n	1.0E-01	n	4.4E-01	n	2.1E-01	n		4.2E-05	n	
2.1E-02	C	6.0E-06	C			1.0E-03	I	V		1		1.4E+03	Allyl Chloride	107-05-1	7.2E-01	c**	3.2E+00	c**	4.7E-01	c**	2.0E+00	c**	7.3E-01	c**		2.3E-04	c**	
				1.0E+00	P	5.0E-03	P			1			Aluminum	7429-90-5	7.7E+04	n	1.1E+06	nm	5.2E+00	n	2.2E+01	n	2.0E+04	n		3.0E+04	n	
				4.0E-04	I					1			Aluminum Phosphide	20859-73-8	3.1E+01	n	4.7E+02	n					8.0E+00	n			n	
				3.0E-04	I					1	0.1		Amdro	67485-29-4	1.9E+01	n	2.5E+02	n					5.9E+00	n		2.1E+03	n	
				9.0E-03	I					1	0.1		Ametryn	834-12-8	5.7E+02	n	7.4E+03	n					1.5E+02	n		1.6E-01	n	
2.1E+01	C	6.0E-03	C							1	0.1		Aminobiphenyl, 4-	92-67-1	2.6E-02	c	1.1E-01	c	4.7E-04	c	2.0E-03	c	3.0E-03	c		1.5E-05	c	
				8.0E-02	P					1	0.1		Aminophenol, m-	591-27-5	5.1E+03	n	6.6E+04	n					1.6E+03	n		6.1E-01	n	
				2.0E-02	P					1	0.1		Aminophenol, p-	123-30-8	1.3E+03	n	1.6E+04	n					4.0E+02	n		1.5E-01	n	
				2.5E-03	I					1	0.1		Amtraz	33089-61-1	1.6E+02	n	2.1E+03	n					8.2E+00	n		4.2E+00	n	
						1.0E-01	I	V		1			Ammonia	7664-41-7					1.0E+02	n	4.4E+02	n						
				2.0E-01	I					1			Ammonium Sulfamate	7773-06-0	1.6E+04	n	2.3E+05	nm					4.0E+03	n			n	
						3.0E-03	X	V		1		1.4E+04	Amyl Alcohol, tert-	75-85-4	8.2E+01	n	3.4E+02	n	3.1E+00	n	1.3E+01	n	6.3E+00	n		1.3E-03	n	
5.7E-03	I	1.6E-06	C	7.0E-03	P	1.0E-03	I			1	0.1		Aniline	62-53-3	9.5E+01	c**	4.0E+02	c*	1.0E+00	n	4.4E+00	n	1.3E+01	c*		4.6E-03	c*	
4.0E-02	P			2.0E-03	X					1	0.1		Anthraquinone, 9,10-	84-65-1	1.4E+01	c**	5.7E+01	c*					1.4E+00	c*		1.4E-02	c*	
				4.0E-04	I					0.15			Antimony (metallic)	7440-36-0	3.1E+01	n	4.7E+02	n					7.8E+00	n	6.0E+00	3.5E-01	n	2.7E-01
				5.0E-04	H					0.15			Antimony Pentoxide	1314-60-9	3.9E+01	n	5.8E+02	n					9.7E+00	n			n	
				9.0E-04	H					0.15			Antimony Potassium Tartrate	11071-15-1	7.0E+01	n	1.1E+03	n					1.8E+01	n			n	
				4.0E-04	H					0.15			Antimony Tetroxide	1332-81-6	3.1E+01	n	4.7E+02	n					7.8E+00	n			n	
						2.0E-04	I			0.15			Antimony Trioxide	1309-64-4	2.8E+05	nm	1.2E+06	nm	2.1E-01	n	8.8E-01	n						
				1.3E-02	I					1	0.1		Apollo	74115-24-5	8.2E+02	n	1.1E+04	n					2.3E+02	n		1.4E+01	n	
2.5E-02	I	7.1E-06	I	5.0E-02	H					1	0.1		Aramite	140-57-8	2.2E+01	c	9.2E+01	c	4.0E-01	c	1.7E+00	c	1.3E+00	c		1.5E-02	c	
1.5E+00	I	4.3E-03	I	3.0E-04	I	1.5E-05	C			1	0.03		Arsenic, Inorganic	7440-38-2	6.8E-01	c*R	3.0E+00	cR	6.5E-04	c*	2.9E-03	c*	5.2E-02	c	1.0E+01	1.5E-03	c	2.9E-01
				3.5E-06	C	5.0E-05	I			1			Arsine	7784-42-1	2.7E-01	n	4.1E+00	n	5.2E-02	n	2.2E-01	n	7.0E-02	n			n	
				9.0E-03	I					1	0.1		Assure	76578-14-8	5.7E+02	n	7.4E+03	n					1.2E+02	n		1.9E+00	n	
				5.0E-02	I					1	0.1		Asulam	3337-71-1	3.2E+03	n	4.1E+04	n					1.0E+03	n		2.6E-01	n	
2.3E-01	C			3.5E-02	I					1	0.1		Atrazine	1912-24-9	2.4E+00	c	1.0E+01	c					3.0E-01	c	3.0E+00	1.9E-04	c	1.9E-03
8.8E-01	C	2.5E-04	C							1	0.1		Auramine	492-80-8	6.2E-01	c	2.6E+00	c	1.1E-02	c	4.9E-02	c	6.6E-02	c		6.0E-04	c	
				4.0E-04	I					1	0.1		Avermectin B1	65195-55-3	2.5E+01	n	3.3E+02	n					8.0E+00	n		1.4E+01	n	
1.1E-01	I	3.1E-05	I					V		1			Azobenzene	103-33-3	5.6E+00	c	2.6E+01	c	9.1E-02	c	4.0E-01	c	1.2E-01	c		9.2E-04	c	
				1.0E+00	P	7.0E-06	P			1	0.1		Azodicarbonamide	123-77-3	8.6E+03	n	4.0E+04	n	7.3E-03	n	3.1E-02	n	2.0E+04	n		6.8E+00	n	
				2.0E-01	I	5.0E-04	H			0.07			Barium	7440-39-3	1.5E+04	n	2.2E+05	nm	5.2E-01	n	2.2E+00	n	3.8E+03	n	2.0E+03	1.6E+02	n	8.2E+01
5.0E-01	C	1.5E-01	C	2.0E-02	C	2.0E-04	C		M	0.025			Barium Chromate	10294-40-3	3.0E-01	c	6.2E+00	c	6.8E-06	c	8.2E-05	c	4.1E-02	c			c	
				4.0E-03	I					1	0.1		Baygon	114-26-1	2.5E+02	n	3.3E+03	n					7.8E+01	n		2.5E-02	n	
				3.0E-02	I					1	0.1		Bayleton	43121-43-3	1.9E+03	n	2.5E+04	n					5.5E+02	n		4.4E-01	n	
				2.5E-02	I					1	0.1		Baythroid	68359-37-5	1.6E+03	n	2.1E+04	n					1.2E+02	n		3.1E+01	n	
				3.0E-01	I			V		1			Benefin	1861-40-1	2.3E+04	n	3.5E+05	nm					1.7E+03	n		5.6E+01	n	
				5.0E-02	I					1	0.1		Benomyl	17804-35-2	3.2E+03	n	4.1E+04	n					9.7E+02	n		8.5E-01	n	
				3.0E-02	I					1	0.1		Bentazon	25057-89-0	1.9E+03	n	2.5E+04	n					5.7E+02	n		1.2E-01	n	
				1.0E-01	I			V		1		1.2E+03	Benzaldehyde	100-52-7	7.8E+03	ns	1.2E+05	nms					1.9E+03	n		4.3E-01	n	
5.5E-02	I	7.8E-06	I	4.0E-03	I	3.0E-02	I	V		1		1.8E+03	Benzene	71-43-2	1.2E+00	c*	5.1E+00	c*	3.6E-01	c*	1.6E+00	c*	4.5E-01	c*	5.0E+00	2.3E-04	c*	2.6E-03
1.0E-01	X			3.0E-04	X					1	0.1		Benzenediamine-2-methyl sulfate, 1,4-	6369-59-1	5.4E+00	c**	2.3E+01	c*					7.8E-01	c**		2.2E-04	c**	
				1.0E-03	P			V		1		1.3E+03	Benzenethiol	108-98-5	7.8E+01	n	1.2E+03	n					1.7E+01	n		1.1E-02	n	
2.3E+02	I	6.7E-02	I	3.0E-03	I				M	1	0.1		Benzidine	92-87-5	5.3E-04	c	1.0E-02	c	1.5E-05	c	1.8E-04	c	1.1E-04	c		2.7E-07	c	
				4.0E+00	I					1	0.1		Benzoic Acid	65-85-0	2.5E+05	nm	3.3E+06	nm					7.5E+04	n		1.8E+01	n	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information													Contaminant		Screening Levels										Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
1.3E+01	I							V		1		3.2E+02	Benzotrichloride	98-07-7	5.3E-02	c	2.5E-01	c					2.9E-03	c		6.5E-06	c	
				1.0E-01	P					1	0.1		Benzyl Alcohol	100-51-6	6.3E+03	n	8.2E+04	n					2.0E+03	n		4.8E-01	n	
1.7E-01	I	4.9E-05	C	2.0E-03	P	1.0E-03	P	V		1		1.5E+03	Benzyl Chloride	100-44-7	1.1E+00	c*	4.8E+00	c*	5.7E-02	c*	2.5E-01	c*	8.9E-02	c*		9.7E-05	c*	
		2.4E-03	I	2.0E-03	I	2.0E-05	I			0.007			Beryllium and compounds	7440-41-7	1.6E+02	n	2.3E+03	n	1.2E-03	c*	5.1E-03	c*	2.5E+01	n	4.0E+00	1.9E+01	n	3.2E+00
				1.0E-04	I					1	0.1		Bidrin	141-66-2	6.3E+00	n	8.2E+01	n					2.0E+00	n		4.7E-04	n	
				9.0E-03	P					1	0.1		Bifenox	42576-02-3	5.7E+02	n	7.4E+03	n					1.0E+02	n		7.6E-01	n	
				1.5E-02	I					1	0.1		Biphenthrin	82657-04-3	9.5E+02	n	1.2E+04	n					3.0E+02	n		1.4E+03	n	
8.0E-03	I			5.0E-01	I	4.0E-04	X	V		1			Biphenyl, 1,1'-	92-52-4	4.7E+01	n	2.0E+02	n	4.2E-01	n	1.8E+00	n	8.3E-01	n		8.7E-03	n	
7.0E-02	H	1.0E-05	H	4.0E-02	I			V		1		1.0E+03	Bis(2-chloro-1-methylethyl) ether	108-60-1	4.9E+00	c	2.2E+01	c	2.8E-01	c	1.2E+00	c	3.6E-01	c		1.3E-04	c	
				3.0E-03	P					1	0.1		Bis(2-chloroethoxy)methane	111-91-1	1.9E+02	n	2.5E+03	n					5.9E+01	n		1.3E-02	n	
1.1E+00	I	3.3E-04	I					V		1		5.1E+03	Bis(2-chloroethyl)ether	111-44-4	2.3E-01	c	1.0E+00	c	8.5E-03	c	3.7E-02	c	1.4E-02	c		3.6E-06	c	
2.2E+02	I	6.2E-02	I					V		1		4.2E+03	Bis(chloromethyl)ether	542-88-1	8.3E-05	c	3.6E-04	c	4.5E-05	c	2.0E-04	c	7.2E-05	c		1.7E-08	c	
				5.0E-02	I					1	0.1		Bisphenol A	80-05-7	3.2E+03	n	4.1E+04	n					7.7E+02	n		5.8E+01	n	
				2.0E-01	I	2.0E-02	H			1			Boron And Borates Only	7440-42-8	1.6E+04	n	2.3E+05	nm	2.1E+01	n	8.8E+01	n	4.0E+03	n		1.3E+01	n	
				2.0E+00	P	2.0E-02	P	V		1			Boron Trichloride	10294-34-5	1.6E+05	nm	2.3E+06	nm	2.1E+01	n	8.8E+01	n	4.2E+01	n			n	
				4.0E-02	C	1.3E-02	C	V		1			Boron Trifluoride	7637-07-2	3.1E+03	n	4.7E+04	n	1.4E+01	n	5.7E+01	n	2.6E+01	n			n	
7.0E-01	I			4.0E-03	I					1			Bromate	15541-45-4	9.9E-01	c	4.7E+00	c					1.1E-01	c	1.0E+01	8.5E-04	c	7.7E-02
2.0E+00	X	6.0E-04	X					V		1		2.4E+03	Bromo-2-chloroethane, 1-	107-04-0	2.6E-02	c	1.1E-01	c	4.7E-03	c	2.0E-02	c	7.4E-03	c		2.1E-06	c	
				8.0E-03	I	6.0E-02	I	V		1		6.8E+02	Bromobenzene	108-86-1	2.9E+02	n	1.8E+03	ns	6.3E+01	n	2.6E+02	n	6.2E+01	n		4.2E-02	n	
						4.0E-02	X	V		1		4.0E+03	Bromochloromethane	74-97-5	1.5E+02	n	6.3E+02	n	4.2E+01	n	1.8E+02	n	8.3E+01	n		2.1E-02	n	
6.2E-02	I	3.7E-05	C	2.0E-02	I			V		1		9.3E+02	Bromodichloromethane	75-27-4	2.9E-01	c	1.3E+00	c	7.6E-02	c	3.3E-01	c	1.3E-01	c	8.0E+01(F)	3.6E-05	c	2.2E-02
7.9E-03	I	1.1E-06	I	2.0E-02	I			V		1		9.2E+02	Bromoform	75-25-2	1.9E+01	c*	8.6E+01	c	2.6E+00	c	1.1E+01	c	3.3E+00	c	8.0E+01(F)	8.7E-04	c	2.1E-02
				1.4E-03	I	5.0E-03	I	V		1		3.6E+03	Bromomethane	74-83-9	6.8E+00	n	3.0E+01	n	5.2E+00	n	2.2E+01	n	7.5E+00	n		1.9E-03	n	
				5.0E-03	H			V		1			Bromophos	2104-96-3	3.9E+02	n	5.8E+03	n					3.5E+01	n		1.5E-01	n	
				2.0E-02	I					1	0.1		Bromoxynil	1689-84-5	1.3E+03	n	1.6E+04	n					3.3E+02	n		2.8E-01	n	
				2.0E-02	I			V		1			Bromoxynil Octanoate	1689-99-2	1.6E+03	n	2.3E+04	n					1.4E+02	n		1.2E+00	n	
3.4E+00	C	3.0E-05	I			2.0E-03	I	V		1		6.7E+02	Butadiene, 1,3-	106-99-0	5.8E-02	c*	2.6E-01	c*	9.4E-02	c*	4.1E-01	c*	1.8E-02	c		9.9E-06	c	
				1.0E-01	I			V		1		7.6E+03	Butanol, N-	71-36-3	7.8E+03	ns	1.2E+05	nms					2.0E+03	n		4.1E-01	n	
1.9E-03	P			2.0E-01	I					1	0.1		Butyl Benzyl Phthlate	85-68-7	2.9E+02	c*	1.2E+03	c					1.6E+01	c		2.3E-01	c	
				2.0E+00	P	3.0E+01	P	V		1		2.1E+04	Butyl alcohol, sec-	78-92-2	1.3E+05	nms	1.5E+06	nms	3.1E+04	n	1.3E+05	n	2.4E+04	n		5.0E+00	n	
				5.0E-02	I			V		1			Butylate	2008-41-5	3.9E+03	n	5.8E+04	n					4.6E+02	n		4.5E-01	n	
2.0E-04	C	5.7E-08	C							1	0.1		Butylated hydroxyanisole	25013-16-5	2.7E+03	c	1.1E+04	c	4.9E+01	c	2.2E+02	c	2.4E+02	c		4.5E-01	c	
3.6E-03	P			3.0E-01	P					1	0.1		Butylated hydroxytoluene	128-37-0	1.5E+02	c	6.4E+02	c					3.3E+00	c		9.7E-02	c	
				5.0E-02	P			V		1		1.1E+02	Butylbenzene, n-	104-51-8	3.9E+03	ns	5.8E+04	ns					1.0E+03	n		3.2E+00	n	
				1.0E-01	X			V		1		1.5E+02	Butylbenzene, sec-	135-98-8	7.8E+03	ns	1.2E+05	nms					2.0E+03	n		5.9E+00	n	
				1.0E-01	X			V		1		1.8E+02	Butylbenzene, tert-	98-06-6	7.8E+03	ns	1.2E+05	nms					6.9E+02	n		1.6E+00	n	
				2.0E-02	A					1	0.1		Cacodylic Acid	75-60-5	1.3E+03	n	1.6E+04	n					4.0E+02	n			n	
		1.8E-03	I	1.0E-03	I	1.0E-05	A			0.025	0.001		Cadmium (Diet)	7440-43-9	7.1E+01	n	9.8E+02	n										
		1.8E-03	I	5.0E-04	I	1.0E-05	A			0.05	0.001		Cadmium (Water)	7440-43-9					1.6E-03	c**	6.8E-03	c**	9.2E+00	n	5.0E+00	6.9E-01	n	3.8E-01
5.0E-01	C	1.5E-01	C	2.0E-02	C	2.0E-04	C		M	0.025			Calcium Chromate	13765-19-0	3.0E-01	c	6.2E+00	c	6.8E-06	c	8.2E-05	c	4.1E-02	c			c	
				5.0E-01	I	2.2E-03	C			1	0.1		Caprolactam	105-60-2	3.1E+04	n	4.0E+05	nm	2.3E+00	n	9.6E+00	n	9.9E+03	n		2.5E+00	n	
1.5E-01	C	4.3E-05	C	2.0E-03	I					1	0.1		Captafol	2425-06-1	3.6E+00	c*	1.5E+01	c	6.5E-02	c	2.9E-01	c	4.0E-01	c*		7.1E-04	c*	
2.3E-03	C	6.6E-07	C	1.3E-01	I					1	0.1		Captan	133-06-2	2.4E+02	c*	1.0E+03	c	4.3E+00	c	1.9E+01	c	3.1E+01	c*		2.2E-02	c*	
				1.0E-01	I					1	0.1		Carbaryl	63-25-2	6.3E+03	n	8.2E+04	n					1.8E+03	n		1.7E+00	n	
				5.0E-03	I					1	0.1		Carbofuran	1563-66-2	3.2E+02	n	4.1E+03	n					9.4E+01	n	4.0E+01	3.7E-02	n	1.6E-02
				1.0E-01	I	7.0E-01	I	V		1		7.4E+02	Carbon Disulfide	75-15-0	7.7E+02	ns	3.5E+03	ns	7.3E+02	n	3.1E+03	n	8.1E+02	n		2.4E-01	n	
7.0E-02	I	6.0E-06	I	4.0E-03	I	1.0E-01	I	V		1		4.6E+02	Carbon Tetrachloride	56-23-5	6.5E-01	c	2.9E+00	c	4.7E-01	c	2.0E+00	c	4.5E-01	c	5.0E+00	1.8E-04	c	1.9E-03
				1.0E-02	I					1	0.1		Carbosulfan	55285-14-8	6.3E+02	n	8.2E+03	n					5.1E+01	n		1.2E+00	n	
				1.0E-01	I					1	0.1		Carboxin	5234-68-4	6.3E+03	n	8.2E+04	n					1.9E+03	n		1.0E+00	n	
						9.0E-04	I			1			Ceric oxide	1306-38-3	1.3E+06	nm	5.4E+06	nm	9.4E-01	n	3.9E+00	n						
				1.0E-01	I			V		1			Chloral Hydrate	302-17-0	7.8E+03	n	1.2E+05	nm					2.0E+03	n		4.0E-01	n	
				1.5E-02	I					1	0.1		Chloramben	133-90-4	9.5E+02	n	1.2E+04	n					2.9E+02	n		7.0E-02	n	
4.0E-01	H									1	0.1		Chloranil	118-75-2	1.3E+00	c	5.7E+00	c					1.8E-01	c		1.5E-04	c	
3.5E-01	I	1.0E-04	I	5.0E-04	I	7.0E-04	I	V		1	0.04		Chlordane	12789-03-6	1.7E+00	c*	7.5E+00	c*	2.8E-02	c*	1.2E-01	c*	4.5E-02	c*	2.0E+00	3.0E-03	c*	1.4E-01
1.0E+01	I	4.6E-03	C	3.0E-04	I					1	0.1		Chlordecone (Kepone)	143-50-0	5.4E-02	c	2.3E-01	c	6.1E-04	c	2.7E-03	c	3.5E-03	c		1.2E-04	c	
				7.0E-04	A					1	0.1		Chlorfenvinphos	470-90-6	4.4E+01	n	5.7E+02	n					1.1E+01	n		3.1E-02	n	
				2.0E-02	I					1	0.1		Chlorimuron, Ethyl-	90982-32-4	1.3E+03	n	1.6E+04	n					3.9E+02	n		1.3E-01	n	
				1.0E-01	I	1.5E-04	A	V		1		2.8E+03	Chlorine	7782-50-5	1.8E-01	n	7.8E-01	n	1.5E-01	n	6.4E-01	n	3.0E-01	n		1.4E-04	n	
				3.0E-02	I	2.0E-04	I	V		1			Chlorine Dioxide	10049-04-4	2.3E+03	n	3.4E+04	n	2.1E-01	n	8.8E-01	n	4.2E-01	n			n	
				3.0E-02	I					1			Chlorite (Sodium Salt)	7758-19-2	2.3E+03	n	3.5E+04	n					6.0E+02	n	1.0E+03		n	
						5.0E+01	I	V		1		1.2E+03	Chloro-1,1-difluoroethane, 1-	75-68-3	5.4E+04	ns	2.3E+05	nms	5.2E+04	n	2.2E+05	n	1.0E+05	n		5.2E+01	n	
		3.0E-04	I	2.0E-02	H	2.0E-02	I	V		1		7.5E+02	Chloro-1,3-butadiene, 2-	126-99-8	1.0E-02	c	4.4E-02	c	9.4E-03	c	4.1E-02	c	1.9E-02	c		9.8E-06	c	
4.6E-01	H									1	0.1		Chloro-2-methylaniline HCl, 4-	3165-93-3	1.2E+00	c	5.0E+00	c					1.7E-01	c		1.5E-04	c	
1.0E-01	P	7.7E-05	C	3.0E-03	X					1	0.1		Chloro-2-methylaniline, 4-	95-69-2	5.4E+00	c*	2.3E+01	c	3.6E-02	c	1.6E-01	c	6.9E-01	c*		3.9E-04	c*	
2.7E-01	X							V		1		2.8E+04	Chloroacetaldehyde, 2-	107-20-0	2.6E+00	c	1.2E+01	c					2.9E-01	c		5.8E-05	c	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
				2.0E-03	H					1	0.1		Chloroacetic Acid	79-11-8	1.3E+02	n	1.6E+03	n					4.0E+01	n	6.0E+01	8.1E-03	n	1.2E-02
						3.0E-05	I			1	0.1		Chloroacetophenone, 2-	532-27-4	4.3E+04	n	1.8E+05	nm	3.1E-02	n	1.3E-01	n						
2.0E-01	P			4.0E-03	I					1	0.1		Chloroaniline, p-	106-47-8	2.7E+00	c*	1.1E+01	c					3.6E-01	c		1.6E-04	c	
				2.0E-02	I	5.0E-02	P	V		1		7.6E+02	Chlorobenzene	108-90-7	2.8E+02	n	1.3E+03	ns	5.2E+01	n	2.2E+02	n	7.8E+01	n	1.0E+02	5.3E-02	n	6.8E-02
1.1E-01	C	3.1E-05	C	2.0E-02	I					1	0.1		Chlorobenzilate	510-15-6	4.9E+00	c	2.1E+01	c	9.1E-02	c	4.0E-01	c	3.1E-01	c		1.0E-03	c	
				3.0E-02	X					1	0.1		Chlorobenzoic Acid, p-	74-11-3	1.9E+03	n	2.5E+04	n					5.1E+02	n		1.3E-01	n	
				3.0E-03	P	3.0E-01	P	V		1		1.2E+02	Chlorobenzotrifluoride, 4-	98-56-6	2.1E+02	ns	2.5E+03	ns	3.1E+02	n	1.3E+03	n	3.5E+01	n		1.2E-01	n	
				4.0E-02	P			V		1		7.3E+02	Chlorobutane, 1-	109-69-3	3.1E+03	ns	4.7E+04	ns					6.4E+02	n		2.6E-01	n	
						5.0E+01	I	V		1		1.7E+03	Chlorodifluoromethane	75-45-6	4.9E+04	ns	2.1E+05	nms	5.2E+04	n	2.2E+05	n	1.0E+05	n		4.3E+01	n	
				2.0E-02	P			V		1		1.1E+05	Chloroethanol, 2-	107-07-3	1.6E+03	n	2.3E+04	n					4.0E+02	n		8.1E-02	n	
3.1E-02	C	2.3E-05	I	1.0E-02	I	9.8E-02	A	V		1		2.5E+03	Chloroform	67-66-3	3.2E-01	c	1.4E+00	c	1.2E-01	c	5.3E-01	c	2.2E-01	c	8.0E+01(F)	6.1E-05	c	2.2E-02
						9.0E-02	I	V		1		1.3E+03	Chloromethane	74-87-3	1.1E+02	n	4.6E+02	n	9.4E+01	n	3.9E+02	n	1.9E+02	n		4.9E-02	n	
2.4E+00	C	6.9E-04	C					V		1		2.6E+04	Chloromethyl Methyl Ether	107-30-2	2.0E-02	c	8.9E-02	c	4.1E-03	c	1.8E-02	c	6.5E-03	c		1.4E-06	c	
3.0E-01	P			3.0E-03	P	1.0E-05	X			1	0.1		Chloronitrobenzene, o-	88-73-3	1.8E+00	c	7.7E+00	c	1.0E-02	n	4.4E-02	n	2.3E-01	c		2.2E-04	c	
6.3E-03	P			1.0E-03	P	6.0E-04	P			1	0.1		Chloronitrobenzene, p-	100-00-5	6.3E+01	n	3.6E+02	c**	6.3E-01	n	2.6E+00	n	1.1E+01	c**		1.0E-02	c**	
				5.0E-03	I			V		1		2.2E+04	Chlorophenol, 2-	95-57-8	3.9E+02	n	5.8E+03	n					9.1E+01	n		7.4E-02	n	
						4.0E-04	C	V		1		6.2E+02	Chloropicrin	76-06-2	2.0E+00	n	8.2E+00	n	4.2E-01	n	1.8E+00	n	8.3E-01	n		2.5E-04	n	
3.1E-03	C	8.9E-07	C	1.5E-02	I					1	0.1		Chlorothalonil	1897-45-6	1.8E+02	c**	7.4E+02	c*	3.2E+00	c	1.4E+01	c	2.2E+01	c*		4.9E-02	c*	
				2.0E-02	I			V		1		9.1E+02	Chlorotoluene, o-	95-49-8	1.6E+03	ns	2.3E+04	ns					2.4E+02	n		2.3E-01	n	
				2.0E-02	X			V		1		2.5E+02	Chlorotoluene, p-	106-43-4	1.6E+03	ns	2.3E+04	ns					2.5E+02	n		2.4E-01	n	
2.4E+02	C	6.9E-02	C							1	0.1		Chlorozotocin	54749-90-5	2.3E-03	c	9.6E-03	c	4.1E-05	c	1.8E-04	c	3.2E-04	c		7.1E-08	c	
				2.0E-01	I					1	0.1		Chlorpropham	101-21-3	1.3E+04	n	1.6E+05	nm					2.8E+03	n		2.6E+00	n	
				1.0E-03	A					1	0.1		Chlorpyrifos	2921-88-2	6.3E+01	n	8.2E+02	n					8.4E+00	n		1.2E-01	n	
				1.0E-02	H					1	0.1		Chlorpyrifos Methyl	5598-13-0	6.3E+02	n	8.2E+03	n					1.2E+02	n		5.4E-01	n	
				5.0E-02	I					1	0.1		Chlorsulfuron	64902-72-3	3.2E+03	n	4.1E+04	n					9.9E+02	n		8.3E-01	n	
				8.0E-04	H					1	0.1		Chlorthiophos	60238-56-4	5.1E+01	n	6.6E+02	n					2.8E+00	n		7.3E-02	n	
				1.5E+00	I					0.013			Chromium(III), Insoluble Salts	16065-83-1	1.2E+05	nm	1.8E+06	nm					2.2E+04	n		4.0E+07	n	
5.0E-01	J	8.4E-02	S	3.0E-03	I	1.0E-04	I		M	0.025			Chromium(VI)	18540-29-9	3.0E-01	c	6.3E+00	c	1.2E-05	c	1.5E-04	c	3.5E-02	c		6.7E-04	c	
										0.013			Chromium, Total	7440-47-3											1.0E+02			1.8E+05
		9.0E-03	P	3.0E-04	P	6.0E-06	P			1			Cobalt	7440-48-4	2.3E+01	n	3.5E+02	n	3.1E-04	c*	1.4E-03	c*	6.0E+00	n		2.7E-01	n	
		6.2E-04	I					V	M	1			Coke Oven Emissions	8007-45-2					1.6E-03	c	2.0E-02	c						
				4.0E-02	H					1			Copper	7440-50-8	3.1E+03	n	4.7E+04	n					8.0E+02	n	1.3E+03	2.8E+01	n	4.6E+01
				5.0E-02	I	6.0E-01	C			1	0.1		Cresol, m-	108-39-4	3.2E+03	n	4.1E+04	n	6.3E+02	n	2.6E+03	n	9.3E+02	n		7.4E-01	n	
				5.0E-02	I	6.0E-01	C			1	0.1		Cresol, p-	95-48-7	3.2E+03	n	4.1E+04	n	6.3E+02	n	2.6E+03	n	9.3E+02	n		7.5E-01	n	
				1.0E-01	A	6.0E-01	C			1	0.1		Cresol, p-	106-44-5	6.3E+03	n	8.2E+04	n	6.3E+02	n	2.6E+03	n	1.9E+03	n		1.5E+00	n	
				1.0E-01	A					1	0.1		Cresol, p-chloro-m-	59-50-7	6.3E+03	n	8.2E+04	n					1.4E+03	n		1.7E+00	n	
				1.0E-01	A	6.0E-01	C			1	0.1		Cresols	1319-77-3	6.3E+03	n	8.2E+04	n	6.3E+02	n	2.6E+03	n	1.9E+03	n		1.5E+00	n	
1.9E+00	H			1.0E-03	P			V		1		1.7E+04	Crotonaldehyde, trans-	123-73-9	3.7E-01	c	1.7E+00	c					4.0E-02	c		8.2E-06	c	
				1.0E-01	I	4.0E-01	I	V		1		2.7E+02	Cumene	98-82-8	1.9E+03	ns	9.9E+03	ns	4.2E+02	n	1.8E+03	n	4.5E+02	n		7.4E-01	n	
2.2E-01	C	6.3E-05	C							1	0.1		Cupferron	135-20-6	2.5E+00	c	1.0E+01	c	4.5E-02	c	1.9E-01	c	3.5E-01	c		6.1E-04	c	
8.4E-01	H			2.0E-03	H					1	0.1		Cyanazine	21725-46-2	6.5E-01	c	2.7E+00	c					8.7E-02	c		4.1E-05	c	
													Cyanides															
				1.0E-03	I					1			~Calcium Cyanide	592-01-8	7.8E+01	n	1.2E+03	n					2.0E+01	n			n	
				5.0E-03	I					1			~Copper Cyanide	544-92-3	3.9E+02	n	5.8E+03	n					1.0E+02	n			n	
				6.0E-04	I	8.0E-04	S	V		1		9.7E+05	~Cyanide (CN-)	57-12-5	2.7E+00	n	1.2E+01	n	8.3E-01	n	3.5E+00	n	1.5E+00	n	2.0E+02	1.5E-02	n	2.0E+00
				1.0E-03	I			V		1			~Cyanogen	460-19-5	7.8E+01	n	1.2E+03	n					2.0E+01	n			n	
				9.0E-02	I			V		1			~Cyanogen Bromide	506-68-3	7.0E+03	n	1.1E+05	nm					1.8E+03	n			n	
				5.0E-02	I			V		1			~Cyanogen Chloride	506-77-4	3.9E+03	n	5.8E+04	n					1.0E+03	n			n	
				6.0E-04	I	8.0E-04	I	V		1		1.0E+07	~Hydrogen Cyanide	74-90-8	2.3E+01	n	1.5E+02	n	8.3E-01	n	3.5E+00	n	1.5E+00	n		1.5E-02	n	
				2.0E-03	I					1			~Potassium Cyanide	151-50-8	1.6E+02	n	2.3E+03	n					4.0E+01	n			n	
				5.0E-03	I					0.04			~Potassium Silver Cyanide	506-61-6	3.9E+02	n	5.8E+03	n					8.2E+01	n			n	
				1.0E-01	I					0.04			~Silver Cyanide	506-64-9	7.8E+03	n	1.2E+05	nm					1.8E+03	n			n	
				1.0E-03	I					1			~Sodium Cyanide	143-33-9	7.8E+01	n	1.2E+03	n					2.0E+01	n	2.0E+02		n	
				2.0E-04	P					1			~Thiocyanates	NA	1.6E+01	n	2.3E+02	n					4.0E+00	n			n	
				2.0E-04	X			V		1			~Thiocyanic Acid	463-56-9	1.6E+01	n	2.3E+02	n					4.0E+00	n			n	
				5.0E-02	I					1			~Zinc Cyanide	557-21-1	3.9E+03	n	5.8E+04	n					1.0E+03	n			n	
						6.0E+00	I	V		1		1.2E+02	Cyclohexane	110-82-7	6.5E+03	ns	2.7E+04	ns	6.3E+03	n	2.6E+04	n	1.3E+04	n		1.3E+01	n	
2.3E-02	H									1	0.1		Cyclohexane, 1,2,3,4,5-pentabromo-6-chloro-	87-84-3	2.4E+01	c	1.0E+02	c					2.4E+00	c		1.4E-02	c	
				5.0E+00	I	7.0E-01	P	V		1		5.1E+03	Cyclohexanone	108-94-1	2.8E+04	ns	1.3E+05	nms	7.3E+02	n	3.1E+03	n	1.4E+03	n		3.4E-01	n	
				5.0E-03	P	1.0E+00	X	V		1		2.8E+02	Cyclohexene	110-83-8	3.1E+02	ns	3.1E+03	ns	1.0E+03	n	4.4E+03	n	7.0E+01	n		4.6E-02	n	
				2.0E-01	I			V		1		2.9E+05	Cyclohexylamine	108-91-8	1.6E+04	n	2.3E+05	nm					3.8E+03	n		1.0E+00	n	
				5.0E-03	I					1	0.1		Cyhalothrin/karate	68085-85-8	3.2E+02	n	4.1E+03	n					1.0E+02	n		6.8E+01	n	
				1.0E-02	I					1	0.1		Cypermethrin	52315-07-8	6.3E+02	n	8.2E+03	n					2.0E+02	n		3.2E+01	n	
				7.5E-03	I					1	0.1		Cyromazine	66215-27-8	4.7E+02	n	6.2E+03	n					1.5E+02	n		3.8E-02	n	
2.4E-01	I	6.9E-05	C							1	0.1		DDD	72-54-8	2.3E+00	c	9.6E+00	c	4.1E-02	c	1.8E-01	c	3.1E-02	c		7.2E-03	c	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels										Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
3.4E-01	I	9.7E-05	C					V		1			DDE, p,p'-	72-55-9	2.0E+00	c	9.3E+00	c	2.9E-02	c	1.3E-01	c	4.6E-02	c		1.1E-02	c	
3.4E-01	I	9.7E-05	I	5.0E-04	I					1	0.03		DDT	50-29-3	1.9E+00	c*	8.5E+00	c*	2.9E-02	c	1.3E-01	c	2.3E-01	c*		7.7E-02	c*	
				1.0E-02	I					1	0.1		Dacthal	1861-32-1	6.3E+02	n	8.2E+03	n					1.2E+02	n		1.5E-01	n	
				3.0E-02	I					1	0.1		Dalapon	75-99-0	1.9E+03	n	2.5E+04	n					6.0E+02	n	2.0E+02	1.2E-01	n	4.1E-02
7.0E-04	I			7.0E-03	I					1	0.1		Decabromodiphenyl ether, 2,2',3,3',4,4',5,5',6,6'- (BDE-209)	1163-19-5	4.4E+02	n	3.3E+03	c**					1.1E+02	c**		6.2E+01	c**	
				4.0E-05	I					1	0.1		Demeton	8065-48-3	2.5E+00	n	3.3E+01	n					6.7E-01	n			n	
1.2E-03	I			6.0E-01	I					1	0.1		Di(2-ethylhexyl)adipate	103-23-1	4.5E+02	c*	1.9E+03	c					6.5E+01	c	4.0E+02	4.7E+00	c	2.9E+01
6.1E-02	H									1	0.1		Diallate	2303-16-4	8.9E+00	c	3.8E+01	c					5.2E-01	c		7.8E-04	c	
				7.0E-04	A					1	0.1		Diazinon	333-41-5	4.4E+01	n	5.7E+02	n					1.0E+01	n		6.5E-02	n	
				1.0E-02	X			V		1			Dibenzothiophene	132-65-0	7.8E+02	n	1.2E+04	n					6.5E+01	n		1.2E+00	n	
8.0E-01	P	6.0E-03	P	2.0E-04	P	2.0E-04	I	V	M	1		9.8E+02	Dibromo-3-chloropropane, 1,2-	96-12-8	5.3E-03	c	6.4E-02	c	1.7E-04	c	2.0E-03	c	3.3E-04	c*	2.0E-01	1.4E-07	c*	8.6E-05
				4.0E-04	X			V		1		1.6E+02	Dibromobenzene, 1,3-	108-36-1	3.1E+01	n	4.7E+02	ns					5.3E+00	n		5.1E-03	n	
				1.0E-02	I			V		1			Dibromobenzene, 1,4-	106-37-6	7.8E+02	n	1.2E+04	n					1.3E+02	n		1.2E-01	n	
8.4E-02	I	2.7E-05	C	2.0E-02	I			V		1		8.0E+02	Dibromochloromethane	124-48-1	7.5E-01	c	3.3E+00	c	1.0E-01	c	4.5E-01	c	1.7E-01	c	8.0E+01(F)	4.5E-05	c	2.1E-02
2.0E+00	I	6.0E-04	I	9.0E-03	I	9.0E-03	I	V		1		1.3E+03	Dibromoethane, 1,2-	106-93-4	3.6E-02	c	1.6E-01	c	4.7E-03	c	2.0E-02	c	7.5E-03	c	5.0E-02	2.1E-06	c	1.4E-05
				1.0E-02	H	4.0E-03	X	V		1		2.8E+03	Dibromomethane (Methylene Bromide)	74-95-3	2.3E+01	n	9.8E+01	n	4.2E+00	n	1.8E+01	n	8.0E+00	n		2.0E-03	n	
				3.0E-04	P					1	0.1		Dibutyltin Compounds	NA	1.9E+01	n	2.5E+02	n					6.0E+00	n			n	
				3.0E-02	I					1	0.1		Dicamba	1918-00-9	1.9E+03	n	2.5E+04	n					5.7E+02	n		1.5E-01	n	
		4.2E-03	P					V		1		5.2E+02	Dichloro-2-butene, 1,4-	764-41-0	8.3E-03	c	3.6E-02	c	6.7E-04	c	2.9E-03	c	1.3E-03	c		6.2E-07	c	
		4.2E-03	P					V		1		5.2E+02	Dichloro-2-butene, cis-1,4-	1476-11-5	7.4E-03	c	3.2E-02	c	6.7E-04	c	2.9E-03	c	1.3E-03	c		6.2E-07	c	
		4.2E-03	P					V		1		7.6E+02	Dichloro-2-butene, trans-1,4-	110-57-6	7.4E-03	c	3.2E-02	c	6.7E-04	c	2.9E-03	c	1.3E-03	c		6.2E-07	c	
5.0E-02	I			4.0E-03	I					1	0.1		Dichloroacetic Acid	79-43-6	1.1E+01	c*	4.6E+01	c*					1.5E+00	c*	6.0E+01	3.1E-04	c*	1.2E-02
				9.0E-02	I	2.0E-01	H	V		1		3.8E+02	Dichlorobenzene, 1,2-	95-50-1	1.8E+03	ns	9.3E+03	ns	2.1E+02	n	8.8E+02	n	3.0E+02	n	6.0E+02	3.0E-01	n	5.8E-01
5.4E-03	C	1.1E-05	C	7.0E-02	A	8.0E-01	I	V		1			Dichlorobenzene, 1,4-	106-46-7	2.6E+00	c	1.1E+01	c	2.6E-01	c	1.1E+00	c	4.8E-01	c	7.5E+01	4.6E-04	c	7.2E-02
4.5E-01	I	3.4E-04	C							1	0.1		Dichlorobenzidine, 3,3'-	91-94-1	1.2E+00	c	5.1E+00	c	8.3E-03	c	3.6E-02	c	1.2E-01	c		8.1E-04	c	
				9.0E-03	X					1	0.1		Dichlorobenzophenone, 4,4'-	90-98-2	5.7E+02	n	7.4E+03	n					7.8E+01	n		4.7E-01	n	
				2.0E-01	I	1.0E-01	X	V		1		8.5E+02	Dichlorodifluoromethane	75-71-8	8.7E+01	n	3.7E+02	n	1.0E+02	n	4.4E+02	n	2.0E+02	n		3.0E-01	n	
5.7E-03	C	1.6E-06	C	2.0E-01	P			V		1		1.7E+03	Dichloroethane, 1,1-	75-34-3	3.6E+00	c	1.6E+01	c	1.8E+00	c	7.7E+00	c	2.7E+00	c		7.8E-04	c	
9.1E-02	I	2.6E-05	I	6.0E-03	X	7.0E-03	P	V		1		3.0E+03	Dichloroethane, 1,2-	107-06-2	4.6E-01	c*	2.0E+00	c*	1.1E-01	c*	4.7E-01	c*	1.7E-01	c*	5.0E+00	4.8E-05	c*	1.4E-03
				5.0E-02	I	2.0E-01	I	V		1		1.2E+03	Dichloroethylene, 1,1-	75-35-4	2.3E+02	n	1.0E+03	n	2.1E+02	n	8.8E+02	n	2.8E+02	n	7.0E+00	1.0E-01	n	2.5E-03
				2.0E-03	I			V		1		2.4E+03	Dichloroethylene, 1,2-cis-	156-59-2	1.6E+02	n	2.3E+03	n					3.6E+01	n	7.0E+01	1.1E-02	n	2.1E-02
				2.0E-02	I			V		1		1.9E+03	Dichloroethylene, 1,2-trans-	156-60-5	1.6E+03	n	2.3E+04	ns					3.6E+02	n	1.0E+02	1.1E-01	n	3.1E-02
				3.0E-03	I					1	0.1		Dichlorophenol, 2,4-	120-83-2	1.9E+02	n	2.5E+03	n					4.6E+01	n		5.4E-02	n	
				1.0E-02	I					1	0.05		Dichlorophenoxy Acetic Acid, 2,4-	94-75-7	7.0E+02	n	9.6E+03	n					1.7E+02	n	7.0E+01	4.5E-02	n	1.8E-02
				8.0E-03	I					1	0.1		Dichlorophenoxybutyric Acid, 4-(2,4-	94-82-6	5.1E+02	n	6.6E+03	n					1.2E+02	n		1.1E-01	n	
3.6E-02	C	1.0E-05	C	9.0E-02	A	4.0E-03	I	V		1		1.4E+03	Dichloropropane, 1,2-	78-87-5	1.0E+00	c*	4.4E+00	c*	2.8E-01	c*	1.2E+00	c*	4.4E-01	c*	5.0E+00	1.5E-04	c*	1.7E-03
				2.0E-02	P			V		1		1.5E+03	Dichloropropane, 1,3-	142-28-9	1.6E+03	ns	2.3E+04	ns					3.7E+02	n		1.3E-01	n	
				3.0E-03	I					1	0.1		Dichloropropanol, 2,3-	616-23-9	1.9E+02	n	2.5E+03	n					5.9E+01	n		1.3E-02	n	
1.0E-01	I	4.0E-06	I	3.0E-02	I	2.0E-02	I	V		1		1.6E+03	Dichloropropene, 1,3-	542-75-6	1.8E+00	c*	8.2E+00	c*	7.0E-01	c*	3.1E+00	c*	4.7E-01	c*		1.7E-04	c*	
2.9E-01	I	8.3E-05	C	5.0E-04	I	5.0E-04	I			1	0.1		Dichlorvos	62-73-7	1.9E+00	c*	7.9E+00	c*	3.4E-02	c*	1.5E-01	c*	2.6E-01	c*		8.1E-05	c*	
				8.0E-02	P	3.0E-04	X	V		1			Dicyclopentadiene	77-73-6	1.3E+00	n	5.4E+00	n	3.1E-01	n	1.3E+00	n	6.3E-01	n		2.2E-03	n	
1.6E+01	I	4.6E-03	I	5.0E-05	I					1	0.1		Dieldrin	60-57-1	3.4E-02	c*	1.4E-01	c	6.1E-04	c	2.7E-03	c	1.7E-03	c		6.9E-05	c	
		3.0E-04	C			5.0E-03	I			1	0.1		Diesel Engine Exhaust	NA					9.4E-03	c	4.1E-02	c						
				2.0E-03	P	2.0E-04	P			1	0.1		Diethanolamine	111-42-2	1.3E+02	n	1.6E+03	n	2.1E-01	n	8.8E-01	n	4.0E+01	n		8.1E-03	n	
				3.0E-02	P	1.0E-04	P			1	0.1		Diethylene Glycol Monobutyl Ether	112-34-5	1.9E+03	n	2.4E+04	n	1.0E-01	n	4.4E-01	n	6.0E+02	n		1.3E-01	n	
				6.0E-02	P	3.0E-04	P			1	0.1		Diethylene Glycol Monoethyl Ether	111-90-0	3.8E+03	n	4.8E+04	n	3.1E-01	n	1.3E+00	n	1.2E+03	n		2.4E-01	n	
				1.0E-03	P			V		1		1.1E+05	Diethylformamide	617-84-5	7.8E+01	n	1.2E+03	n					2.0E+01	n		4.1E-03	n	
3.5E+02	C	1.0E-01	C							1	0.1		Diethylstilbestrol	56-53-1	1.6E-03	c	6.6E-03	c	2.8E-05	c	1.2E-04	c	4.9E-05	c		2.7E-05	c	
				8.0E-02	I					1	0.1		Difenzoquat	43222-48-6	5.1E+03	n	6.6E+04	n					1.6E+03	n			n	
				2.0E-02	I					1	0.1		Diflubenzuron	35367-38-5	1.3E+03	n	1.6E+04	n					2.9E+02	n		3.3E-01	n	
						4.0E+01	I	V		1		1.4E+03	Difluoroethane, 1,1-	75-37-6	4.8E+04	ns	2.0E+05	nms	4.2E+04	n	1.8E+05	n	8.3E+04	n		2.8E+01	n	
4.4E-02	C	1.3E-05	C					V		1			Dihydrosafrole	94-58-6	3.2E-01	c	1.4E+00	c	2.2E-01	c	9.4E-01	c	3.0E-01	c		3.7E-04	c	
						7.0E-01	P	V		1		2.3E+03	Diisopropyl Ether	108-20-3	2.2E+03	n	9.4E+03	ns	7.3E+02	n	3.1E+03	n	1.5E+03	n		3.7E-01	n	
				8.0E-02	I			V		1		5.3E+02	Diisopropyl Methylphosphonate	1445-75-6	6.3E+03	ns	9.3E+04	ns					1.6E+03	n		4.5E-01	n	
				2.0E-02	I					1	0.1		Dimethipin	55290-64-7	1.3E+03	n	1.6E+04	n					4.0E+02	n		8.8E-02	n	
				2.0E-04	I					1	0.1		Dimethoate	60-51-5	1.3E+01	n	1.6E+02	n					4.0E+00	n		9.0E-04	n	
1.6E+00	P									1	0.1		Dimethoxybenzidine, 3,3'-	119-90-4	3.4E-01	c	1.4E+00	c					4.7E-02	c		5.7E-05	c	
1.7E-03	P			6.0E-02	P					1	0.1		Dimethyl methylphosphonate	756-79-6	3.2E+02	c*	1.4E+03	c*					4.6E+01	c*		9.6E-03	c*	
4.6E+00	C	1.3E-03	C							1	0.1		Dimethylamino azobenzene [p-]	60-11-7	1.2E-01	c	5.0E-01	c	2.2E-03	c	9.4E-03	c	4.9E-03	c		2.1E-05	c	
5.8E-01	H									1	0.1		Dimethylaniline HCl, 2,4-	21436-96-4	9.4E-01	c	4.0E+00	c					1.3E-01	c		1.2E-04	c	
2.0E-01	P			2.0E-03	X					1	0.1		Dimethylaniline, 2,4-	95-68-1	2.7E+00	c*	1.1E+01	c					3.7E-01	c		2.1E-04	c	
				2.0E-03	I			V		1		8.3E+02	Dimethylaniline, N,N-	121-69-7	1.6E+02	n	2.3E+03	ns					3.5E+01	n		1.3E-02	n	
1.1E+01	P									1	0.1		Dimethylbenzidine, 3,3'-	119-93-7	4.9E-02	c	2.1E-01	c					6.5E-03	c		4.3E-05	c	
				1.0E-01	P	3.0E-02	I	V		1		1.1E+05	Dimethylformamide	68-12-2	2.6E+03	n	1.5E+04	n	3.1E+01	n	1.3E+02	n	6.1E+01	n		1.2E-02	n	
				1.0E-04	X	2.0E-06	X	V		1		1.7E+05	Dimethylhydrazine, 1,1-	57-14-7	3.2E-01	n	1.4E+00	n	2.1E-03	n	8.8E-03	n	4.2E-03	n		9.3E-07	n	

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Toxicity and Chemical-specific Information													Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	vol	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
5.5E+02	C	1.6E-01	C					V		1		1.9E+05	Dimethylhydrazine, 1,2-	540-73-8	8.8E-04	c	4.1E-03	c	1.8E-05	c	7.7E-05	c	2.8E-05	c		6.5E-09	c		
				2.0E-02	I					1	0.1		Dimethylphenol, 2,4-	105-67-9	1.3E+03	n	1.6E+04	n					3.6E+02	n		4.2E-01	n		
				6.0E-04	I					1	0.1		Dimethylphenol, 2,6-	576-26-1	3.8E+01	n	4.9E+02	n					1.1E+01	n		1.3E-02	n		
				1.0E-03	I					1	0.1		Dimethylphenol, 3,4-	95-65-8	6.3E+01	n	8.2E+02	n					1.8E+01	n		2.1E-02	n		
4.5E-02	C	1.3E-05	C					V		1		1.1E+03	Dimethylvinylchloride	513-37-1	2.1E-01	c	9.4E-01	c	2.2E-01	c	9.4E-01	c	3.3E-01	c		2.0E-04	c		
				8.0E-05	X					1	0.1		Dinitro-o-cresol, 4,6-	534-52-1	5.1E+00	n	6.6E+01	n					1.5E+00	n		2.6E-03	n		
				2.0E-03	I					1	0.1		Dinitro-o-cyclohexyl Phenol, 4,6-	131-89-5	1.3E+02	n	1.6E+03	n					2.3E+01	n		7.7E-01	n		
				1.0E-04	P					1	0.1		Dinitrobenzene, 1,2-	528-29-0	6.3E+00	n	8.2E+01	n					1.9E+00	n		1.8E-03	n		
				1.0E-04	I					1	0.1		Dinitrobenzene, 1,3-	99-65-0	6.3E+00	n	8.2E+01	n					2.0E+00	n		1.8E-03	n		
				1.0E-04	P					1	0.1		Dinitrobenzene, 1,4-	100-25-4	6.3E+00	n	8.2E+01	n					2.0E+00	n		1.8E-03	n		
				2.0E-03	I					1	0.1		Dinitrophenol, 2,4-	51-28-5	1.3E+02	n	1.6E+03	n					3.9E+01	n		4.4E-02	n		
6.8E-01	I									1	0.1		Dinitrotoluene Mixture, 2,4/2,6-	NA	8.0E-01	c	3.4E+00	c					1.1E-01	c		1.5E-04	c		
3.1E-01	C	8.9E-05	C	2.0E-03	I					1	0.102		Dinitrotoluene, 2,4-	121-14-2	1.7E+00	c*	7.4E+00	c	3.2E-02	c	1.4E-01	c	2.4E-01	c		3.2E-04	c		
1.5E+00	P			3.0E-04	X					1	0.099		Dinitrotoluene, 2,6-	606-20-2	3.6E-01	c*	1.5E+00	c					4.8E-02	c		6.7E-05	c		
				2.0E-03	S					1	0.006		Dinitrotoluene, 2-Amino-4,6-	35572-78-2	1.5E+02	n	2.3E+03	n					3.9E+01	n		3.0E-02	n		
				2.0E-03	S					1	0.009		Dinitrotoluene, 4-Amino-2,6-	19406-51-0	1.5E+02	n	2.3E+03	n					3.9E+01	n		3.0E-02	n		
4.5E-01	X			9.0E-04	X					1	0.1		Dinitrotoluene, Technical grade	25321-14-6	1.2E+00	c*	5.1E+00	c					1.6E-01	c		2.2E-04	c		
				1.0E-03	I					1	0.1		Dinoseb	88-85-7	6.3E+01	n	8.2E+02	n					1.5E+01	n	7.0E+00	1.3E-01	n	6.2E-02	
1.0E-01	I	5.0E-06	I	3.0E-02	I	3.0E-02	I	V		1		1.2E+05	Dioxane, 1,4-	123-91-1	5.3E+00	c	2.4E+01	c	5.6E-01	c*	2.5E+00	c*	4.6E-01	c		9.4E-05	c		
													Dioxins																	
6.2E+03	I	1.3E+00	I							1	0.03		1-Hexachlorodibenzo-p-dioxin, Mixture	NA	1.9E-04	c	4.7E-04	c	2.2E-06	c	9.4E-06	c	1.3E-05	c		1.7E-05	c		
1.3E+05	C	3.8E+01	C	7.0E-10	I	4.0E-08	C	V		1	0.03		2,3,7,8-TCDD	1746-01-6	4.8E-06	c*	2.2E-05	c*	7.4E-08	c	3.2E-07	c	1.2E-07	c	3.0E-05	5.9E-08	c	1.5E-05	
				3.0E-02	I					1	0.1		Diphenamid	957-51-7	1.9E+03	n	2.5E+04	n					5.3E+02	n		5.2E+00	n		
				8.0E-04	X					1	0.1		Diphenyl Sulfone	127-63-9	5.1E+01	n	6.6E+02	n					1.5E+01	n		3.6E-02	n		
				2.5E-02	I					1	0.1		Diphenylamine	122-39-4	1.6E+03	n	2.1E+04	n					3.1E+02	n		5.8E-01	n		
8.0E-01	I	2.2E-04	I							1	0.1		Diphenylhydrazine, 1,2-	122-66-7	6.8E-01	c	2.9E+00	c	1.3E-02	c	5.6E-02	c	7.7E-02	c		2.5E-04	c		
				2.2E-03	I					1	0.1		Diquat	85-00-7	1.4E+02	n	1.8E+03	n					4.4E+01	n	2.0E+01	8.3E-01	n	3.7E-01	
7.1E+00	C	1.4E-01	C							1	0.1		Direct Black 38	1937-37-7	7.6E-02	c	3.2E-01	c	2.0E-05	c	8.8E-05	c	1.1E-02	c		5.3E+00	c		
7.4E+00	C	1.4E-01	C							1	0.1		Direct Blue 6	2602-46-2	7.3E-02	c	3.1E-01	c	2.0E-05	c	8.8E-05	c	1.1E-02	c		1.7E+01	c		
6.7E+00	C	1.4E-01	C							1	0.1		Direct Brown 95	16071-86-6	8.1E-02	c	3.4E-01	c	2.0E-05	c	8.8E-05	c	1.2E-02	c			c		
				4.0E-05	I					1	0.1		Disulfoton	298-04-4	2.5E+00	n	3.3E+01	n					5.0E-01	n		9.4E-04	n		
				1.0E-02	I			V		1			Dithiane, 1,4-	505-29-3	7.8E+02	n	1.2E+04	n					2.0E+02	n		9.7E-02	n		
				2.0E-03	I					1	0.1		Diuron	330-54-1	1.3E+02	n	1.6E+03	n					3.6E+01	n		1.5E-02	n		
				4.0E-03	I					1	0.1		Dodine	2439-10-3	2.5E+02	n	3.3E+03	n					8.0E+01	n		4.1E-01	n		
				2.5E-02	I			V		1			EPTC	759-94-4	2.0E+03	n	2.9E+04	n					3.8E+02	n		2.0E-01	n		
				6.0E-03	I			V		1			Endosulfan	115-29-7	4.7E+02	n	7.0E+03	n					1.0E+02	n		1.4E+00	n		
				2.0E-02	I					1	0.1		Endothall	145-73-3	1.3E+03	n	1.6E+04	n					3.8E+02	n	1.0E+02	9.1E-02	n	2.4E-02	
				3.0E-04	I					1	0.1		Endrin	72-20-8	1.9E+01	n	2.5E+02	n					2.3E+00	n	2.0E+00	9.2E-02	n	8.1E-02	
9.9E-03	I	1.2E-06	I	6.0E-03	P	1.0E-03	I	V		1		1.1E+04	Epichlorohydrin	106-89-8	1.9E+01	n	8.2E+01	n	1.0E+00	n	4.4E+00	n	2.0E+00	n		4.5E-04	n		
						2.0E-02	I	V		1		1.5E+04	Epoxybutane, 1,2-	106-88-7	1.6E+02	n	6.7E+02	n	2.1E+01	n	8.8E+01	n	4.2E+01	n		9.2E-03	n		
				5.0E-03	I					1	0.1		Ethephon	16672-87-0	3.2E+02	n	4.1E+03	n					1.0E+02	n		2.1E-02	n		
				5.0E-04	I					1	0.1		Ethion	563-12-2	3.2E+01	n	4.1E+02	n					4.3E+00	n		8.5E-03	n		
				1.0E-01	P	6.0E-02	P	V		1		3.1E+04	Ethoxyethanol Acetate, 2-	111-15-9	2.6E+03	n	1.4E+04	n	6.3E+01	n	2.6E+02	n	1.2E+02	n		2.5E-02	n		
				9.0E-02	P	2.0E-01	I	V		1		1.1E+05	Ethoxyethanol, 2-	110-80-5	5.2E+03	n	4.7E+04	n	2.1E+02	n	8.8E+02	n	3.4E+02	n		6.8E-02	n		
				9.0E-01	I	7.0E-02	P	V		1		1.1E+04	Ethyl Acetate	141-78-6	6.2E+02	n	2.6E+03	n	7.3E+01	n	3.1E+02	n	1.4E+02	n		3.1E-02	n		
4.8E-02	H			5.0E-03	P	8.0E-03	P	V		1		2.5E+03	Ethyl Acrylate	140-88-5	1.4E+01	c**	6.8E+01	c**	8.3E+00	n	3.5E+01	n	1.6E+00	c**		3.5E-04	c**		
						1.0E+01	I	V		1		2.1E+03	Ethyl Chloride (Chloroethane)	75-00-3	1.4E+04	ns	5.7E+04	ns	1.0E+04	n	4.4E+04	n	2.1E+04	n		5.9E+00	n		
				2.0E-01	I			V		1		1.0E+04	Ethyl Ether	60-29-7	1.6E+04	ns	2.3E+05	nms					3.9E+03	n		8.8E-01	n		
				9.0E-02	H	3.0E-01	P	V		1		1.1E+03	Ethyl Methacrylate	97-63-2	1.4E+03	ns	7.1E+03	ns	3.1E+02	n	1.3E+03	n	4.6E+02	n		1.1E-01	n		
				1.0E-05	I					1	0.1		Ethyl-p-nitrophenyl Phosphonate	2104-64-5	6.3E-01	n	8.2E+00	n					8.9E-02	n		2.8E-03	n		
1.1E-02	C	2.5E-06	C	1.0E-01	I	1.0E+00	I	V		1		4.8E+02	Ethylbenzene	100-41-4	5.8E+00	c	2.5E+01	c	1.1E+00	c	4.9E+00	c	1.5E+00	c	7.0E+02	1.7E-03	c	7.8E-01	
				7.0E-02	P					1	0.1		Ethylene Cyanohydrin	109-78-4	4.4E+03	n	5.7E+04	n					1.4E+03	n		2.8E-01	n		
				9.0E-02	P			V		1		1.9E+05	Ethylene Diamine	107-15-3	7.0E+03	n	1.1E+05	nm					1.8E+03	n		4.1E-01	n		
				2.0E+00	I	4.0E-01	C			1	0.1		Ethylene Glycol	107-21-1	1.3E+05	nm	1.6E+06	nm	4.2E+02	n	1.8E+03	n	4.0E+04	n		8.1E+00	n		
				1.0E-01	I	1.6E+00	I			1	0.1		Ethylene Glycol Monobutyl Ether	111-76-2	6.3E+03	n	8.2E+04	n	1.7E+03	n	7.0E+03	n	2.0E+03	n		4.1E-01	n		
3.1E-01	C	8.8E-05	C			3.0E-02	C	V		1		1.2E+05	Ethylene Oxide	75-21-8	1.8E-01	c	7.9E-01	c	3.2E-02	c	1.4E-01	c	5.1E-02	c		1.1E-05	c		
4.5E-02	C	1.3E-05	C	8.0E-05	I					1	0.1		Ethylene Thiourea	96-45-7	5.1E+00	n	5.1E+01	c**	2.2E-01	c	9.4E-01	c	1.6E+00	n		3.6E-04	n		
6.5E+01	C	1.9E-02	C					V		1		1.5E+05	Ethyleneimine	151-56-4	2.7E-03	c	1.2E-02	c	1.5E-04	c	6.5E-04	c	2.4E-04	c		5.2E-08	c		
				3.0E+00	I					1	0.1		Ethylphthalyl Ethyl Glycolate	84-72-0	1.9E+05	nm	2.5E+06	nm					5.8E+04	n		1.3E+02	n		
				8.0E-03	I					1	0.1		Express	101200-48-0	5.1E+02	n	6.6E+03	n					1.6E+02	n		6.1E-02	n		
				2.5E-04	I					1	0.1		Fenamiphos	22224-92-6	1.6E+01	n	2.1E+02	n					4.4E+00	n		4.3E-03	n		
				2.5E-02	I					1	0.1		Fenpropathrin	39515-41-8	1.6E+03	n	2.1E+04	n					6.4E+01	n		2.9E+00	n		
				1.3E-02	I					1	0.1		Fluometuron	2164-17-2	8.2E+02	n	1.1E+04	n					2.4E+02	n		1.9E-01	n		
				4.0E-02	C	1.3E-02	C			1			Fluoride	16984-48-8	3.1E+03	n	4.7E+04	n	1.4E+01	n	5.7E+01	n	8.0E+02	n		1.2E+02	n		
				6.0E-02	I	1.3E-02	C			1			Fluorine (Soluble Fluoride)	7782-41-4	4.7E+03	n	7.0E+04	n	1.4E+01	n	5.7E+01	n	1.2E+03	n	4.0E+03	1.8E+02	n	6.0E+02	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
				8.0E-02	I					1	0.1		Fluridone	59756-60-4	5.1E+03	n	6.6E+04	n					1.4E+03	n		1.6E+02	n	
				2.0E-02	I					1	0.1		Flurprimidol	56425-91-3	1.3E+03	n	1.6E+04	n					3.4E+02	n		1.6E+00	n	
				6.0E-02	I					1	0.1		Flutolanil	66332-96-5	3.8E+03	n	4.9E+04	n					9.5E+02	n		5.0E+00	n	
				1.0E-02	I					1	0.1		Fluvalinate	69409-94-5	6.3E+02	n	8.2E+03	n					2.0E+02	n		2.9E+02	n	
3.5E-03	I			1.0E-01	I					1	0.1		Folpet	133-07-3	1.6E+02	c*	6.6E+02	c					2.0E+01	c*		4.7E-03	c*	
1.9E-01	I									1	0.1		Fomesafen	72178-02-0	2.9E+00	c	1.2E+01	c					3.9E-01	c		1.3E-03	c	
				2.0E-03	I					1	0.1		Fonofos	944-22-9	1.3E+02	n	1.6E+03	n					2.4E+01	n		4.7E-02	n	
		1.3E-05	I	2.0E-01	I	9.8E-03	A	V		1		4.2E+04	Formaldehyde	50-00-0	1.7E+01	c*	7.3E+01	c*	2.2E-01	c*	9.4E-01	c*	4.3E-01	c*		8.7E-05	c*	
				9.0E-01	P	3.0E-04	X	V		1		1.1E+05	Formic Acid	64-18-6	2.9E+01	n	1.2E+02	n	3.1E-01	n	1.3E+00	n	6.3E-01	n		1.3E-04	n	
				3.0E+00	I					1	0.1		Fosetyl-AL	39148-24-8	1.9E+05	nm	2.5E+06	nm					6.0E+04	n			n	
													Furans																
				1.0E-03	X			V		1	0.03		~Dibenzofuran	132-64-9	7.3E+01	n	1.0E+03	n					7.9E+00	n		1.5E-01	n	
				1.0E-03	I			V		1	0.03	6.2E+03	~Furan	110-00-9	7.3E+01	n	1.0E+03	n					1.9E+01	n		7.3E-03	n	
				9.0E-01	I	2.0E+00	I	V		1	0.03	1.7E+05	~Tetrahydrofuran	109-99-9	1.8E+04	n	9.6E+04	n	2.1E+03	n	8.8E+03	n	3.4E+03	n		7.5E-01	n	
3.8E+00	H									1	0.1		Furazolidone	67-45-8	1.4E-01	c	6.0E-01	c					2.0E-02	c		3.9E-05	c	
				3.0E-03	I	5.0E-02	H	V		1		1.0E+04	Furfural	98-01-1	2.1E+02	n	2.6E+03	n	5.2E+01	n	2.2E+02	n	3.8E+01	n		8.1E-03	n	
1.5E+00	C	4.3E-04	C							1	0.1		Furium	531-82-8	3.6E-01	c	1.5E+00	c	6.5E-03	c	2.9E-02	c	5.0E-02	c		6.8E-05	c	
3.0E-02	I	8.6E-06	C							1	0.1		Furmecyclox	60568-05-0	1.8E+01	c	7.7E+01	c	3.3E-01	c	1.4E+00	c	1.1E+00	c		1.2E-03	c	
				4.0E-04	I					1	0.1		Glufosinate, Ammonium	77182-82-2	2.5E+01	n	3.3E+02	n					8.0E+00	n		1.8E-03	n	
						8.0E-05	C			1	0.1		Glutaraldehyde	111-30-8	1.1E+05	nm	4.8E+05	nm	8.3E-02	n	3.5E-01	n						
				4.0E-04	I	1.0E-03	H	V		1		1.1E+05	Glycidyl	765-34-4	2.2E+01	n	1.9E+02	n	1.0E+00	n	4.4E+00	n	1.7E+00	n		3.3E-04	n	
				1.0E-01	I					1	0.1		Glyphosate	1071-83-6	6.3E+03	n	8.2E+04	n					2.0E+03	n	7.0E+02	8.8E+00	n	3.1E+00
				3.0E-03	I					1	0.1		Goal	42874-03-3	1.9E+02	n	2.5E+03	n					3.2E+01	n		2.5E+00	n	
				1.0E-02	X			V		1			Guanidine	113-00-8	7.8E+02	n	1.2E+04	n					2.0E+02	n		4.5E-02	n	
				2.0E-02	P					1	0.1		Guanidine Chloride	50-01-1	1.3E+03	n	1.6E+04	n					4.0E+02	n			n	
				3.0E-03	A	1.0E-02	A			1	0.1		Guthion	86-50-0	1.9E+02	n	2.5E+03	n	1.0E+01	n	4.4E+01	n	5.6E+01	n		1.7E-02	n	
				5.0E-05	I					1	0.1		Haloxyfop, Methyl	69806-40-2	3.2E+00	n	4.1E+01	n					7.6E-01	n		8.4E-03	n	
				1.3E-02	I					1	0.1		Harmony	79277-27-3	8.2E+02	n	1.1E+04	n					2.6E+02	n		7.8E-02	n	
4.5E+00	I	1.3E-03	I	5.0E-04	I			V		1			Heptachlor	76-44-8	1.3E-01	c	6.3E-01	c	2.2E-03	c	9.4E-03	c	1.4E-03	c	4.0E-01	1.1E-04	c	3.3E-02
9.1E+00	I	2.6E-03	I	1.3E-05	I			V		1			Heptachlor Epoxide	1024-57-3	7.0E-02	c*	3.3E-01	c*	1.1E-03	c	4.7E-03	c	1.4E-03	c*	2.0E-01	2.8E-05	c*	4.1E-03
				2.0E-03	I			V		1			Hexabromobenzene	87-82-1	1.6E+02	n	2.3E+03	n					4.0E+01	n		2.3E-01	n	
				2.0E-04	I					1	0.1		Hexabromodiphenyl ether, 2,2',4,4',5,5'- (BDE-153)	68631-49-2	1.3E+01	n	1.6E+02	n					4.0E+00	n			n	
1.6E+00	I	4.6E-04	I	8.0E-04	I			V		1			Hexachlorobenzene	118-74-1	2.1E-01	c	9.6E-01	c	6.1E-03	c	2.7E-02	c	9.8E-03	c	1.0E+00	1.2E-04	c	1.3E-02
7.8E-02	I	2.2E-05	I	1.0E-03	P			V		1		1.7E+01	Hexachlorobutadiene	87-68-3	1.2E+00	c*	5.3E+00	c	1.3E-01	c	5.6E-01	c	1.4E-01	c*		2.6E-04	c*	
6.3E+00	I	1.8E-03	I	8.0E-03	A					1	0.1		Hexachlorocyclohexane, Alpha-	319-84-6	8.6E-02	c	3.6E-01	c	1.6E-03	c	6.8E-03	c	7.1E-03	c		4.1E-05	c	
1.8E+00	I	5.3E-04	I							1	0.1		Hexachlorocyclohexane, Beta-	319-85-7	3.0E-01	c	1.3E+00	c	5.3E-03	c	2.3E-02	c	2.5E-02	c		1.4E-04	c	
1.1E+00	C	3.1E-04	C	3.0E-04	I					1	0.04		Hexachlorocyclohexane, Gamma- (Lindane)	58-89-9	5.7E-01	c*	2.5E+00	c	9.1E-03	c	4.0E-02	c	4.1E-02	c*	2.0E-01	2.4E-04	c*	1.2E-03
1.8E+00	I	5.1E-04	I							1	0.1		Hexachlorocyclohexane, Technical	608-73-1	3.0E-01	c	1.3E+00	c	5.5E-03	c	2.4E-02	c	2.5E-02	c		1.4E-04	c	
				6.0E-03	I	2.0E-04	I	V		1		1.6E+01	Hexachlorocyclopentadiene	77-47-4	1.8E+00	n	7.5E+00	n	2.1E-01	n	8.8E-01	n	4.1E-01	n	5.0E+01	1.3E-03	n	1.6E-01
4.0E-02	I	1.1E-05	C	7.0E-04	I	3.0E-02	I	V		1			Hexachloroethane	67-72-1	1.8E+00	c*	8.0E+00	c*	2.6E-01	c	1.1E+00	c	3.3E-01	c*		2.0E-04	c*	
				3.0E-04	I					1	0.1		Hexachlorophene	70-30-4	1.9E+01	n	2.5E+02	n					6.0E+00	n		8.0E+00	n	
1.1E-01	I			3.0E-03	I					1	0.015		Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	6.1E+00	c*	2.8E+01	c					7.0E-01	c*		2.7E-04	c*	
						1.0E-05	I	V		1		5.2E+03	Hexamethylene Diisocyanate, 1,6-	822-06-0	3.1E+00	n	1.3E+01	n	1.0E-02	n	4.4E-02	n	2.1E-02	n		2.1E-04	n	
				4.0E-04	P					1	0.1		Hexamethylphosphoramide	680-31-9	2.5E+01	n	3.3E+02	n					8.0E+00	n		1.8E-03	n	
				6.0E-02	H	7.0E-01	I	V		1		1.4E+02	Hexane, N-	110-54-3	5.4E+02	ns	2.5E+03	ns	7.3E+02	n	3.1E+03	n	3.2E+02	n		2.3E+00	n	
				2.0E+00	P					1	0.1		Hexanedioic Acid	124-04-9	1.3E+05	nm	1.6E+06	nm					4.0E+04	n		9.9E+00	n	
				5.0E-03	I	3.0E-02	I	V		1		3.3E+03	Hexanone, 2-	591-78-6	2.0E+02	n	1.3E+03	n	3.1E+01	n	1.3E+02	n	3.8E+01	n		8.8E-03	n	
				3.3E-02	I					1	0.1		Hexazinone	51235-04-2	2.1E+03	n	2.7E+04	n					6.4E+02	n		3.0E-01	n	
3.0E+00	I	4.9E-03	I			3.0E-05	P	V		1			Hydrazine	302-01-2	2.3E-01	c	1.1E+00	c	5.7E-04	c*	2.5E-03	c*	1.1E-03	c*			c*	
3.0E+00	I	4.9E-03	I							1			Hydrazine Sulfate	10034-93-2	2.3E-01	c	1.1E+00	c	5.7E-04	c	2.5E-03	c	2.6E-02	c			c	
						2.0E-02	I	V		1			Hydrogen Chloride	7647-01-0	2.8E+07	nm	1.2E+08	nm	2.1E+01	n	8.8E+01	n	4.2E+01	n			n	
				4.0E-02	C	1.4E-02	C	V		1			Hydrogen Fluoride	7664-39-3	3.1E+03	n	4.7E+04	n	1.5E+01	n	6.1E+01	n	2.8E+01	n			n	
						2.0E-03	I	V		1			Hydrogen Sulfide	7783-06-4	2.8E+06	nm	1.2E+07	nm	2.1E+00	n	8.8E+00	n	4.2E+00	n			n	
6.0E-02	P			4.0E-02	P					1	0.1		Hydroquinone	123-31-9	9.0E+00	c	3.8E+01	c					1.3E+00	c		8.7E-04	c	
				1.3E-02	I					1	0.1		Imazalil	35554-44-0	8.2E+02	n	1.1E+04	n					1.9E+02	n		3.2E+00	n	
				2.5E-01	I					1	0.1		Imazaquin	81335-37-7	1.6E+04	n	2.1E+05	nm					4.9E+03	n		2.4E+01	n	
				1.0E-02	A					1			Iodine	7553-56-2	7.8E+02	n	1.2E+04	n					2.0E+02	n		1.2E+01	n	
				4.0E-02	I					1	0.1		Iprodione	36734-19-7	2.5E+03	n	3.3E+04	n					7.4E+02	n		2.2E-01	n	
				7.0E-01	P					1			Iron	7439-89-6	5.5E+04	n	8.2E+05	nm					1.4E+04	n		3.5E+02	n	
				3.0E-01	I			V		1		1.0E+04	Isobutyl Alcohol	78-83-1	2.3E+04	ns	3.5E+05	nms					5.9E+03	n		1.2E+00	n	
9.5E-04	I			2.0E-01	I	2.0E+00	C			1	0.1		Isophorone	78-59-1	5.7E+02	c*	2.4E+03	c*	2.1E+03	n	8.8E+03	n	7.8E+01	c*		2.6E-02	c*	
				1.5E-02	I			V		1			Isopropalin	33820-53-0	1.2E+03	n	1.8E+04	n					4.0E+01	n		9.2E-01	n	
				2.0E+00	P	2.0E-01	P	V		1		1.1E+05	Isopropanol	67-63-0	5.6E+03	n	2.4E+04	n	2.1E+02	n	8.8E+02	n	4.1E+02	n		8.4E-02	n	
				1.0E-01	I					1	0.1		Isopropyl Methyl Phosphonic Acid	1832-54-8	6.3E+03	n	8.2E+04	n					2.0E+03	n		4.3E-01	n	
				5.0E-02	I					1	0.1		Isoxaben	82558-50-7	3.2E+03	n	4.1E+04	n					7.3E+02	n		2.0E+00	n	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information													Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
						3.0E-01	A	V		1			JP-7	NA	4.3E+08	nm	1.8E+09	nm	3.1E+02	n	1.3E+03	n	6.3E+02	n			n	
				7.5E-02	I					1	0.1		Kerb	23950-58-5	4.7E+03	n	6.2E+04	n					1.2E+03	n		1.2E+00	n	
				2.0E-03	I					1	0.1		Lactofen	77501-63-4	1.3E+02	n	1.6E+03	n					2.5E+01	n		1.2E+00	n	
													Lead Compounds															
5.0E-01	C	1.5E-01	C	2.0E-02	C	2.0E-04	C		M	0.025			~Lead Chromate	7758-97-6	3.0E-01	c	6.2E+00	c	6.8E-06	c	8.2E-05	c	4.1E-02	c			c	
8.5E-03	C	1.2E-05	C							1			~Lead Phosphate	7446-27-7	8.2E+01	c	3.8E+02	c	2.3E-01	c	1.0E+00	c	9.1E+00	c			c	
2.8E-01	C	8.0E-05	C							1	0.1		~Lead acetate	301-04-2	1.9E+00	c	8.2E+00	c	3.5E-02	c	1.5E-01	c	2.8E-01	c			c	
										1			~Lead and Compounds	7439-92-1	4.0E+02		8.0E+02	L	1.5E-01	L			1.5E+01	L			L	
8.5E-03	C	1.2E-05	C							1	0.1		~Lead subacetate	1335-32-6	6.4E+01	c	2.7E+02	c	2.3E-01	c	1.0E+00	c	9.2E+00	c			c	
				1.0E-07	I			V		1		2.4E+00	~Tetraethyl Lead	78-00-2	7.8E-03	n	1.2E-01	n					1.3E-03	n		4.7E-06	n	
				2.0E-03	I					1	0.1		Linuron	330-55-2	1.3E+02	n	1.6E+03	n					3.3E+01	n		2.9E-02	n	
				2.0E-03	P					1			Lithium	7439-93-2	1.6E+02	n	2.3E+03	n					4.0E+01	n		1.2E+01	n	
				2.0E-01	I					1	0.1		Londax	83055-99-6	1.3E+04	n	1.6E+05	nm					3.9E+03	n		1.0E+00	n	
				5.0E-04	I					1	0.1		MCPA	94-74-6	3.2E+01	n	4.1E+02	n					7.5E+00	n		2.0E-03	n	
				1.0E-02	I					1	0.1		MCPB	94-81-5	6.3E+02	n	8.2E+03	n					1.5E+02	n		5.8E-02	n	
				1.0E-03	I					1	0.1		MCPP	93-65-2	6.3E+01	n	8.2E+02	n					1.6E+01	n		4.6E-03	n	
				2.0E-02	I					1	0.1		Malathion	121-75-5	1.3E+03	n	1.6E+04	n					3.9E+02	n		1.0E-01	n	
				1.0E-01	I	7.0E-04	C			1	0.1		Maleic Anhydride	108-31-6	6.3E+03	n	8.0E+04	n	7.3E-01	n	3.1E+00	n	1.9E+03	n		3.8E-01	n	
				5.0E-01	I					1	0.1		Maleic Hydrazide	123-33-1	3.2E+04	n	4.1E+05	nm					1.0E+04	n		2.1E+00	n	
				1.0E-04	P					1	0.1		Malononitrile	109-77-3	6.3E+00	n	8.2E+01	n					2.0E+00	n		4.1E-04	n	
				3.0E-02	H					1	0.1		Mancozeb	8018-01-7	1.9E+03	n	2.5E+04	n					5.4E+02	n			n	
				5.0E-03	I					1	0.1		Maneb	12427-38-2	3.2E+02	n	4.1E+03	n					9.8E+01	n		1.4E-01	n	
				1.4E-01	I	5.0E-05	I			1			Manganese (Diet)	7439-96-5														
				2.4E-02	S	5.0E-05	I			0.04			Manganese (Non-diet)	7439-96-5	1.8E+03	n	2.6E+04	n	5.2E-02	n	2.2E-01	n	4.3E+02	n		2.8E+01	n	
				9.0E-05	H					1	0.1		Mephosfolan	950-10-7	5.7E+00	n	7.4E+01	n					1.8E+00	n		2.6E-03	n	
				3.0E-02	I					1	0.1		Mepiquat Chloride	24307-26-4	1.9E+03	n	2.5E+04	n					6.0E+02	n		2.0E-01	n	
													Mercury Compounds															
				3.0E-04	I	3.0E-04	S			0.07			~Mercuric Chloride (and other Mercury salts)	7487-94-7	2.3E+01	n	3.5E+02	n	3.1E-01	n	1.3E+00	n	5.7E+00	n	2.0E+00		n	
						3.0E-04	I	V		1		3.1E+00	~Mercury (elemental)	7439-97-6	9.4E+00	ns	4.0E+01	ns	3.1E-01	n	1.3E+00	n	6.3E-01	n	2.0E+00	3.3E-02	n	1.0E-01
				1.0E-04	I					1			~Methyl Mercury	22967-92-6	7.8E+00	n	1.2E+02	n					2.0E+00	n			n	
				8.0E-05	I					1	0.1		~Phenylmercuric Acetate	62-38-4	5.1E+00	n	6.6E+01	n					1.6E+00	n		5.0E-04	n	
				3.0E-05	I			V		1			Merphos	150-50-5	2.3E+00	n	3.5E+01	n					6.0E-01	n		5.9E-02	n	
				3.0E-05	I					1	0.1		Merphos Oxide	78-48-8	1.9E+00	n	2.5E+01	n					8.5E-02	n		4.2E-04	n	
				6.0E-02	I					1	0.1		Metalaxyl	57837-19-1	3.8E+03	n	4.9E+04	n					1.2E+03	n		3.3E-01	n	
				1.0E-04	I	3.0E-02	P	V		1		4.6E+03	Methacrylonitrile	126-98-7	7.5E+00	n	1.0E+02	n	3.1E+01	n	1.3E+02	n	1.9E+00	n		4.3E-04	n	
				5.0E-05	I					1	0.1		Methamidophos	10265-92-6	3.2E+00	n	4.1E+01	n					1.0E+00	n		2.1E-04	n	
				2.0E+00	I	2.0E+01	I	V		1		1.1E+05	Methanol	67-56-1	1.2E+05	nms	1.2E+06	nms	2.1E+04	n	8.8E+04	n	2.0E+04	n		4.1E+00	n	
				1.0E-03	I					1	0.1		Methidathion	950-37-8	6.3E+01	n	8.2E+02	n					1.9E+01	n		4.7E-03	n	
				2.5E-02	I					1	0.1		Methomyl	16752-77-5	1.6E+03	n	2.1E+04	n					5.0E+02	n		1.1E-01	n	
4.9E-02	C	1.4E-05	C							1	0.1		Methoxy-5-nitroaniline, 2-	99-59-2	1.1E+01	c	4.7E+01	c	2.0E-01	c	8.8E-01	c	1.5E+00	c		5.3E-04	c	
				5.0E-03	I					1	0.1		Methoxychlor	72-43-5	3.2E+02	n	4.1E+03	n					3.7E+01	n	4.0E+01	2.0E+00	n	2.2E+00
				8.0E-03	P	1.0E-03	P	V		1		1.2E+05	Methoxyethanol Acetate, 2-	110-49-6	1.1E+02	n	5.1E+02	n	1.0E+00	n	4.4E+00	n	2.1E+00	n		4.2E-04	n	
				5.0E-03	P	2.0E-02	I	V		1		1.1E+05	Methoxyethanol, 2-	109-86-4	3.3E+02	n	3.5E+03	n	2.1E+01	n	8.8E+01	n	2.9E+01	n		5.9E-03	n	
				1.0E+00	X			V		1		2.9E+04	Methyl Acetate	79-20-9	7.8E+04	ns	1.2E+06	nms					2.0E+04	n		4.1E+00	n	
				3.0E-02	H	2.0E-02	P	V		1		6.8E+03	Methyl Acrylate	96-33-3	1.4E+02	n	6.0E+02	n	2.1E+01	n	8.8E+01	n	3.9E+01	n		8.3E-03	n	
				6.0E-01	I	5.0E+00	I	V		1		2.8E+04	Methyl Ethyl Ketone (2-Butanone)	78-93-3	2.7E+04	n	1.9E+05	nms	5.2E+03	n	2.2E+04	n	5.6E+03	n		1.2E+00	n	
		1.0E-03	X	1.0E-03	P	2.0E-05	X	V		1		1.8E+05	Methyl Hydrazine	60-34-4	4.4E-01	c**	1.9E+00	c**	2.8E-03	c**	1.2E-02	c**	5.6E-03	c**		1.3E-06	c**	
				8.0E-02	H	3.0E+00	I	V		1		3.4E+03	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	108-10-1	5.3E+03	ns	5.6E+04	ns	3.1E+03	n	1.3E+04	n	1.2E+03	n		2.8E-01	n	
						1.0E-03	C	V		1		1.7E+04	Methyl Isocyanate	624-83-9	4.6E+00	n	1.9E+01	n	1.0E+00	n	4.4E+00	n	2.1E+00	n		5.9E-04	n	
				1.4E+00	I	7.0E-01	I	V		1		2.4E+03	Methyl Methacrylate	80-62-6	4.4E+03	ns	1.9E+04	ns	7.3E+02	n	3.1E+03	n	1.4E+03	n		3.0E-01	n	
				2.5E-04	I					1	0.1		Methyl Parathion	298-00-0	1.6E+01	n	2.1E+02	n					4.5E+00	n		7.4E-03	n	
				6.0E-02	X					1	0.1		Methyl Phosphonic Acid	993-13-5	3.8E+03	n	4.9E+04	n					1.2E+03	n		2.4E-01	n	
				6.0E-03	H	4.0E-02	H	V		1		3.9E+02	Methyl Styrene (Mixed Isomers)	25013-15-4	2.4E+02	n	1.6E+03	ns	4.2E+01	n	1.8E+02	n	3.8E+01	n		6.2E-02	n	
9.9E-02	C	2.8E-05	C							1	0.1		Methyl methanesulfonate	66-27-3	5.5E+00	c	2.3E+01	c	1.0E-01	c	4.4E-01	c	7.9E-01	c		1.6E-04	c	
1.8E-03	C	2.6E-07	C			3.0E+00	I	V		1		8.9E+03	Methyl tert-Butyl Ether (MTBE)	1634-04-4	4.7E+01	c	2.1E+02	c	1.1E+01	c	4.7E+01	c	1.4E+01	c		3.2E-03	c	
				3.0E-04	X					1	0.1		Methyl-1,4-benzenediamine dihydrochloride, 2-	615-45-2	1.9E+01	n	2.5E+02	n					6.0E+00	n		3.6E-03	n	
9.0E-03	P			2.0E-02	X					1	0.1		Methyl-5-Nitroaniline, 2-	99-55-8	6.0E+01	c*	2.6E+02	c*					8.1E+00	c*		4.5E-03	c*	
8.3E+00	C	2.4E-03	C							1	0.1		Methyl-N-nitro-N-nitrosoguanidine, N-	70-25-7	6.5E-02	c	2.8E-01	c	1.2E-03	c	5.1E-03	c	9.4E-03	c		3.2E-06	c	
1.3E-01	C	3.7E-05	C							1	0.1		Methylaniline Hydrochloride, 2-	636-21-5	4.2E+00	c	1.8E+01	c	7.6E-02	c	3.3E-01	c	6.0E-01	c		2.6E-04	c	
				1.0E-02	A					1	0.1		Methylarsonic acid	124-58-3	6.3E+02	n	8.2E+03	n					2.0E+02	n			n	
				2.0E-04	X					1	0.1		Methylbenzene,1-4-diamine monohydrochloride, 2-	74612-12-7	1.3E+01	n	1.6E+02	n					4.0E+00	n			n	
1.0E-01	X			3.0E-04	X					1	0.1		Methylbenzene-1,4-diamine sulfate, 2-	615-50-9	5.4E+00	c**	2.3E+01	c*					7.8E-01	c**			c**	
2.2E+01	C	6.3E-03	C						M	1	0.1		Methylcholanthrene, 3-	56-49-5	5.5E-03	c	1.0E-01	c	1.6E-04	c	1.9E-03	c	1.1E-03	c		2.2E-03	c	
2.0E-03	I	1.0E-08	I	6.0E-03	I	6.0E-01	I	V	M	1		3.3E+03	Methylene Chloride	75-09-2	5.7E+01	c**	1.0E+03	c**	1.0E+02	c**	1.2E+03	c**	1.1E+01	c**	5.0E+00	2.9E-03	c**	1.3E-03
1.0E-01	P	4.3E-04	C	2.0E-03	P				M	1	0.1		Methylene-bis(2-chloroaniline), 4,4'-	101-14-4	1.2E+00	c	2.3E+01	c*	2.4E-03	c	2.9E-02	c	1.6E-01	c		1.8E-03	c	

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Toxicity and Chemical-specific Information													Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
4.6E-02	I	1.3E-05	C							1	0.1		Methylene-bis(N,N-dimethyl) Aniline, 4,4'-	101-61-1	1.2E+01	c	5.0E+01	c	2.2E-01	c	9.4E-01	c	4.6E-01	c		2.6E-03	c	
1.6E+00	C	4.6E-04	C			2.0E-02	C			1	0.1		Methylenebisbenzenamine, 4,4'-	101-77-9	3.4E-01	c	1.4E+00	c	6.1E-03	c	2.7E-02	c	4.7E-02	c		2.1E-04	c	
						6.0E-04	I			1	0.1		Methylenediphenyl Diisocyanate	101-68-8	8.5E+05	nm	3.6E+06	nm	6.3E-01	n	2.6E+00	n						
				7.0E-02	H			V		1		5.0E+02	Methylstyrene, Alpha-	98-83-9	5.5E+03	ns	8.2E+04	ns					7.8E+02	n		1.2E+00	n	
				1.5E-01	I					1	0.1		Metolachlor	51218-45-2	9.5E+03	n	1.2E+05	nm					2.7E+03	n		3.2E+00	n	
				2.5E-02	I					1	0.1		Metribuzin	21087-64-9	1.6E+03	n	2.1E+04	n					4.9E+02	n		1.5E-01	n	
				3.0E+00	P			V		1		3.4E-01	Mineral oils	8012-95-1	2.3E+05	nms	3.5E+06	nms					6.0E+04	n		2.4E+03	n	
1.8E+01	C	5.1E-03	C	2.0E-04	I			V		1			Mirex	2385-85-5	3.6E-02	c	1.7E-01	c	5.5E-04	c	2.4E-03	c	8.8E-04	c		6.3E-04	c	
				2.0E-03	I					1	0.1		Molinate	2212-67-1	1.3E+02	n	1.6E+03	n					3.0E+01	n		1.7E-02	n	
				5.0E-03	I					1			Molybdenum	7439-98-7	3.9E+02	n	5.8E+03	n					1.0E+02	n		2.0E+00	n	
				1.0E-01	I					1			Monochloramine	10599-90-3	7.8E+03	n	1.2E+05	nm					2.0E+03	n	4.0E+03		n	
				2.0E-03	P					1	0.1		Monomethylaniline	100-61-8	1.3E+02	n	1.6E+03	n					3.8E+01	n		1.4E-02	n	
				3.0E-04	X					1	0.1		N,N'-Diphenyl-1,4-benzenediamine	74-31-7	1.9E+01	n	2.5E+02	n					3.6E+00	n		3.7E-01	n	
				2.0E-03	I			V		1			Naled	300-76-5	1.6E+02	n	2.3E+03	n					4.0E+01	n		1.8E-02	n	
				3.0E-02	X	1.0E-01	P	V		1			Naphtha, High Flash Aromatic (HFAN)	64742-95-6	2.3E+03	n	3.5E+04	n	1.0E+02	n	4.4E+02	n	1.5E+02	n			n	
1.8E+00	C	0.0E+00	C							1	0.1		Naphthylamine, 2-	91-59-8	3.0E-01	c	1.3E+00	c					3.9E-02	c		2.0E-04	c	
				1.0E-01	I					1	0.1		Napropamide	15299-99-7	6.3E+03	n	8.2E+04	n					1.6E+03	n		1.1E+01	n	
		2.6E-04	C	1.1E-02	C	1.4E-05	C			1	0.1		Nickel Acetate	373-02-4	6.7E+02	n	8.1E+03	n	1.1E-02	c**	4.7E-02	c**	2.2E+02	n			n	
		2.6E-04	C	1.1E-02	C	1.4E-05	C			1	0.1		Nickel Carbonate	3333-67-3	6.7E+02	n	8.1E+03	n	1.1E-02	c**	4.7E-02	c**	2.2E+02	n			n	
		2.6E-04	C	1.1E-02	C	1.4E-05	C	V		1			Nickel Carbonyl	13463-39-3	8.2E+02	n	1.1E+04	n	1.1E-02	c**	4.7E-02	c**	2.2E-02	c**			c**	
		2.6E-04	C	1.1E-02	C	1.4E-05	C			0.04			Nickel Hydroxide	12054-48-7	8.2E+02	n	1.1E+04	n	1.1E-02	c**	4.7E-02	c**	2.0E+02	n			n	
		2.6E-04	C	1.1E-02	C	2.0E-05	C			0.04			Nickel Oxide	1313-99-1	8.4E+02	n	1.2E+04	n	1.1E-02	c**	4.7E-02	c**	2.0E+02	n			n	
		2.4E-04	I	1.1E-02	C	1.4E-05	C			0.04			Nickel Refinery Dust	NA	8.2E+02	n	1.1E+04	n	1.2E-02	c**	5.1E-02	c**	2.2E+02	n		3.2E+01	n	
		2.6E-04	C	2.0E-02	I	9.0E-05	A			0.04			Nickel Soluble Salts	7440-02-0	1.5E+03	n	2.2E+04	n	1.1E-02	c**	4.7E-02	c**	3.9E+02	n		2.6E+01	n	
1.7E+00	C	4.8E-04	I	1.1E-02	C	1.4E-05	C			0.04			Nickel Subsulfide	12035-72-2	4.1E-01	c	1.9E+00	c	5.8E-03	c**	2.6E-02	c**	4.5E-02	c			c	
		2.6E-04	C	1.1E-02	C	1.4E-05	C			1	0.1		Nickelocene	1271-28-9	6.7E+02	n	8.1E+03	n	1.1E-02	c**	4.7E-02	c**	2.2E+02	n			n	
				1.6E+00	I					1			Nitrate	14797-55-8	1.3E+05	nm	1.9E+06	nm					3.2E+04	n	1.0E+04		n	
										1			Nitrate + Nitrite (as N)	NA											1.0E+04			
				1.0E-01	I					1			Nitrite	14797-65-0	7.8E+03	n	1.2E+05	nm					2.0E+03	n	1.0E+03		n	
				1.0E-02	X	5.0E-05	X			1	0.1		Nitroaniline, 2-	88-74-4	6.3E+02	n	8.0E+03	n	5.2E-02	n	2.2E-01	n	1.9E+02	n		8.0E-02	n	
2.0E-02	P			4.0E-03	P	6.0E-03	P			1	0.1		Nitroaniline, 4-	100-01-6	2.7E+01	c**	1.1E+02	c*	6.3E+00	n	2.6E+01	n	3.8E+00	c*		1.6E-03	c*	
		4.0E-05	I	2.0E-03	I	9.0E-03	I	V		1		3.1E+03	Nitrobenzene	98-95-3	5.1E+00	c*	2.2E+01	c*	7.0E-02	c	3.1E-01	c	1.4E-01	c*		9.2E-05	c*	
				3.0E+03	P					1	0.1		Nitrocellulose	9004-70-0	1.9E+08	nm	2.5E+09	nm					6.0E+07	n		1.3E+04	n	
				7.0E-02	H					1	0.1		Nitrofurantoin	67-20-9	4.4E+03	n	5.7E+04	n					1.4E+03	n		6.1E-01	n	
1.3E+00	C	3.7E-04	C							1	0.1		Nitrofurazone	59-87-0	4.2E-01	c	1.8E+00	c	7.6E-03	c	3.3E-02	c	6.0E-02	c		5.4E-05	c	
1.7E-02	P			1.0E-04	P					1	0.1		Nitroglycerin	55-63-0	6.3E+00	n	8.2E+01	n					2.0E+00	n		8.5E-04	n	
				1.0E-01	I					1	0.1		Nitroguanidine	556-88-7	6.3E+03	n	8.2E+04	n					2.0E+03	n		4.8E-01	n	
		8.8E-06	P			5.0E-03	P	V		1		1.8E+04	Nitromethane	75-52-5	5.4E+00	c*	2.4E+01	c*	3.2E-01	c*	1.4E+00	c*	6.4E-01	c*		1.4E-04	c*	
		2.7E-03	H			2.0E-02	I	V		1		4.9E+03	Nitropropane, 2-	79-46-9	1.4E-02	c	6.0E-02	c	1.0E-03	c	4.5E-03	c	2.1E-03	c		5.4E-07	c	
2.7E+01	C	7.7E-03	C						M	1	0.1		Nitroso-N-ethylurea, N-	759-73-9	4.5E-03	c	8.5E-02	c	1.3E-04	c	1.6E-03	c	9.2E-04	c		2.2E-07	c	
1.2E+02	C	3.4E-02	C						M	1	0.1		Nitroso-N-methylurea, N-	684-93-5	1.0E-03	c	1.9E-02	c	3.0E-05	c	3.6E-04	c	2.1E-04	c		4.6E-08	c	
5.4E+00	I	1.6E-03	I					V		1			Nitroso-di-N-butylamine, N-	924-16-3	9.9E-02	c	4.6E-01	c	1.8E-03	c	7.7E-03	c	2.7E-03	c		5.5E-06	c	
7.0E+00	I	2.0E-03	C							1	0.1		Nitroso-di-N-propylamine, N-	621-64-7	7.8E-02	c	3.3E-01	c	1.4E-03	c	6.1E-03	c	1.1E-02	c		8.1E-06	c	
2.8E+00	I	8.0E-04	C							1	0.1		Nitrosodiethanolamine, N-	1116-54-7	1.9E-01	c	8.2E-01	c	3.5E-03	c	1.5E-02	c	2.8E-02	c		5.6E-06	c	
1.5E+02	I	4.3E-02	I						M	1	0.1		Nitrosodiethylamine, N-	*55-18-5*	*8.1E-04*	c	1.5E-02	c	2.4E-05	c	2.9E-04	c	1.7E-04	c		6.0E-08	c	
5.1E+01	I	1.4E-02	I	8.0E-06	P	4.0E-05	X	V	M	1		2.4E+05	Nitrosodimethylamine, N-	62-75-9	2.0E-03	c	3.4E-02	c	7.2E-05	c	8.8E-04	c	1.1E-04	c		2.8E-08	c	
4.9E-03	I	2.6E-06	C							1	0.1		Nitrosodiphenylamine, N-	86-30-6	1.1E+02	c	4.7E+02	c	1.1E+00	c	4.7E+00	c	1.2E+01	c		6.6E-02	c	
2.2E+01	I	6.3E-03	C					V		1		1.1E+05	Nitrosomethylethylamine, N-	10595-95-6	2.0E-02	c	9.1E-02	c	4.5E-04	c	1.9E-03	c	7.1E-04	c		2.0E-07	c	
6.7E+00	C	1.9E-03	C							1	0.1		Nitrosomorpholine [N-]	59-89-2	8.1E-02	c	3.4E-01	c	1.5E-03	c	6.5E-03	c	1.2E-02	c		2.8E-06	c	
9.4E+00	C	2.7E-03	C							1	0.1		Nitrosopiperidine [N-]	100-75-4	5.8E-02	c	2.4E-01	c	1.0E-03	c	4.5E-03	c	8.2E-03	c		4.4E-06	c	
2.1E+00	I	6.1E-04	I							1	0.1		Nitrosopyrrolidine, N-	930-55-2	2.6E-01	c	1.1E+00	c	4.6E-03	c	2.0E-02	c	3.7E-02	c		1.4E-05	c	
				1.0E-04	X					1	0.1		Nitrotoluene, m-	99-08-1	6.3E+00	n	8.2E+01	n					1.7E+00	n		1.6E-03	n	
2.2E-01	P			9.0E-04	P			V		1		1.5E+03	Nitrotoluene, o-	88-72-2	3.2E+00	c*	1.5E+01	c*					3.1E-01	c*		2.9E-04	c*	
1.6E-02	P			4.0E-03	P					1	0.1		Nitrotoluene, p-	99-99-0	3.4E+01	c**	1.4E+02	c*					4.2E+00	c*		3.9E-03	c*	
				3.0E-04	X	2.0E-02	P	V		1		6.9E+00	Nonane, n-	111-84-2	1.1E+01	ns	7.2E+01	ns	2.1E+01	n	8.8E+01	n	5.3E+00	n		7.5E-02	n	
				4.0E-02	I					1	0.1		Norflurazon	27314-13-2	2.5E+03	n	3.3E+04	n					7.7E+02	n		5.0E+00	n	
				7.0E-04	I					1	0.1		Nustar	85509-19-9	4.4E+01	n	5.7E+02	n					1.1E+01	n		1.8E+00	n	
				3.0E-03	I					1	0.1		Octabromodiphenyl Ether	32536-52-0	1.9E+02	n	2.5E+03	n					6.0E+01	n		1.2E+01	n	
				5.0E-02	I					1	0.006		Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	3.9E+03	n	5.7E+04	n					1.0E+03	n		1.3E+00	n	
				2.0E-03	H					1	0.1		Octamethylpyrophosphoramide	152-16-9	1.3E+02	n	1.6E+03	n					4.0E+01	n		9.6E-03	n	
				5.0E-02	I					1	0.1		Oryzalin	19044-88-3	3.2E+03	n	4.1E+04	n					8.1E+02	n		1.5E+00	n	
				5.0E-03	I					1	0.1		Oxadiazon	19666-30-9	3.2E+02	n	4.1E+03	n					4.7E+01	n		4.8E-01	n	
				2.5E-02	I					1	0.1		Oxamyl	23135-22-0	1.6E+03	n	2.1E+04	n					5.0E+02	n	2.0E+02	1.1E-01	n	4.4E-02
				1.3E-02	I					1	0.1		Paclobutrazol	76738-62-0	8.2E+02	n	1.1E+04	n					2.3E+02	n		4.6E-01	n	
				4.5E-03	I					1	0.1		Paraquat Dichloride	1910-42-5	2.8E+02	n	3.7E+03	n					9.0E+01	n		1.2E+00	n	

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Toxicity and Chemical-specific Information												Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	voc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
				6.0E-03	H					1	0.1		Parathion	56-38-2	3.8E+02	n	4.9E+03	n					8.6E+01	n		4.3E-01	n	
				5.0E-02	H			V		1			Pebulate	1114-71-2	3.9E+03	n	5.8E+04	n					5.6E+02	n		4.5E-01	n	
				4.0E-02	I					1	0.1		Pendimethalin	40487-42-1	2.5E+03	n	3.3E+04	n					1.8E+02	n		2.1E+00	n	
				2.0E-03	I					1	0.1		Pentabromodiphenyl Ether	32534-81-9	1.3E+02	n	1.6E+03	n					4.0E+01	n		1.7E+00	n	
				1.0E-04	I					1	0.1		Pentabromodiphenyl ether, 2,2',4,4',5- (BDE-99)	60348-60-9	6.3E+00	n	8.2E+01	n					2.0E+00	n		8.7E-02	n	
				8.0E-04	I			V		1			Pentachlorobenzene	608-93-5	6.3E+01	n	9.3E+02	n					3.2E+00	n		2.4E-02	n	
9.0E-02	P							V		1		4.5E+02	Pentachloroethane	76-01-7	7.7E+00	c	3.6E+01	c					6.4E-01	c		3.1E-04	c	
2.6E-01	H			3.0E-03	I			V		1			Pentachloronitrobenzene	82-68-8	2.7E+00	c*	1.3E+01	c					1.2E-01	c		1.4E-03	c	
4.0E-01	I	5.1E-06	C	5.0E-03	I					1	0.25		Pentachlorophenol	87-86-5	1.0E+00	c	4.0E+00	c	5.5E-01	c	2.4E+00	c	4.0E-02	c	1.0E+00	4.0E-04	c	1.0E-02
4.0E-03	X			2.0E-03	P					1	0.1		Pentaerythritol tetranitrate (PETN)	78-11-5	1.3E+02	n	5.7E+02	c**					1.9E+01	c**		2.8E-02	c**	
						1.0E+00	P	V		1		3.9E+02	Pentane, n-	109-66-0	8.1E+02	ns	3.4E+03	ns	1.0E+03	n	4.4E+03	n	2.1E+03	n		1.0E+01	n	
													Perchlorates															
				7.0E-04	I					1			~Ammonium Perchlorate	7790-98-9	5.5E+01	n	8.2E+02	n					1.4E+01	n			n	
				7.0E-04	I					1			~Lithium Perchlorate	7791-03-9	5.5E+01	n	8.2E+02	n					1.4E+01	n			n	
				7.0E-04	I					1			~Perchlorate and Perchlorate Salts	14797-73-0	5.5E+01	n	8.2E+02	n					1.4E+01	n	1.5E+01(F)		n	
				7.0E-04	I					1			~Potassium Perchlorate	7778-74-7	5.5E+01	n	8.2E+02	n					1.4E+01	n			n	
				7.0E-04	I					1			~Sodium Perchlorate	7601-89-0	5.5E+01	n	8.2E+02	n					1.4E+01	n			n	
				2.0E-02	P			V		1			Perfluorobutane Sulfonate	375-73-5	1.6E+03	n	2.3E+04	n					3.8E+02	n		2.1E-01	n	
				5.0E-02	I					1	0.1		Permethrin	52645-53-1	3.2E+03	n	4.1E+04	n					1.0E+03	n		2.4E+02	n	
2.2E-03	C	6.3E-07	C							1	0.1		Phenacetin	62-44-2	2.5E+02	c	1.0E+03	c	4.5E+00	c	1.9E+01	c	3.4E+01	c		9.7E-03	c	
				2.5E-01	I					1	0.1		Phenmedipham	13684-63-4	1.6E+04	n	2.1E+05	nm					4.0E+03	n		2.1E+01	n	
				3.0E-01	I	2.0E-01	C			1	0.1		Phenol	108-95-2	1.9E+04	n	2.5E+05	nm	2.1E+02	n	8.8E+02	n	5.8E+03	n		3.3E+00	n	
				5.0E-04	X					1	0.1		Phenothrazine	92-84-2	3.2E+01	n	4.1E+02	n					4.3E+00	n		1.4E-02	n	
				6.0E-03	I					1	0.1		Phenylenediamine, m-	108-45-2	3.8E+02	n	4.9E+03	n					1.2E+02	n		3.2E-02	n	
4.7E-02	H									1	0.1		Phenylenediamine, o-	95-54-5	1.2E+01	c	4.9E+01	c					1.6E+00	c		4.4E-04	c	
				1.9E-01	H					1	0.1		Phenylenediamine, p-	106-50-3	1.2E+04	n	1.6E+05	nm					3.8E+03	n		1.0E+00	n	
1.9E-03	H									1	0.1		Phenylphenol, 2-	90-43-7	2.8E+02	c	1.2E+03	c					3.0E+01	c		4.0E-01	c	
				2.0E-04	H					1	0.1		Phorate	298-02-2	1.3E+01	n	1.6E+02	n					3.0E+00	n		3.4E-03	n	
						3.0E-04	I	V		1		1.6E+03	Phosgene	75-44-5	3.1E-01	n	1.3E+00	n	3.1E-01	n	1.3E+00	n						
				2.0E-02	I					1	0.1		Phosmet	732-11-6	1.3E+03	n	1.6E+04	n					3.7E+02	n		8.2E-02	n	
													Phosphates, Inorganic															
				4.9E+01	P					1			~Aluminum metaphosphate	13776-88-0	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Ammonium polyphosphate	68333-79-9	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Calcium pyrophosphate	7790-76-3	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Diammonium phosphate	7783-28-0	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Dicalcium phosphate	7757-93-9	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Dimagnesium phosphate	7782-75-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Dipotassium phosphate	7758-11-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Disodium phosphate	7558-79-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monoaluminum phosphate	13530-50-2	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monoammonium phosphate	7722-76-1	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monocalcium phosphate	7758-23-8	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monomagnesium phosphate	7757-86-0	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monopotassium phosphate	7778-77-0	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Monosodium phosphate	7558-80-7	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Polyphosphoric acid	8017-16-1	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Potassium tripolyphosphate	13845-36-8	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium acid pyrophosphate	7758-16-9	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium aluminum phosphate (acidic)	7785-88-8	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium aluminum phosphate (anhydrous)	10279-59-1	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium aluminum phosphate (tetrahydrate)	10305-76-7	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium hexametaphosphate	10124-56-8	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium polyphosphate	68915-31-1	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium trimetaphosphate	7785-84-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Sodium tripolyphosphate	7758-29-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Tetrapotassium phosphate	7320-34-5	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Tetrasodium pyrophosphate	7722-88-5	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Trialuminum sodium tetra decahydrogenoctaorthophosphate (dihydrate)	15136-87-5	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Tricalcium phosphate	7758-87-4	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Trimagnesium phosphate	7757-87-1	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Tripotassium phosphate	7778-53-2	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				4.9E+01	P					1			~Trisodium phosphate	7601-54-9	3.8E+06	nm	5.7E+07	nm					9.7E+05	n			n	
				3.0E-04	I	3.0E-04	I	V		1			Phosphine	7803-51-2	2.3E+01	n	3.5E+02	n	3.1E-01	n	1.3E+00	n	5.7E-01	n			n	
				4.9E+01	P	1.0E-02	I			1			Phosphoric Acid	7664-38-2	3.0E+06	nm	2.9E+07	nm	1.0E+01	n	4.4E+01	n	9.7E+05	n			n	
				2.0E-05	I			V		1			Phosphorus, White	7723-14-0	1.6E+00	n	2.3E+01	n					4.0E-01	n		1.5E-03	n	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volatile	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
													Phthalates															
1.4E-02	I	2.4E-06	C	2.0E-02	I					1	0.1		~Bis(2-ethylhexyl)phthalate	117-81-7	3.9E+01	c*	1.6E+02	c	1.2E+00	c	5.1E+00	c	5.6E+00	c*	6.0E+00	1.3E+00	c*	1.4E+00
				1.0E+00	I					1	0.1		~Butylphthalyl Butylglycolate	85-70-1	6.3E+04	n	8.2E+05	nm					1.3E+04	n		3.0E+02	n	
				1.0E-01	I					1	0.1		~Dibutyl Phthalate	84-74-2	6.3E+03	n	8.2E+04	n					9.0E+02	n		2.3E+00	n	
				8.0E-01	I					1	0.1		~Diethyl Phthalate	84-66-2	5.1E+04	n	6.6E+05	nm					1.5E+04	n		6.1E+00	n	
				1.0E-01	I			V		1			~Dimethylterephthalate	120-61-6	7.8E+03	n	1.2E+05	nm					1.9E+03	n		4.9E-01	n	
				1.0E-02	P					1	0.1		~Octyl Phthalate, di-N-	117-84-0	6.3E+02	n	8.2E+03	n					2.0E+02	n		5.7E+01	n	
				1.0E+00	H					1	0.1		~Phthalic Acid, P-	100-21-0	6.3E+04	n	8.2E+05	nm					1.9E+04	n		6.8E+00	n	
				2.0E+00	I	2.0E-02	C			1	0.1		~Phthalic Anhydride	85-44-9	1.3E+05	nm	1.6E+06	nm	2.1E+01	n	8.8E+01	n	3.9E+04	n		8.5E+00	n	
				7.0E-02	I					1	0.1		Picloram	1918-02-1	4.4E+03	n	5.7E+04	n					1.4E+03	n	5.0E+02	3.8E-01	n	1.4E-01
				1.0E-04	X					1	0.1		Picramic Acid (2-Amino-4,6-dinitrophenol)	96-91-3	6.3E+00	n	8.2E+01	n					2.0E+00	n		1.3E-03	n	
				1.0E-02	I					1	0.1		Pirimiphos, Methyl	29232-93-7	6.3E+02	n	8.2E+03	n					1.2E+02	n		1.2E-01	n	
3.0E+01	C	8.6E-03	C	7.0E-06	H					1	0.1		Polybrominated Biphenyls	59536-65-1	1.8E-02	c*	7.7E-02	c*	3.3E-04	c	1.4E-03	c	2.6E-03	c*			c*	
													Polychlorinated Biphenyls (PCBs)															
7.0E-02	S	2.0E-05	S	7.0E-05	I			V		1	0.14		~Aroclor 1016	12674-11-2	4.1E+00	n	2.7E+01	c**	1.4E-01	c	6.1E-01	c	2.2E-01	c**		2.1E-02	c**	
2.0E+00	S	5.7E-04	S					V		1	0.14		~Aroclor 1221	11104-28-2	1.7E-01	c	7.2E-01	c	4.9E-03	c	2.1E-02	c	4.6E-03	c		7.9E-05	c	
2.0E+00	S	5.7E-04	S					V		1	0.14		~Aroclor 1232	11141-16-5	1.7E-01	c	7.2E-01	c	4.9E-03	c	2.1E-02	c	4.6E-03	c		7.9E-05	c	
2.0E+00	S	5.7E-04	S					V		1	0.14		~Aroclor 1242	53469-21-9	2.3E-01	c	9.7E-01	c	4.9E-03	c	2.1E-02	c	7.8E-03	c		1.2E-03	c	
2.0E+00	S	5.7E-04	S					V		1	0.14		~Aroclor 1248	12672-29-6	2.3E-01	c	9.4E-01	c	4.9E-03	c	2.1E-02	c	7.8E-03	c		1.2E-03	c	
2.0E+00	S	5.7E-04	S	2.0E-05	I			V		1	0.14		~Aroclor 1254	11097-69-1	2.4E-01	c**	9.7E-01	c*	4.9E-03	c	2.1E-02	c	7.8E-03	c*		2.0E-03	c*	
2.0E+00	S	5.7E-04	S					V		1	0.14		~Aroclor 1260	11096-82-5	2.4E-01	c	9.9E-01	c	4.9E-03	c	2.1E-02	c	7.8E-03	c		5.5E-03	c	
				6.0E-04	X			V		1	0.14		~Aroclor 5460	11126-42-4	3.5E+01	n	4.4E+02	n					1.2E+01	n		2.0E+00	n	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Heptachlorobiphenyl, 2,3,3',4,4',5,5'- (PCB 189)	39635-31-9	1.2E-01	c*	5.1E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		2.8E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Hexachlorobiphenyl, 2,3',4,4',5,5'- (PCB 167)	52663-72-6	1.2E-01	c*	5.1E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.7E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Hexachlorobiphenyl, 2,3,3',4,4',5'- (PCB 157)	69782-90-7	1.2E-01	c*	5.1E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.7E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Hexachlorobiphenyl, 2,3,3',4,4',5- (PCB 156)	38380-08-4	1.2E-01	c*	5.1E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.7E-03	c	
3.9E+00	E	1.1E+00	E	2.3E-08	E	1.3E-06	E	V		1	0.14		~Hexachlorobiphenyl, 3,3',4,4',5,5'- (PCB 169)	32774-16-6	1.2E-04	c*	5.1E-04	c*	2.5E-06	c	1.1E-05	c	4.0E-06	c		1.7E-06	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Pentachlorobiphenyl, 2',3,4,4',5- (PCB 123)	65510-44-3	1.2E-01	c*	5.0E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.0E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Pentachlorobiphenyl, 2,3',4,4',5- (PCB 118)	31508-00-6	1.2E-01	c*	5.0E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.0E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Pentachlorobiphenyl, 2,3,3',4,4'- (PCB 105)	32598-14-4	1.2E-01	c*	5.0E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.0E-03	c	
3.9E+00	E	1.1E-03	E	2.3E-05	E	1.3E-03	E	V		1	0.14		~Pentachlorobiphenyl, 2,3,4,4',5- (PCB 114)	74472-37-0	1.2E-01	c*	5.0E-01	c*	2.5E-03	c	1.1E-02	c	4.0E-03	c		1.0E-03	c	
1.3E+04	E	3.8E+00	E	7.0E-09	E	4.0E-07	E	V		1	0.14		~Pentachlorobiphenyl, 3,3',4,4',5- (PCB 126)	57465-28-8	3.7E-05	c*	1.5E-04	c*	7.4E-07	c	3.2E-06	c	1.2E-06	c		3.0E-07	c	
2.0E+00	I	5.7E-04	I					V		1	0.14		~Polychlorinated Biphenyls (high risk)	1336-36-3	2.3E-01	c	9.7E-01	c	4.9E-03	c	2.1E-02	c						
4.0E-01	I	1.0E-04	I					V		1	0.14		~Polychlorinated Biphenyls (low risk)	1336-36-3					2.8E-02	c	1.2E-01	c	4.4E-02	c	5.0E-01	6.8E-03	c	7.8E-02
7.0E-02	I	2.0E-05	I					V		1	0.14		~Polychlorinated Biphenyls (lowest risk)	1336-36-3					1.4E-01	c	6.1E-01	c						
1.3E+01	E	3.8E-03	E	7.0E-06	E	4.0E-04	E			1	0.14		~Tetrachlorobiphenyl, 3,3',4,4'- (PCB 77)	32598-13-3	3.8E-02	c*	1.6E-01	c*	7.4E-04	c	3.2E-03	c	6.0E-03	c*		9.4E-04	c*	
3.9E+01	E	1.1E-02	E	2.3E-06	E	1.3E-04	E	V		1	0.14		~Tetrachlorobiphenyl, 3,4,4',5- (PCB 81)	70362-50-4	1.2E-02	c*	4.9E-02	c*	2.5E-04	c	1.1E-03	c	4.0E-04	c		6.2E-05	c	
						6.0E-04	I			1	0.1		Polymeric Methylene Diphenyl Diisocyanate (PMDI)	9016-87-9	8.5E+05	nm	3.6E+06	nm	6.3E-01	n	2.6E+00	n						
													Polynuclear Aromatic Hydrocarbons (PAHs)															
				6.0E-02	I			V		1	0.13		~Acenaphthene	83-32-9	3.6E+03	n	4.5E+04	n					5.3E+02	n		5.5E+00	n	
				3.0E-01	I			V		1	0.13		~Anthracene	120-12-7	1.8E+04	n	2.3E+05	nm					1.8E+03	n		5.8E+01	n	
7.3E-01	E	1.1E-04	C					V	M	1	0.13		~Benz[a]anthracene	56-55-3	1.6E-01	c	2.9E+00	c	9.2E-03	c	1.1E-01	c	1.2E-02	c		4.3E-03	c	
1.2E+00	C	1.1E-04	C							1	0.13		~Benzo(j)fluoranthene	205-82-3	4.2E-01	c	1.8E+00	c	2.6E-02	c	1.1E-01	c	6.5E-02	c		7.8E-02	c	
7.3E+00	I	1.1E-03	C						M	1	0.13		~Benzo[a]pyrene	50-32-8	1.6E-02	c	2.9E-01	c	9.2E-04	c	1.1E-02	c	3.4E-03	c	2.0E-01	4.0E-03	c	2.4E-01
7.3E-01	E	1.1E-04	C						M	1	0.13		~Benzo[b]fluoranthene	205-99-2	1.6E-01	c	2.9E+00	c	9.2E-03	c	1.1E-01	c	3.4E-02	c		4.1E-02	c	
7.3E-02	E	1.1E-04	C						M	1	0.13		~Benzo[k]fluoranthene	207-08-9	1.6E+00	c	2.9E+01	c	9.2E-03	c	1.1E-01	c	3.4E-01	c		4.0E-01	c	
				8.0E-02	I			V		1	0.13		~Chloronaphthalene, Beta-	91-58-7	4.8E+03	n	6.0E+04	n					7.5E+02	n		3.8E+00	n	
7.3E-03	E	1.1E-05	C						M	1	0.13		~Chrysene	218-01-9	1.6E+01	c	2.9E+02	c	9.2E-02	c	1.1E+00	c	3.4E+00	c		1.2E+00	c	
7.3E+00	E	1.2E-03	C						M	1	0.13		~Dibenz[a,h]anthracene	53-70-3	1.6E-02	c	2.9E-01	c	8.4E-04	c	1.0E-02	c	3.4E-03	c		1.3E-02	c	
1.2E+01	C	1.1E-03	C							1	0.13		~Dibenzo(a,e)pyrene	192-65-4	4.2E-02	c	1.8E-01	c	2.6E-03	c	1.1E-02	c	6.5E-03	c		8.4E-02	c	
2.5E+02	C	7.1E-02	C						M	1	0.13		~Dimethylbenz(a)anthracene, 7,12-	57-97-6	4.6E-04	c	8.4E-03	c	1.4E-05	c	1.7E-04	c	1.0E-04	c		9.9E-05	c	
				4.0E-02	I					1	0.13		~Fluoranthene	206-44-0	2.4E+03	n	3.0E+04	n					8.0E+02	n		8.9E+01	n	
				4.0E-02	I			V		1	0.13		~Fluorene	86-73-7	2.4E+03	n	3.0E+04	n					2.9E+02	n		5.4E+00	n	
7.3E-01	E	1.1E-04	C						M	1	0.13		~Indeno[1,2,3-cd]pyrene	193-39-5	1.6E-01	c	2.9E+00	c	9.2E-03	c	1.1E-01	c	3.4E-02	c		1.3E-01	c	
2.9E-02	P			7.0E-02	A			V		1	0.13		~Methylnaphthalene, 1-	90-12-0	1.8E+01	c	7.3E+01	c					1.1E+00	c		5.8E-03	c	
				4.0E-03	I			V		1	0.13		~Methylnaphthalene, 2-	91-57-6	2.4E+02	n	3.0E+03	n					3.6E+01	n		1.9E-01	n	
		3.4E-05	C	2.0E-02	I	3.0E-03	I	V		1	0.13		~Naphthalene	91-20-3	3.8E+00	c*	1.7E+01	c*	8.3E-02	c*	3.6E-01	c*	1.7E-01	c*		5.4E-04	c*	
1.2E+00	C	1.1E-04	C							1	0.13		~Nitropyrene, 4-	57835-92-4	4.2E-01	c	1.8E+00	c	2.6E-02	c	1.1E-01	c	1.9E-02	c		3.2E-03	c	
				3.0E-02	I			V		1	0.13		~Pyrene	129-00-0	1.8E+03	n	2.3E+04	n					1.2E+02	n		1.3E+01	n	
				2.0E-02	P					1	0.1		Potassium Perfluorobutane Sulfonate	29420-49-3	1.3E+03	n	1.6E+04	n					4.0E+02	n				
1.5E-01	I			9.0E-03	I					1	0.1		Prochloraz	67747-09-5	3.6E+00	c	1.5E+01	c					3.7E-01	c		1.9E-03	c	
				6.0E-03	H			V		1			Profluralin	26399-36-0	4.7E+02	n	7.0E+03	n					2.6E+01	n		1.6E+00	n	
				1.5E-02	I					1	0.1		Prometon	1610-18-0	9.5E+02	n	1.2E+04	n					2.5E+02	n		1.2E-01	n	
				4.0E-03	I					1	0.1		Prometryn	7287-19-6	2.5E+02	n	3.3E+03	n					6.0E+01	n		9.0E-02	n	
				1.3E-02	I					1	0.1		Propachlor	1918-16-7	8.2E+02	n	1.1E+04	n					2.5E+02	n		1.5E-01	n	

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Toxicity and Chemical-specific Information												Contaminant		Screening Levels										Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
				5.0E-03	I					1	0.1		Propanil	709-98-8	3.2E+02	n	4.1E+03	n					8.2E+01	n		4.5E-02	n	
				2.0E-02	I					1	0.1		Propargite	2312-35-8	1.3E+03	n	1.6E+04	n					1.6E+02	n		1.2E+01	n	
				2.0E-03	I			V		1		1.1E+05	Propargyl Alcohol	107-19-7	1.6E+02	n	2.3E+03	n					4.0E+01	n		8.1E-03	n	
				2.0E-02	I					1	0.1		Propazine	139-40-2	1.3E+03	n	1.6E+04	n					3.4E+02	n		3.0E-01	n	
				2.0E-02	I					1	0.1		Propham	122-42-9	1.3E+03	n	1.6E+04	n					3.5E+02	n		2.2E-01	n	
				1.3E-02	I					1	0.1		Propiconazole	60207-90-1	8.2E+02	n	1.1E+04	n					2.1E+02	n		6.9E-01	n	
						8.0E-03	I	V		1		3.3E+04	Propionaldehyde	123-38-6	7.5E+01	n	3.1E+02	n	8.3E+00	n	3.5E+01	n	1.7E+01	n		3.4E-03	n	
				1.0E-01	X	1.0E+00	X	V		1		2.6E+02	Propyl benzene	103-65-1	3.8E+03	ns	2.4E+04	ns	1.0E+03	n	4.4E+03	n	6.6E+02	n		1.2E+00	n	
						3.0E+00	C	V		1		3.5E+02	Propylene	115-07-1	2.2E+03	ns	9.3E+03	ns	3.1E+03	n	1.3E+04	n	6.3E+03	n		6.0E+00	n	
				2.0E+01	P					1	0.1		Propylene Glycol	57-55-6	1.3E+06	nm	1.6E+07	nm					4.0E+05	n		8.1E+01	n	
						2.7E-04	A			1	0.1		Propylene Glycol Dinitrate	6423-43-4	3.9E+05	nm	1.6E+06	nm	2.8E-01	n	1.2E+00	n						
				7.0E-01	H			V		1		8.5E+04	Propylene Glycol Monoethyl Ether	1569-02-4	5.5E+04	n	8.2E+05	nms					1.4E+04	n		2.8E+00	n	
				7.0E-01	H	2.0E+00	I	V		1		1.1E+05	Propylene Glycol Monomethyl Ether	107-98-2	4.1E+04	n	3.7E+05	nms	2.1E+03	n	8.8E+03	n	3.2E+03	n		6.5E-01	n	
2.4E-01	I	3.7E-06	I			3.0E-02	I	V		1		7.8E+04	Propylene Oxide	75-56-9	2.1E+00	c	9.7E+00	c	7.6E-01	c*	3.3E+00	c*	2.7E-01	c		5.6E-05	c	
				2.5E-01	I					1	0.1		Pursuit	81335-77-5	1.6E+04	n	2.1E+05	nm					4.7E+03	n		4.1E+00	n	
				2.5E-02	I					1	0.1		Pydrin	51630-58-1	1.6E+03	n	2.1E+04	n					5.0E+02	n		3.2E+02	n	
				1.0E-03	I			V		1		5.3E+05	Pyridine	110-86-1	7.8E+01	n	1.2E+03	n					2.0E+01	n		6.8E-03	n	
				5.0E-04	I					1	0.1		Quinalphos	13593-03-8	3.2E+01	n	4.1E+02	n					5.1E+00	n		4.3E-02	n	
3.0E+00	I									1	0.1		Quinoline	91-22-5	1.8E-01	c	7.7E-01	c					2.4E-02	c		7.8E-05	c	
						3.0E-02	A			1			Refractory Ceramic Fibers	NA	4.3E+07	nm	1.8E+08	nm	3.1E+01	n	1.3E+02	n						
				3.0E-02	I					1	0.1		Resmethrin	10453-86-8	1.9E+03	n	2.5E+04	n					6.7E+01	n		4.2E+01	n	
				5.0E-02	H			V		1			Ronnel	299-84-3	3.9E+03	n	5.8E+04	n					4.1E+02	n		3.7E+00	n	
				4.0E-03	I					1	0.1		Rotenone	83-79-4	2.5E+02	n	3.3E+03	n					6.1E+01	n		3.2E+01	n	
2.2E-01	C	6.3E-05	C						M	1	0.1		Safrole	94-59-7	5.5E-01	c	1.0E+01	c	1.6E-02	c	1.9E-01	c	9.5E-02	c		5.9E-05	c	
				2.5E-02	I					1	0.1		Savey	78587-05-0	1.6E+03	n	2.1E+04	n					1.1E+02	n		5.0E-01	n	
				5.0E-03	I					1			Selenious Acid	7783-00-8	3.9E+02	n	5.8E+03	n					1.0E+02	n			n	
				5.0E-03	I	2.0E-02	C			1			Selenium	7782-49-2	3.9E+02	n	5.8E+03	n	2.1E+01	n	8.8E+01	n	1.0E+02	n	5.0E+01	5.2E-01	n	2.6E-01
				5.0E-03	C	2.0E-02	C			1			Selenium Sulfide	7446-34-6	3.9E+02	n	5.8E+03	n	2.1E+01	n	8.8E+01	n	1.0E+02	n			n	
				9.0E-02	I					1	0.1		Sethoxydim	74051-80-2	5.7E+03	n	7.4E+04	n					1.0E+03	n		9.3E+00	n	
						3.0E-03	C			1			Silica (crystalline, respirable)	7631-86-9	4.3E+06	nm	1.8E+07	nm	3.1E+00	n	1.3E+01	n						
				5.0E-03	I						0.04		Silver	7440-22-4	3.9E+02	n	5.8E+03	n					9.4E+01	n		8.0E-01	n	
1.2E-01	H			5.0E-03	I					1	0.1		Simazine	122-34-9	4.5E+00	c*	1.9E+01	c					6.1E-01	c	4.0E+00	3.0E-04	c	2.0E-03
				1.3E-02	I					1	0.1		Sodium Acifluorfen	62476-59-9	8.2E+02	n	1.1E+04	n					2.6E+02	n		2.1E+00	n	
				4.0E-03	I					1			Sodium Azide	26628-22-8	3.1E+02	n	4.7E+03	n					8.0E+01	n			n	
5.0E-01	C	1.5E-01	C	2.0E-02	C	2.0E-04	C		M	0.025			Sodium Dichromate	10588-01-9	3.0E-01	c	6.2E+00	c	6.8E-06	c	8.2E-05	c	4.1E-02	c			c	
2.7E-01	H			3.0E-02	I					1	0.1		Sodium Diethyldithiocarbamate	148-18-5	2.0E+00	c	8.5E+00	c					2.9E-01	c			c	
				5.0E-02	A	1.3E-02	C			1			Sodium Fluoride	7681-49-4	3.9E+03	n	5.8E+04	n	1.4E+01	n	5.7E+01	n	1.0E+03	n			n	
				2.0E-05	I					1	0.1		Sodium Fluoroacetate	62-74-8	1.3E+00	n	1.6E+01	n					4.0E-01	n		8.1E-05	n	
				1.0E-03	H					1			Sodium Metavanadate	13718-26-8	7.8E+01	n	1.2E+03	n					2.0E+01	n			n	
2.4E-02	H			3.0E-02	I					1	0.1		Stirofos (Tetrachlorovinphos)	961-11-5	2.3E+01	c*	9.6E+01	c					2.8E+00	c		8.1E-03	c	
5.0E-01	C	1.5E-01	C	2.0E-02	C	2.0E-04	C		M	0.025			Strontium Chromate	7789-06-2	3.0E-01	c	6.2E+00	c	6.8E-06	c	8.2E-05	c	4.1E-02	c			c	
				6.0E-01	I					1			Strontium, Stable	7440-24-6	4.7E+04	n	7.0E+05	nm					1.2E+04	n		4.2E+02	n	
				3.0E-04	I					1	0.1		Strychnine	57-24-9	1.9E+01	n	2.5E+02	n					5.9E+00	n		6.5E-02	n	
				2.0E-01	I	1.0E+00	I	V		1		8.7E+02	Styrene	100-42-5	6.0E+03	ns	3.5E+04	ns	1.0E+03	n	4.4E+03	n	1.2E+03	n	1.0E+02	1.3E+00	n	1.1E-01
				3.0E-03	P					1	0.1		Styrene-Acrylonitrile (SAN) Trimer	NA	1.9E+02	n	2.5E+03	n					4.8E+01	n			n	
				1.0E-03	P	2.0E-03	X			1	0.1		Sulfolane	126-33-0	6.3E+01	n	8.2E+02	n	2.1E+00	n	8.8E+00	n	2.0E+01	n		4.4E-03	n	
				8.0E-04	P					1	0.1		Sulfonylbis(4-chlorobenzene), 1,1'-	80-07-9	5.1E+01	n	6.6E+02	n					1.1E+01	n		6.5E-02	n	
						1.0E-03	C	V		1			Sulfur Trioxide	7446-11-9	1.4E+06	nm	6.0E+06	nm	1.0E+00	n	4.4E+00	n	2.1E+00	n			n	
						1.0E-03	C			1			Sulfuric Acid	7664-93-9	1.4E+06	nm	6.0E+06	nm	1.0E+00	n	4.4E+00	n						
				2.5E-02	I					1	0.1		Systhane	88671-89-0	1.6E+03	n	2.1E+04	n					4.5E+02	n		5.6E+00	n	
				3.0E-02	H					1	0.1		TCMTB	21564-17-0	1.9E+03	n	2.5E+04	n					4.8E+02	n		3.3E+00	n	
				7.0E-02	I					1	0.1		Tebuthiuron	34014-18-1	4.4E+03	n	5.7E+04	n					1.4E+03	n		3.9E-01	n	
				2.0E-02	H					1	0.1		Temephos	3383-96-8	1.3E+03	n	1.6E+04	n					4.0E+02	n		7.6E+01	n	
				1.3E-02	I					1	0.1		Terbacil	5902-51-2	8.2E+02	n	1.1E+04	n					2.5E+02	n		7.5E-02	n	
				2.5E-05	H			V		1		3.1E+01	Terbufos	13071-79-9	2.0E+00	n	2.9E+01	n					2.4E-01	n		5.2E-04	n	
				1.0E-03	I					1	0.1		Terbutryn	886-50-0	6.3E+01	n	8.2E+02	n					1.3E+01	n		1.9E-02	n	
				1.0E-04	I					1	0.1		Tetrabromodiphenyl ether, 2,2',4,4'- (BDE-47)	5436-43-1	6.3E+00	n	8.2E+01	n					2.0E+00	n		5.3E-02	n	
				3.0E-04	I			V		1			Tetrachlorobenzene, 1,2,4,5-	95-94-3	2.3E+01	n	3.5E+02	n					1.7E+00	n		7.9E-03	n	
2.6E-02	I	7.4E-06	I	3.0E-02	I			V		1		6.8E+02	Tetrachloroethane, 1,1,1,2-	630-20-6	2.0E+00	c	8.8E+00	c	3.8E-01	c	1.7E+00	c	5.7E-01	c		2.2E-04	c	
2.0E-01	I	5.8E-05	C	2.0E-02	I			V		1		1.9E+03	Tetrachloroethane, 1,1,2,2-	79-34-5	6.0E-01	c	2.7E+00	c	4.8E-02	c	2.1E-01	c	7.6E-02	c		3.0E-05	c	
2.1E-03	I	2.6E-07	I	6.0E-03	I	4.0E-02	I	V		1		1.7E+02	Tetrachloroethylene	127-18-4	2.4E+01	c**	1.0E+02	c**	1.1E+01	c**	4.7E+01	c**	1.1E+01	c**	5.0E+00	5.1E-03	c**	2.3E-03
				3.0E-02	I					1	0.1		Tetrachlorophenol, 2,3,4,6-	58-90-2	1.9E+03	n	2.5E+04	n					2.4E+02	n		1.5E+00	n	
2.0E+01	H							V		1			Tetrachlorotoluene, p- alpha, alpha, alpha-	5216-25-1	3.5E-02	c	1.6E-01	c					1.3E-03	c		4.4E-06	c	
				5.0E-04	I					1	0.1		Tetraethyl Dithiopyrophosphate	3689-24-5	3.2E+01	n	4.1E+02	n					7.1E+00	n		5.2E-03	n	
						8.0E+01	I	V		1		1.1E+03	Tetrafluoroethane, 1,1,1,2-	811-97-2	1.0E+05	nms	4.3E+05	nms	8.3E+04	n	3.5E+05	n	1.7E+05	n		9.3E+01	n	

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Toxicity and Chemical-specific Information													Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	volc	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
				2.0E-03	P					1	0.0007		Tetryl (Trinitrophenylmethylnitramine)	479-45-8	1.6E+02	n	2.3E+03	n					3.9E+01	n		3.7E-01	n	
				7.0E-06	X					1			Thallium (I) Nitrate	10102-45-1	5.5E-01	n	8.2E+00	n					1.4E-01	n			n	
				1.0E-05	X					1			Thallium (Soluble Salts)	7440-28-0	7.8E-01	n	1.2E+01	n					2.0E-01	n	2.0E+00	1.4E-02	n	1.4E-01
				6.0E-06	X			V		1	0.1		Thallium Acetate	563-68-8	3.8E-01	n	4.9E+00	n					1.2E-01	n			n	
				2.0E-05	X					1	0.1		Thallium Carbonate	6533-73-9	1.3E+00	n	1.6E+01	n					4.0E-01	n			n	
				6.0E-06	X					1			Thallium Chloride	7791-12-0	4.7E-01	n	7.0E+00	n					1.2E-01	n			n	
				2.0E-05	X					1			Thallium Sulfate	7446-18-6	1.6E+00	n	2.3E+01	n					4.0E-01	n			n	
				1.0E-02	I					1	0.1		Thiobencarb	28249-77-6	6.3E+02	n	8.2E+03	n					1.6E+02	n		5.5E-01	n	
				7.0E-02	X					1	0.0075		Thiodiglycol	111-48-8	5.4E+03	n	7.9E+04	n					1.4E+03	n		2.8E-01	n	
				3.0E-04	H					1	0.1		Thiofanox	39196-18-4	1.9E+01	n	2.5E+02	n					5.3E+00	n		1.8E-03	n	
				8.0E-02	I					1	0.1		Thiophanate, Methyl	23564-05-8	5.1E+03	n	6.6E+04	n					1.6E+03	n		1.4E+00	n	
				5.0E-03	I					1	0.1		Thiram	137-26-8	3.2E+02	n	4.1E+03	n					9.8E+01	n		1.4E-01	n	
				6.0E-01	H					1			Tin	7440-31-5	4.7E+04	n	7.0E+05	nm					1.2E+04	n		3.0E+03	n	
						1.0E-04	A	V		1			Titanium Tetrachloride	7550-45-0	1.4E+05	nm	6.0E+05	nm	1.0E-01	n	4.4E-01	n	2.1E-01	n			n	
				8.0E-02	I	5.0E+00	I	V		1		8.2E+02	Toluene	108-88-3	4.9E+03	ns	4.7E+04	ns	5.2E+03	n	2.2E+04	n	1.1E+03	n	1.0E+03	7.6E-01	n	6.9E-01
1.8E-01	X			2.0E-04	X					1	0.1		Toluene-2,5-diamine	95-70-5	3.0E+00	c**	1.3E+01	c*					4.3E-01	c**		1.3E-04	c**	
3.0E-02	P			4.0E-03	X					1	0.1		Toluidine, p-	106-49-0	1.8E+01	c*	7.7E+01	c*					2.5E+00	c*		1.1E-03	c*	
				3.0E+00	P			V		1		3.4E-01	Total Petroleum Hydrocarbons (Aliphatic High)	NA	2.3E+05	nms	3.5E+06	nms					6.0E+04	n		2.4E+03	n	
						6.0E-01	P	V		1		1.4E+02	Total Petroleum Hydrocarbons (Aliphatic Low)	NA	5.2E+02	ns	2.2E+03	ns	6.3E+02	n	2.6E+03	n	1.3E+03	n		8.8E+00	n	
				1.0E-02	X	1.0E-01	P	V		1		6.9E+00	Total Petroleum Hydrocarbons (Aliphatic Medium)	NA	9.6E+01	ns	4.4E+02	ns	1.0E+02	n	4.4E+02	n	1.0E+02	n		1.5E+00	n	
				4.0E-02	P					1	0.1		Total Petroleum Hydrocarbons (Aromatic High)	NA	2.5E+03	n	3.3E+04	n					8.0E+02	n		8.9E+01	n	
				4.0E-03	P	3.0E-02	P	V		1		1.8E+03	Total Petroleum Hydrocarbons (Aromatic Low)	NA	8.2E+01	n	4.2E+02	n	3.1E+01	n	1.3E+02	n	3.3E+01	n		1.7E-02	n	
				4.0E-03	P	3.0E-03	P	V		1			Total Petroleum Hydrocarbons (Aromatic Medium)	NA	1.1E+02	n	6.0E+02	n	3.1E+00	n	1.3E+01	n	5.5E+00	n		2.3E-02	n	
1.1E+00	I	3.2E-04	I							1	0.1		Toxaphene	8001-35-2	4.9E-01	c	2.1E+00	c	8.8E-03	c	3.8E-02	c	1.5E-02	c	3.0E+00	2.4E-03	c	4.6E-01
				7.5E-03	I					1	0.1		Tralomethrin	66841-25-6	4.7E+02	n	6.2E+03	n					1.5E+02	n		5.8E+01	n	
				3.0E-04	A			V		1			Tri-n-butyltin	688-73-3	2.3E+01	n	3.5E+02	n					3.7E+00	n		8.2E-02	n	
				8.0E+01	X					1	0.1		Triacetin	102-76-1	5.1E+06	nm	6.6E+07	nm					1.6E+06	n		4.5E+02	n	
				1.3E-02	I			V		1			Triallate	2303-17-5	1.0E+03	n	1.5E+04	n					1.2E+02	n		2.6E-01	n	
				1.0E-02	I					1	0.1		Triasulfuron	82097-50-5	6.3E+02	n	8.2E+03	n					2.0E+02	n		2.1E-01	n	
				5.0E-03	I			V		1			Tribromobenzene, 1,2,4-	615-54-3	3.9E+02	n	5.8E+03	n					4.5E+01	n		6.4E-02	n	
9.0E-03	P			1.0E-02	P					1	0.1		Tributyl Phosphate	126-73-8	6.0E+01	c*	2.6E+02	c*					5.1E+00	c*		2.5E-02	c*	
				3.0E-04	P					1	0.1		Tributyltin Compounds	NA	1.9E+01	n	2.5E+02	n					6.0E+00	n			n	
				3.0E-04	I					1	0.1		Tributyltin Oxide	56-35-9	1.9E+01	n	2.5E+02	n					5.7E+00	n		2.9E+02	n	
				3.0E+01	I	3.0E+01	H	V		1		9.1E+02	Trichloro-1,2,2-trifluoroethane, 1,1,2-	76-13-1	4.0E+04	ns	1.7E+05	nms	3.1E+04	n	1.3E+05	n	5.5E+04	n		1.4E+02	n	
7.0E-02	I			2.0E-02	I					1	0.1		Trichloroacetic Acid	76-03-9	7.8E+00	c	3.3E+01	c					1.1E+00	c	6.0E+01	2.2E-04	c	1.2E-02
2.9E-02	H									1	0.1		Trichloroaniline HCl, 2,4,6-	33663-50-2	1.9E+01	c	7.9E+01	c					2.7E+00	c		7.4E-03	c	
7.0E-03	X			3.0E-05	X					1	0.1		Trichloroaniline, 2,4,6-	634-93-5	1.9E+00	n	2.5E+01	n					4.0E-01	n		3.6E-03	n	
				8.0E-04	X			V		1			Trichlorobenzene, 1,2,3-	87-61-6	6.3E+01	n	9.3E+02	n					7.0E+00	n		2.1E-02	n	
2.9E-02	P			1.0E-02	I	2.0E-03	P	V		1		4.0E+02	Trichlorobenzene, 1,2,4-	120-82-1	2.4E+01	c**	1.1E+02	c**	2.1E+00	n	8.8E+00	n	1.1E+00	c**	7.0E+01	3.3E-03	c**	2.0E-01
				2.0E+00	I	5.0E+00	I	V		1		6.4E+02	Trichloroethane, 1,1,1-	71-55-6	8.1E+03	ns	3.6E+04	ns	5.2E+03	n	2.2E+04	n	8.0E+03	n	2.0E+02	2.8E+00	n	7.0E-02
5.7E-02	I	1.6E-05	I	4.0E-03	I	2.0E-04	X	V		1		2.2E+03	Trichloroethane, 1,1,2-	79-00-5	1.1E+00	c**	5.0E+00	c**	1.8E-01	c**	7.7E-01	c**	2.8E-01	c**	5.0E+00	8.9E-05	c**	1.6E-03
4.6E-02	I	4.1E-06	I	5.0E-04	I	2.0E-03	I	V	M	1		6.9E+02	Trichloroethylene	79-01-6	9.4E-01	c**	6.0E+00	c**	4.8E-01	c**	3.0E+00	c**	4.9E-01	c**	5.0E+00	1.8E-04	c**	1.8E-03
				3.0E-01	I	7.0E-01	H	V		1		1.2E+03	Trichlorofluoromethane	75-69-4	7.3E+02	n	3.1E+03	ns	7.3E+02	n	3.1E+03	n	1.1E+03	n		7.3E-01	n	
				1.0E-01	I					1	0.1		Trichlorophenol, 2,4,5-	95-95-4	6.3E+03	n	8.2E+04	n					1.2E+03	n		4.4E+00	n	
1.1E-02	I	3.1E-06	I	1.0E-03	P					1	0.1		Trichlorophenol, 2,4,6-	88-06-2	4.9E+01	c**	2.1E+02	c**	9.1E-01	c	4.0E+00	c	4.0E+00	c**		1.5E-02	c**	
				1.0E-02	I					1	0.1		Trichlorophenoxyacetic Acid, 2,4,5-	93-76-5	6.3E+02	n	8.2E+03	n					1.6E+02	n		6.7E-02	n	
				8.0E-03	I					1	0.1		Trichlorophenoxypropionic acid, -2,4,5	93-72-1	5.1E+02	n	6.6E+03	n					1.1E+02	n	5.0E+01	6.1E-02	n	2.8E-02
				5.0E-03	I			V		1		1.3E+03	Trichloropropane, 1,1,2-	598-77-6	3.9E+02	n	5.8E+03	ns					8.8E+01	n		3.5E-02	n	
3.0E+01	I			4.0E-03	I	3.0E-04	I	V	M	1		1.4E+03	Trichloropropane, 1,2,3-	96-18-4	5.1E-03	c	1.1E-01	c	3.1E-01	n	1.3E+00	n	7.5E-04	c		3.2E-07	c	
				3.0E-03	X	3.0E-04	P	V		1		4.5E+02	Trichloropropene, 1,2,3-	96-19-5	7.3E-01	n	3.1E+00	n	3.1E-01	n	1.3E+00	n	6.2E-01	n		3.1E-04	n	
				2.0E-02	A					1	0.1		Tricresyl Phosphate (TCP)	1330-78-5	1.3E+03	n	1.6E+04	n					1.6E+02	n		1.5E+01	n	
				3.0E-03	I					1	0.1		Tridiphane	58138-08-2	1.9E+02	n	2.5E+03	n					1.8E+01	n		1.3E-01	n	
						7.0E-03	I	V		1		2.8E+04	Triethylamine	121-44-8	1.2E+02	n	4.8E+02	n	7.3E+00	n	3.1E+01	n	1.5E+01	n		4.4E-03	n	
				2.0E+00	P					1	0.1		Triethylene Glycol	112-27-6	1.3E+05	nm	1.6E+06	nm					4.0E+04	n		8.8E+00	n	
7.7E-03	I			7.5E-03	I			V		1			Trifluralin	1582-09-8	9.0E+01	c**	4.2E+02	c*					2.5E+00	c*		8.2E-02	c*	
2.0E-02	P			1.0E-02	P					1	0.1		Trimethyl Phosphate	512-56-1	2.7E+01	c*	1.1E+02	c*					3.9E+00	c*		8.6E-04	c*	
						5.0E-03	P	V		1		2.9E+02	Trimethylbenzene, 1,2,3-	526-73-8	4.9E+01	n	2.1E+02	n	5.2E+00	n	2.2E+01	n	1.0E+01	n		1.5E-02	n	
						7.0E-03	P	V		1		2.2E+02	Trimethylbenzene, 1,2,4-	95-63-6	5.8E+01	n	2.4E+02	ns	7.3E+00	n	3.1E+01	n	1.5E+01	n		2.1E-02	n	
				1.0E-02	X			V		1		1.8E+02	Trimethylbenzene, 1,3,5-	108-67-8	7.8E+02	ns	1.2E+04	ns					1.2E+02	n		1.7E-01	n	
				3.0E-02	I					1	0.019		Trinitrobenzene, 1,3,5-	99-35-4	2.2E+03	n	3.2E+04	n					5.9E+02	n		2.1E+00	n	
3.0E-02	I			5.0E-04	I					1	0.032		Trinitrotoluene, 2,4,6-	118-96-7	2.1E+01	c**	9.6E+01	c**					2.5E+00	c**		1.5E-02	c**	
				2.0E-02	P					1	0.1		Triphenylphosphine Oxide	791-28-6	1.3E+03	n	1.6E+04	n					3.6E+02	n		1.5E+00	n	
				2.0E-02	A					1	0.1		Tris(1,3-Dichloro-2-propyl) Phosphate	13674-87-8	1.3E+03	n	1.6E+04	n					3.6E+02	n		8.0E+00	n	
				1.0E-02	X					1	0.1		Tris(1-chloro-2-propyl)phosphate	13674-84-5	6.3E+02	n	8.2E+03	n					1.9E+02	n		6.5E-01	n	
2.3E+00	C	6.6E-04	C					V		1		4.7E+02	Tris(2,3-dibromopropyl)phosphate	126-72-7	2.8E-01	c	1.3E+00	c	4.3E-03	c	1.9E-02	c	6.8E-03	c		1.3E-04	c	

Key: I = IRIS; P = PPRTV; A = ATSDR; C = Cal EPA; X = APPENDIX PPRTV SCREEN (See FAQ #27); H = HEAST; J = New Jersey; O = EPA Office of Water; F = See FAQ; E = Environmental Criteria and Assessment Office; S = see user guide Section 5; L = see user guide on lead; M = mutagen; V = volatile; R = RBA applied (See User Guide for Arsenic notice) ; c = cancer; * = where: n SL < 100X c SL; ** = where n SL < 10X c SL; n = noncancer; m = Concentration may exceed ceiling limit (See User Guide); s = Concentration may exceed Csat (See User Guide); SSL values are based on DAF=1

Toxicity and Chemical-specific Information												Contaminant		Screening Levels												Protection of Ground Water SSLs		
SFO (mg/kg-day)$^{-1}$	key	IUR (ug/m^3)$^{-1}$	key	RfD_o (mg/kg-day)	key	RfC_i (mg/m^3)	key	vol	muta-gen	GIABS	ABS	C_{sat} (mg/kg)	Analyte	CAS No.	Resident Soil (mg/kg)	key	Industrial Soil (mg/kg)	key	Resident Air (ug/m^3)	key	Industrial Air (ug/m^3)	key	Tapwater (ug/L)	key	MCL (ug/L)	Risk-based SSL (mg/kg)	key	MCL-based SSL (mg/kg)
2.0E-02	P			7.0E-03	P					1	0.1		Tris(2-chloroethyl)phosphate	115-96-8	2.7E+01	c*	1.1E+02	c*					3.8E+00	c*		3.8E-03	c*	
3.2E-03	P			1.0E-01	P					1	0.1		Tris(2-ethylhexyl)phosphate	78-42-2	1.7E+02	c*	7.2E+02	c					2.4E+01	c*		1.2E+02	c*	
				3.0E-03	I	4.0E-05	A			1			Uranium (Soluble Salts)	NA	2.3E+02	n	3.5E+03	n	4.2E-02	n	1.8E-01	n	6.0E+01	n	3.0E+01	2.7E+01	n	1.4E+01
1.0E+00	C	2.9E-04	C						M	1	0.1		Urethane	51-79-6	1.2E-01	c	2.3E+00	c	3.5E-03	c	4.2E-02	c	2.5E-02	c		5.6E-06	c	
		8.3E-03	P	9.0E-03	I	7.0E-06	P			0.026			Vanadium Pentoxide	1314-62-1	4.6E+02	c**	2.0E+03	c**	3.4E-04	c*	1.5E-03	c*	1.5E+02	n			n	
				5.0E-03	S	1.0E-04	A			0.026			Vanadium and Compounds	7440-62-2	3.9E+02	n	5.8E+03	n	1.0E-01	n	4.4E-01	n	8.6E+01	n		8.6E+01	n	
				1.0E-03	I			V		1			Vernolate	1929-77-7	7.8E+01	n	1.2E+03	n					1.1E+01	n		8.9E-03	n	
				2.5E-02	I					1	0.1		Vinclozolin	50471-44-8	1.6E+03	n	2.1E+04	n					4.4E+02	n		3.4E-01	n	
				1.0E+00	H	2.0E-01	I	V		1		2.8E+03	Vinyl Acetate	108-05-4	9.1E+02	n	3.8E+03	ns	2.1E+02	n	8.8E+02	n	4.1E+02	n		8.7E-02	n	
		3.2E-05	H			3.0E-03	I	V		1		3.4E+03	Vinyl Bromide	593-60-2	1.2E-01	c*	5.2E-01	c*	8.8E-02	c*	3.8E-01	c*	1.8E-01	c*		5.1E-05	c*	
7.2E-01	I	4.4E-06	I	3.0E-03	I	1.0E-01	I	V	M	1		3.9E+03	Vinyl Chloride	75-01-4	5.9E-02	c	1.7E+00	c	1.7E-01	c	2.8E+00	c	1.9E-02	c	2.0E+00	6.5E-06	c	6.9E-04
				3.0E-04	I					1	0.1		Warfarin	81-81-2	1.9E+01	n	2.5E+02	n					5.6E+00	n		5.9E-03	n	
				2.0E-01	S	1.0E-01	S	V		1		3.9E+02	Xylene, p-	106-42-3	5.6E+02	ns	2.4E+03	ns	1.0E+02	n	4.4E+02	n	1.9E+02	n		1.9E-01	n	
				2.0E-01	S	1.0E-01	S	V		1		3.9E+02	Xylene, m-	108-38-3	5.5E+02	ns	2.4E+03	ns	1.0E+02	n	4.4E+02	n	1.9E+02	n		1.9E-01	n	
				2.0E-01	S	1.0E-01	S	V		1		4.3E+02	Xylene, o-	95-47-6	6.5E+02	ns	2.8E+03	ns	1.0E+02	n	4.4E+02	n	1.9E+02	n		1.9E-01	n	
				2.0E-01	I	1.0E-01	I	V		1		2.6E+02	Xylenes	1330-20-7	6.5E+02	ns	2.8E+03	ns	1.0E+02	n	4.4E+02	n	1.9E+02	n	1.0E+04	1.9E-01	n	9.8E+00
				3.0E-04	I					1			Zinc Phosphide	1314-84-7	2.3E+01	n	3.5E+02	n					6.0E+00	n			n	
				3.0E-01	I					1			Zinc and Compounds	7440-66-6	2.3E+04	n	3.5E+05	nm					6.0E+03	n		3.7E+02	n	
				5.0E-02	I					1	0.1		Zineb	12122-67-7	3.2E+03	n	4.1E+04	n					9.9E+02	n		2.9E+00	n	
				8.0E-05	X					1			Zirconium	7440-67-7	6.3E+00	n	9.3E+01	n					1.6E+00	n		4.8E+00	n	

11.0 Final SCO Tables from Part 375

Tables 11-1 and 11-2 show the final SCOs as presented in 6 NYCRR Part 375-6.8. The Unrestricted use values shown in Table 11-1 were derived from the final human-health based SCOs (Table 5.6-1), the groundwater SCOs (Table 7-1) and the ecological SCOs (Table 8.6-1). The lowest of these values was selected as the final SCO, unless a corresponding rural soil background concentration (Tables 9.1-9 and 9.2-1) was higher, in which case the lowest rural soil background concentration was selected as the final SCO. If the final SCO was lower than the CRQL for a chemical (Section 9.4), the CRQL was substituted as the final SCO.

Table 11-1. Final Unrestricted Use SCOs as Presented in 6 NYCRR Part 375-6.8(a).

Unrestricted Use Soil Cleanup Objectives		
Contaminant	**CAS Number**	**Unrestricted Use**
Metals		
Arsenic	7440-38-2	13[c]
Barium	7440-39-3	350[c]
Beryllium	7440-41-7	7.2
Cadmium	7440-43-9	2.5[c]
Chromium, hexavalent[e]	18540-29-9	1[b]
Chromium, trivalent[e]	16065-83-1	30[c]
Copper	7440-50-8	50
Total Cyanide[e,f]		27
Lead	7439-92-1	63[c]
Manganese	7439-96-5	1600[c]
Total Mercury		0.18[c]
Nickel	7440-02-0	30
Selenium	7782-49-2	3.9[c]
Silver	7440-22-4	2
Zinc	7440-66-6	109[c]
PCBs/Pesticides		
2,4,5-TP Acid (Silvex)[f]	93-72-1	3.8
4,4’-DDE	72-55-9	0.0033[b]
4,4’-DDT	50-29-3	0.0033[b]
4,4'-DDD	72-54-8	0.0033[b]
Aldrin	309-00-2	0.005[c]
alpha-BHC	319-84-6	0.02
beta-BHC	319-85-7	0.036

Unrestricted Use Soil Cleanup Objectives		
Contaminant	**CAS Number**	**Unrestricted Use**
Chlordane (alpha)	5103-71-9	0.094
delta-BHC	319-86-8	0.04
Dibenzofuran[f]	132-64-9	7
Dieldrin	60-57-1	0.005[c]
Endosulfan I[d,f]	959-98-8	2.4
Endosulfan II[d,f]	33213-65-9	2.4
Endosulfan sulfate[d,f]	1031-07-8	2.4
Endrin	72-20-8	0.014
Heptachlor	76-44-8	0.042
Lindane	58-89-9	0.1
Polychlorinated biphenyls	1336-36-3	0.1
Semivolatile organic compounds		
Acenaphthene	83-32-9	20
Acenapthylene[f]	208-96-8	100[a]
Anthracene[f]	120-12-7	100[a]
Benz(a)anthracene[f]	56-55-3	1[c]
Benzo(a)pyrene	50-32-8	1[c]
Benzo(b)fluoranthene[f]	205-99-2	1[c]
Benzo(g,h,i)perylene[f]	191-24-2	100
Benzo(k)fluoranthene[f]	207-08-9	0.8[c]
Chrysene[f]	218-01-9	1[c]
Dibenz(a,h)anthracene[f]	53-70-3	0.33[b]
Fluoranthene[f]	206-44-0	100[a]
Fluorene	86-73-7	30
Indeno(1,2,3-cd)pyrene[f]	193-39-5	0.5[c]
m-Cresol[f]	108-39-4	0.33[b]

Unrestricted Use Soil Cleanup Objectives		
Contaminant	**CAS Number**	**Unrestricted Use**
Naphthalene[f]	91-20-3	12
o-Cresol[f]	95-48-7	0.33[b]
p-Cresol[f]	106-44-5	0.33[b]
Pentachlorophenol	87-86-5	0.8[b]
Phenanthrene[f]	85-01-8	100
Phenol	108-95-2	0.33[b]
Pyrene[f]	129-00-0	100
Volatile organic compounds		
1,1,1-Trichloroethane[f]	71-55-6	0.68
1,1-Dichloroethane[f]	75-34-3	0.27
1,1-Dichloroethene[f]	75-35-4	0.33
1,2-Dichlorobenzene[f]	95-50-1	1.1
1,2-Dichloroethane	107-06-2	0.02[c]
cis-1,2-Dichloroethene[f]	156-59-2	0.25
trans-1,2-Dichloroethene[f]	156-60-5	0.19
1,3-Dichlorobenzene[f]	541-73-1	2.4
1,4-Dichlorobenzene	106-46-7	1.8
1,4-Dioxane	123-91-1	0.1[b]
Acetone	67-64-1	0.05
Benzene	71-43-2	0.06
n-Butylbenzene[f]	104-51-8	12
Carbon tetrachloride[f]	56-23-5	0.76
Chlorobenzene	108-90-7	1.1
Chloroform	67-66-3	0.37
Ethylbenzene[f]	100-41-4	1
Hexachlorobenzene[f]	118-74-1	0.33[b]

Unrestricted Use Soil Cleanup Objectives		
Contaminant	**CAS Number**	**Unrestricted Use**
Methyl ethyl ketone	78-93-3	0.12
Methyl tert-butyl ether[f]	1634-04-4	0.93
Methylene chloride	75-09-2	0.05
n-Propylbenzene[f]	103-65-1	3.9
sec-Butylbenzene[f]	135-98-8	11
tert-Butylbenzene[f]	98-06-6	5.9
Tetrachloroethene	127-18-4	1.3
Toluene	108-88-3	0.7
Trichloroethene	79-01-6	0.47
1,2,4-Trimethylbenzene[f]	95-63-6	3.6
1,3,5-Trimethylbenzene[f]	108-67-8	8.4
Vinyl chloride[f]	75-01-4	0.02
Xylene (mixed)	1330-20-7	0.26

All Soil clean up objectives (SCOs) are in parts per million (ppm).
Footnotes:
[a] The SCOs for unrestricted use were capped at a maximum value of 100 ppm, as discussed in the TSD.
[b] For constituents where the calculated SCO was lower than the Contract Required Quantitation Limit (CRQL), the CRQL is used as the Track 1 SCO value.
[c] For constituents where the calculated SCO was lower than the rural soil background concentration as determined by the DEC/DOH rural soil survey, the rural soil background concentration is used as the Track 1 SCO value for this use of the site.
[d] SCO is the sum of Endosulfan I, Endosulfan II and Endosulfan Sulfate.
[e] The SCO for this specific compound (or family of compounds) is considered to be met if the analysis for the total species of this contaminant is below the specific SCO.
[f] Protection of ecological resources soil cleanup objectives were not developed for contaminants identified in Table 375-6.7(b) with "NS". Where such contaminants appear in Table 375-6.7(a), the applicant may be required by the Department to calculate a protection of ecological resources soil cleanup objective according to the Technical Support Document.

ATTACHMENT B

FIELD DATA COLLECTION FORMS

Boring Log	**Project Name:**	**Log of Boring**
	Client:	
	Project Number:	

Date(s) Drilled	**Logged By**	**Checked By**	**Total Depth of Borehole (ft)**	**Depth to Water (bgs)**
Drilling Method	**Diameter of Borehole (in)**		**Ground Surface Elevation (ft-msl)**	
Drill Rig Type	**Drilling Company**		**Groundwater Elevation (ft-msl)**	
Driller's Name	**Sampler Type**		**Measuring Point Elevation (ft-msl)**	
Description of Sample Location			**Northing Easting**	

Depth (ft-bgs)	**SAMPLES**			**USCS Symbol**	**Graphic Log**	**MATERIAL DESCRIPTION**	**REMARKS**
	Blows/6"	**Recovery (ft)**	**PID (ppm)**				
1							
2							
3							
4							
5							
6							
7							

Boring Log	**Project Name:**	**Log of Boring** _____
	Client:	
	Project Number:	

Depth (ft-bgs)	SAMPLES: Blows/Foot	SAMPLES: Recovery (ft)	SAMPLES: PID (ppm)	USCS Symbol	Graphic Log	MATERIAL DESCRIPTION	REMARKS
8							
9							
10							
11							
12							
13							
14							
15							
16							

Monitoring Well Construction Log Form

Project Name: _____ **Well No.** _____

Project Location: _____ **Project No.** _____

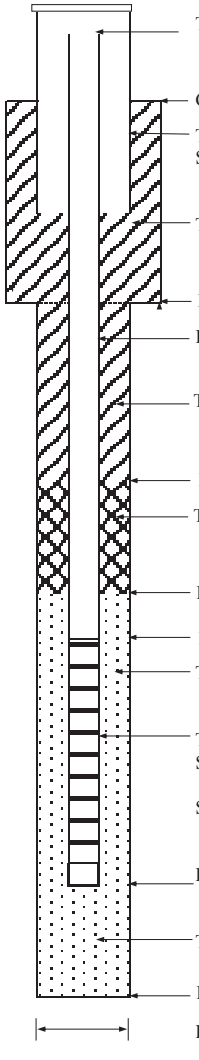
Installed By: _____ **Observed By:** _____ **Date of Well Completion** _____

Method of Installation: _____

Well Type (circle one): Single Cased Double Cased

Coordinates: Northing _____ **Survey Datums:** _____

Easting _____ _____



	feet bgs	Elevation (feet MSL)
Top of Casing		
Ground Elevation		
Type of surface casing		
Surface Casing ID		
Type of surface seal		
Depth of surface seal	____/	____/
I.D./Type of riser pipe		
Type of grout		
Depth to top of seal	____/	____/
Type of seal		
Depth to top of filter pack	____/	____/
Depth to top of screen	____/	____/
Type of filter pack		
Type of screen		
Screen ID		
Screen slot size		
Depth to bottom of well	____/	____/
Type of backfill		
Depth to bottom of boring	____/	____/
Diameter of boring		

Diagram Not To Scale

Notes:
bgs = Below ground surface
MSL = Mean sea level
NA = Not applicable
ID = Inside diameter

WELL DEVELOPMENT LOG

Page ___ of ___

WELL NUMBER:_____ LOCATION:_____

DATE/TIME:_____/_____ WEATHER:_____

PROJECT/NUMBER:_____ REPORTED BY:_____

FIELD MEASUREMENTS

DEVELOPMENT STARTED:_____ DEVELOPMENT ENDED:_____

DEPTH TO WATER BELOW TOC (ft.):_____ WELL DEPTH BELOW TOC (ft.):_____

WATER COLUMN HEIGHT (ft.):_____ CASING DIAMETER (ft.): _____

3 WELL VOLUMES (gal.):_____ DISCHARGE VOLUME (gal.): _____

DEVELOPMENT METHOD/TOOLS USED:_____

Time	Cumulative Volume Purged (gal.)	pH	Conductivity (ms/cm)	T (°C)	Turbidity (NTU)	Comments (Water clarity, odor, etc.)

COMMENTS:

TIDEWATER, INC.

Project name:
Location:
Client:
Project Number:
Date:

SOIL SAMPLING LOG

Boring No.				**Elevation:**	
Sample Depth, Ft.	**Time**	**Moisture**	**PID Reading, ppm**	**Soil Description**	**Sample Sent to Analyt. LAB**
Bottom of boring at				Groundwater encountered at	

Boring No.				**Elevation:**	
Sample	**Time**	**Moisture**	**PID Reading, ppm**	**Soil Description**	**Sample Sent to Analyt. LAB**
Botto of boring at				Groundwater encountered at	

Boring No.				**Elevation:**	
Sample Depth, Ft.	**Time**	**Moisture**	**PID Reading, ppm**	**Soil Description**	**Sample Sent to Analyt. LAB**
Bottom of boring				Gorundwater encountered at	

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Chain-of-Custody

Form 202r8

WORKORDER #	

		SAMPLER		DATE		PAGE	of
PROJECT NAME		SITE ID		TURNAROUND		DISPOSAL	By Lab or Return to Client
PROJECT No.		EDD FORMAT					
		PURCHASE ORDER					
COMPANY NAME		BILL TO COMPANY					
SEND REPORT TO		INVOICE ATTN TO					
ADDRESS		ADDRESS					
CITY / STATE / ZIP		CITY / STATE / ZIP					
PHONE		PHONE					
FAX		FAX					
E-MAIL		E-MAIL					

Lab ID	Field ID	Matrix	Sample Date	Sample Time	# Bottles	Pres.	QC

*Time Zone (Circle): EST CST MST PST Matrix: O = oil S = soil NS = non-soil solid W = water L = liquid E = extract F = filter

For metals or anions, please detail analytes below.

Comments:		QC PACKAGE (check below)
		LEVEL II (Standard QC)
		LEVEL III (Std QC + forms)
		LEVEL IV (Std QC + forms + raw data)

Preservative Key: 1-HCl 2-HNO3 3-H2SO4 4-NaOH 5-NaHSO4 7-Other 8-4 degrees C 9-5035

	SIGNATURE	PRINTED NAME	DATE	TIME
RELINQUISHED BY				
RECEIVED BY				
RELINQUISHED BY				
RECEIVED BY				
RELINQUISHED BY				
RECEIVED BY				

ATTACHMENT C

PROJECT STANDARD OPERATING PROCEDURES

Standard Operating Procedure No. 1

Water Level Measurement

SOP No. 1 – Water Level Measurement

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for measuring water elevations in monitoring wells included in environmental monitoring programs. This procedure describes equipment and field procedures necessary to collect water elevation measurements. The well locations and frequency of measurement are specified in project-specific work plans and QAPPs.

2.0 EQUIPMENT AND MATERIALS

The equipment and materials necessary that may be used to measure water levels include:

- Electronic water level indicator capable of producing measurements to a precision of 0.01 feet
- 5 gallon buckets or equivalent for decontamination
- Brushes for decontamination
- Water Level Measurement Form or Groundwater Sampling Form
- Field notebook
- Chemical-free paper towels or Kimwipes
- Alconox soap
- Potable water
- Garden-type spray bottle filled with deionized or distilled water
- Appropriate health and safety equipment

3.0 WATER ELEVATION MEASUREMENT PROCEDURE

3.1 DISCUSSION

Generally, water elevation measurements are used to construct potentiometric surface maps. Therefore, water level measurements at a given site should be collected within a 24 hour period. The device used to measure water levels should be adequate to attain an accuracy of 0.01 feet. Water levels should be allowed to stabilize for a minimum of 24 hours after well construction and development before measurements are taken.

3.2 MEASUREMENT PROCEDURE

This section gives the steps to follow when measuring water levels. Note that appropriate health and safety steps should be implemented and health and safety equipment should be worn during well opening, well measurement, and decontamination.

- Before any measurement is taken, the water level indicator shall be decontaminated. Decontamination procedures are discussed in SOP – Sampling Equipment Decontamination.
- Confirm that the monitoring well is labeled and the location ID is visible on the protective casing and that the ID coincides with the expected location.

- After opening the well cover, measure the depth of the static water level and the total depth of the well using an electronic water level indicator. The measuring point for all the wells shall be the top of PVC or steel well casing. The measuring point will be marked by a notch or other mark in the PVC or steel casing. If no mark is present, measure from the top of the north side of the casing.
- The static water level and the depth of the well shall be measured with the indicator, logged on the field data sheet or field notebook as feet below top of casing (ft.-TOC), and verified before the indicator is removed from the well. Note any significant changes in water level, by comparing the most recent measurement with past measurements, if appropriate.
- The water level depth below the measuring point (ft.-TOC) will be subtracted from the measuring point elevation to determine the elevation of the static water level. If measuring point elevations are available at the time of water level measurement, the calculated water elevation (ft.-MSL) should be checked in the field to see that it is reasonable and the subtraction was performed correctly. If there is a significant discrepancy in the measured water level or calculated water elevation, the well should be measured again.
- All columns of field data sheets shall be completed, including time of measurement. If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA). A sample field data sheet for water elevation measurement is shown (Figure 1).

3.3 DECONTAMINATION

The water level indicator must be decontaminated before use, between wells, and at the conclusion of measurements. The probe will be decontaminated according to the procedure for decontamination of sampling equipment described in SOP – Sampling Equipment Decontamination.

4.0 DOCUMENTATION

Documentation of observations and data acquired in the field will provide information on the activities concluded and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages, and on field data sheets.

4.1 FIELD DATA SHEET FOR WATER LEVEL MEASUREMENTS

A field sampling data sheet for groundwater samples will be completed at each sampling location (sample attached). If items on the sheet do not apply to a specific location, the item will be labeled as not applicable (NA). The information on the data sheet includes the following:

- Well number
- Field book reference number

SOP No. 1 – Water Level Measurement

- Field personnel
- Well I.D.
- Date and time of measurements
- Sample identification number
- Water level (ft.-TOC)
- Static Water Elevation Data

The measurement point elevation (ft.-MSL) should be filled in if it has been determined at the time of measurement. If it is not known the form will be completed at a later time when further information is available. Any irregularities or problems that may have a bearing on sampling quality should be noted in the field.

Standard Operating Procedure No. 2

Groundwater Sampling

SOP No. 2 – Groundwater Sampling

MONITORING WELL SAMPLING

1.0 OBJECTIVE

The purpose of this document is to define the standard operating procedure (SOP) for measuring collecting groundwater samples during environmental monitoring programs. This procedure describes equipment and field procedures necessary to collect groundwater samples using several methods. Procedures for groundwater sampling from a monitoring well are designed to obtain a sample for chemical analysis that is representative of natural aquifer conditions. Samples will be collected using disposable bailers or low-flow electric or pneumatic pumps.

2.0 METHODOLOGY

2.1 LOW-FLOW SAMPLING USING A SUBMERSIBLE PUMP

Low-flow purging is the preferred method because it results in (1) minimal loss of VOCs, (2) minimal mixing of chemically distinct zones, (3) minimal production of artificial turbidity and oxidation, and (4) minimal production of potentially contaminated purge water. The micropurging procedures described in this section are adapted from those procedures recommended by the *EPA in Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (EPA 1996)*.

Low-flow purging will be conducted using an electric submersible pump, or bladder pump, equipped with a positive-foot check valve to prevent purge water from flowing back into the well. Teflon®-lined polyethylene tubing will be used for each monitor well. The water quality parameters listed in Table 1 will be regularly measured during purging. All water quality monitoring instruments will be calibrated and measurements collected in accordance with the procedures specified in SOP – Instrument Calibration.

The following procedure will be used when purging wells using the low-flow method.

1. Decontaminate any equipment (i.e., water level indicators, pumps, and down-well probes) before placing in the well in accordance with the procedures specified in SOP – Sampling Equipment Decontamination.
2. Measure the water level in the well in accordance with the procedure described in SOP – Water Level Measurement, before beginning purging. Record the water level on the Monitor Well Sampling Form. Do not measure the total well depth before low-flow sampling so that any sediment in the bottom of the well, if present, will not be disturbed and entrained in the water column.
3. Lower the pump into the well in a controlled manner until the pump intake is situated at the approximate midpoint of the saturated screened interval, as determined from well completion logs, or midpoint of saturated screen interval (information concerning total depth of well and screened interval can be obtained from the Monitoring Well Construction Form for each well). Do not allow the pump to touch or settle on the well

bottom to prevent excess agitation and entrainment of sediment that may have accumulated in the bottom of the well.

4. Start the pump and determine the pumping rate by measuring the time required to fill a calibrated container. Adjust the pumping rate so that is does not exceed 0.5 liter (0.13 gallon) per minute in order to minimize the potential for stagnant water to be drawn into the pump intake.
5. Verify that the pumping rate is low enough by measuring the water level in the well every 3 to 5 minutes during purging. Adjust the pumping rate to maintain a drawdown of the water from the pre-sampling level at less than 0.1 meter (0.3 foot), if possible. When parameters are collected within in-line flow-through cells, the frequency of measurements shall be equal to the time required to provide complete exchange of the cell volume. If the drawdown cannot be maintained at less than 0.1 meter (0.3 foot) by reducing the pumping rate, note the condition on the Monitoring Well Sampling Form and continue to purge the well until (1) parameters and drawdown stabilize, (2) parameters stabilize and three casing volumes are removed, or (3) the well is purged dry.
6. Measure water quality parameters (as indicated in Table 1) at the start of purging and then every 3 to 5 minutes thereafter to determine when the well has been adequately purged, as discussed below.
7. Purge the well until either (1) the water quality parameters stabilize over three consecutive readings, (2) three casing volumes of water are removed, (3) the well is purged dry, (4) or the well has been pumped for at least 2 hours without drawdown. The stabilization criteria and proposed measurement methods for each of the parameters is presented in Table 1. If the well is purged dry, samples from the well can be collected when a sufficient volume of water has recharged to begin filling sample containers.
8. Collect samples as described in Sample Collection section below.
9. Sample containers will be labeled, and packed on ice for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management.
10. Contain and handle all purge water removed from well in accordance with the IDW management procedures specified in SOP – IDW Management.
11. Decontaminate all sampling and drilling equipment per SOP – Sampling Equipment Decontamination.

Table 1
WATER QUALITY PARAMETER STABILIZATION CRITERIA
AND PROPOSED MEASUREMENT METHODS

Parameter	Stabilization Criteria	Measurement Method[1]
Dissolved Oxygen	± 10 percent	Water quality meter
pH	± 0.2 units	Water quality meter
Specific Conductance	± 10 percent	Water quality meter
Temperature	± 10 percent	Water quality meter
Oxidation-reduction potential (ORP)	± 10 percent (or ± 10 units)	Water quality meter
Turbidity	50 NTU (or ± 10 percent)[1]	Water quality meter

Notes:

1 When possible, dissolved oxygen and ORP should be measured with a flow-through cell or down-well probe to avoid atmospheric influences. Other parameters may be measured using a flow-through cell or alternate method (i.e., Horiba U10 or equivalent).
2 For Turbidity, three consecutive readings within 10 percent is acceptable if the 5 NTU turbidity goal cannot be reasonably achieved. For ORP, ± 10 units is acceptable if ± 10% cannot be achieved (i.e., ORP is very close to zero and therefore ± 10% of the current reading is a very small number compared with the total ORP range).

NTU Nephelometric turbidity units

2.2 SAMPLING USING A BAILER

In the event that a monitor well cannot be purged using a submersible (or equivalent) pump, the alternate method will be to purge the well using a bailer. A new Teflon® bailer, equipped with new nylon line, will be used for purging each monitor well. Water quality parameters, as indicated in Table 1, will be regularly measured during purging. All water quality monitoring instruments will be calibrated and measurements collected in accordance with the procedures specified in SOP – Equipment Calibration.

The following procedure will be used when purging wells using the bailing method:

1. Measure the water level and total well depth in accordance with the procedures specified in SOP – Water Level Measurement before beginning purging. Record the water level and total depth on the Monitor Well Sampling Form.
2. Determine the height of the water column in the well by subtracting the depth to water from the total depth of the well. Record the water column height on the Monitor Well Sampling Form.
3. Calculate the well casing volume and enter it on the form. The casing volume is calculated as:

$$V = H \times F$$

Where:

V = one casing volume (gallons)

H = height of water in well (total well depth – depth to water) (feet)

F = water volume per foot of well casing (gallons/foot)

The volume of water per foot of well casing (F) can be calculated as:

$$F = \pi (d/2)^2 \times 7.48 \text{ gallon/foot}^3$$

Where d is the inside diameter (in feet) of the well casing.

4. Calculate the total purge volume as three times the casing volume (3V). Enter this quantity on the sampling form.
5. Measure water quality (as indicated in Table 1) at the start of purging and then every casing volume thereafter to determine when the well has been adequately purged, as discussed below. Monitor the water volume purged from the well by filling a calibrated container, such as a five-gallon bucket.
6. Purge the well until (1) the water quality parameters stabilize over three consecutive readings and a minimum of three casing volumes of water have been removed from the well, or (2) the well is purged dry. The stabilization criteria and proposed measurement methods for each of the parameters are presented in Table 1. If a well is purged dry, samples from the well will be collected as described in the Sample Collection section, when a sufficient volume of water has recharged to begin filling sample containers.
7. Sample containers will be labeled, and packed on ice for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management.
8. Contain and handle all purge water in accordance with the IDW management procedures specified in SOP – IDW Management.

2.2.1 Sample Collection

Purging and sample collection will not be conducted within 24 hours of monitoring well development. Groundwater samples will be collected by pumping water through the discharge tubing (or pouring from the bailer) directly into the sample containers. The discharge tubing will not be allowed to contact the sample container.

Before collecting groundwater samples, the sampler will don clean, nitrile gloves. Samples will be collected using a submersible (or equivalent) pump (or bailer if appropriate).

In general, samples will be collected for laboratory analysis from the most volatile to the least volatile. The specific order of sample collection will be as follows:

- VOCs
- Other organic compounds
- Inorganic compounds
- Metals
- Other analyses

Required sample containers, preservation methods, volumes, and holding times are given in the QAPP. Field parameters will be measured as described in Table 1. Sampling equipment will be decontaminated in accordance with SOP – Sample Equipment Deconatmination.

If dense nonaqueous phase liquids (DNAPLs) are suspected, a bailer will be lowered to the bottom of the well before purging, retrieved, and observed for the presence of DNAPLs. Disposable nylon rope will be used to lower and retrieve the bailers. A new length of nylon rope will be used for each well, and the rope will be disposed of following the sampling activities. Each bailer will be equipped with a dedicated stainless steel- or Teflon-coated leader so that the nylon rope will not contact the water in the well.

The sample will be collected from the pump using a slow, controlled pour down the side of tilted sample vial to minimize volatilization. When collecting samples for VOCs, the pump discharge rate should be kept low, preferably 0.1 to 0.3 L/min. When sampling for VOCs, the sample vial will be filled until a meniscus is visible and immediately sealed. When the bottle is capped, it will be inverted and gently tapped to ensure no air bubbles are present in the vial. If after the initial filling bubbles are present, the vials will be discarded and the VOC sampling effort will be repeated. Refilling of vials will result in loss of preservatives. After the containers are sealed, sample degassing may cause bubbles to form. These bubbles will be left in the container. These samples will never be composited, homogenized, or filtered.

Sample containers will be labeled, and packed on ice for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management.

2.3 SAMPLING GROUNDWATER WELLS USING A PASSIVE DIFFUSION SAMPLER

A passive diffusion sampler (PDS) consists of low-density polyethylene (LDPE) tubing, containing laboratory-grade water, and sealed at both ends. LDPE is permeable to some types of volatile organic compounds (VOCs). Therefore, VOCs in groundwater (if present) will diffuse through the LDPE and into the laboratory-grade water until the VOC concentration within the PDS is in equilibrium with the VOC concentration in the surrounding groundwater. PDS' will be placed in the selected wells a minimum of two weeks prior to the start of the quarterly sampling event. PDS' may either be prepared in the field by the LTM contractor or may be obtained, already constructed, from an analytical laboratory.

The following procedure will be used if PDS' are prepared in the field:

- Two-inch diameter flat LDPE tubing will be cut into 18 inch long pieces using a clean oil-free blade and heat-sealed on one end to form tubes.

- The tubes will be filled with ASTM water, being careful to avoid large air bubbles.
- The open ends of the tubes will be heat-sealed on a diagonal line (for ease of pouring later).
- The tubes may be encased with LDPE mesh tubing to provide abrasion resistance.
- The PDS' should be malleable enough to bend at least 90 degrees without bursting and have approximately 12 to 13 inches of ASTM water enclosed. Samplers not meeting this standard, or leaking samplers, will be discarded.
- The constructed samplers will be stored in a clean container for transport to wells for installation.

PDS' will be hung from a measured length of nylon rope or stainless steel cable sufficient to situate the middle of the sampler at the approximate midpoint of the well screen (if the water table is above the top of the well screen), or at the midpoint of the water column zone (if the water table is below the top of the well screen). A brass or stainless steel weight will be hung from the bottom of the PDS to prevent the sampler from floating above the desired sample interval. Multiple PDS' may be used if additional sample volume is required for quality control samples. The well cover will be closed and the PDS left in the well until the next scheduled sampling event, or for a minimum of two weeks before sample collection.

PDS' will be removed and the tubes cut open along the diagonal heat-seal with a clean blade. The contents of the tubes will be slowly poured into 40 mL vials to reduce sample agitation. Sample containers will be labeled, and packed on ice for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management.

After retrieving the PDS from the well, the person(s) collecting the sample will record one measurement of field parameters by lowering the field parameter sensor with a protective sleeve down into the well so that it is at least five feet below the water surface. The person(s) will wait at least two minutes before recording each of the parameters, including: pH, ORP, temperature, dissolved oxygen, and specific conductance.

SOP No. 2 – Groundwater Sampling

SAMPLING WITH DIRECT PUSH

1.0 OBJECTIVE

Direct push (DPT) sampling involves advancing a sampling probe to a desired depth to collect a groundwater sample. Procedures will be followed to ensure that samples collected are representative of the groundwater within a specific interval. Due to the narrow diameter of the DPT borings, direct push locations will be sampled using a portable low-flow peristaltic pump or disposable bailer.

2.0 EQUIPMENT AND MATERIALS

- Portable low-flow peristaltic purge/sample pump with polyethylene and silicon tubing
- Electronic water level indicator
- Multi-parameter water quality meter
- Decontamination supplies
- Sample containers, preservatives, coolers, sample labels, and ice (as specified in the QAPP)
- Groundwater sampling forms and chain-of-custody forms
- Waste containers as specified in Work Plan
- Health and safety equipment as specified in the Site-Specific Health and Safety Plan

3.0 METHODOLOGY

1. Ensure that sample equipment is operating properly and calibrated by following the equipment manuals provided by the equipment manufacturer.
2. Ensure that all DPT and sampling equipment has been properly decontaminated (SOP – Sampling Equipment Decontamination)
3. Using the DPT drill rig, advance a sample probe by direct hydraulic pressure or by using a slide or rotary hammer, to the desired depth.
4. When the probe is at the proper depth, raise the tooling to open the sample port of the sample probe.
5. Measure the depth to water and total probe depth with a decontaminated electronic water level indicator and record on Groundwater Sampling Form (See SOP – Water Level Measurement).
6. Insert disposable polyethylene tubing through the drill tooling to the screened interval and withdraw groundwater using the peristaltic pump.
7. Collect the appropriate analytical samples including quality assurance/quality control (QA/QC) samples as specified in the QAPP, collecting VOC samples first VOC samples may be collected using a disposable bailer). If air bubbles or gaps of air within the sample line are observed, the pumping rate will be reduced in an effort to minimize or eliminate air in the line.
8. Collect additional sample and measure and record field parameters as described in Table 1.

9. If a sufficient amount of water is not has not recharged into the boring to allow for collection of a complete set of samples, a temporary well may be set so the boring can be sampled when sufficient water has entered the boring.
10. Sample containers will be labeled, and packed on ice for shipment to the analytical laboratory under appropriate chain of custody protocol and SOP – Sample Handling and Management.
11. After the sample has been collected, remove the drill tooling or temporary well from the boring and abandon the borehole (See SOP – Borehole Abandonment).
12. Contain and handle all purge water removed from well in accordance with the IDW management procedures specified in SOP – IDW Management.
13. Decontaminate all sampling and drilling equipment per SOP – Sampling Equipment Decontamination.

Standard Operating Procedure No. 3

Sample Handling and Management

SOP No. 3 – Sample Handling and Management

1.0 PURPOSE AND SCOPE

The purpose of this document is to define the standard operating procedure (SOP) for sample management including sample handling, documentation, and analysis for environmental samples collected for chemical analyses including: sediment, soil, surface water and groundwater. This procedure is intended to be used together with the other SOPs.

2.0 EQUIPMENT AND MATERIALS

The following equipment will be used for sample management:

- Shipping forms
- Sample containers
- Ziploc bags
- Ice
- Tape (clear and strapping)
- Scissors/knife
- Cooler/ice chest
- Custody seal
- Garbage bags
- Waterproof Pens
- Chain of Custody (COC) Forms
- Sample Labels
- Logbook
- Gloves
- Preservative (if necessary)
- Packing material
- Trip blank (as necessary)
- Temperature blank

3.0 PROCEDURES FOR SAMPLE HANDLING, DOCUMENTATION, AND ANALYSIS

3.1 SAMPLE LABELING

All sample labels should be filled out with waterproof ink. Soil and water sample labels may be supplied by the laboratory. For soil and sediment samples collected in jars and sample bottles for groundwater and surface water analyses, sample labels may be completed and attached prior to sample collection. Labels may be partially completed prior to sample collection. The date and time should not be completed until the time of sample collection. At a minimum, each label shall contain the following information:

- Project/Facility Name
- Grab or composite sample
- Sampler's company affiliation

- Date and time of sample collection
- Analyses required
- Preservation used
- Sampler's initials
- Filtered (if applicable)
- Sample identification (see Section 5.2 below)

3.2 SAMPLE NOMENCLATURE SCHEME

The sample identification (ID) varies significantly with each project. At minimum the sample ID should contain enough information to be correctly associated with a specific sampling location. The sample ID shall also be recorded on the sample form for the respective location. Additionally, Quality Control/Quality Assurance (QA/QC) samples should contain a sample ID such that the laboratory would not know it is a QA/QC sample.

3.3 SAMPLE HANDLING

This section discusses proper sample containers, preservatives, and handling and shipping procedures. The QAPP also summarize the information contained in this section and also includes the sample holding times for each analysis.

3.3.1 Sample Containers

Certified, commercially clean sample containers shall be obtained from the contract analytical lab. If appropriate, the bottles shall be labeled by the laboratory to indicate the type of sample to be collected. Required preservatives shall be prepared and placed in the bottles for aqueous analyses at the laboratory prior to shipment to the site.

3.3.2 Sample Preservation

With the exception of samples that are to be hand-delivered to the laboratory during the day of sample collection, samples will be stored on ice to obtain a temperature of 4ºC in an insulated cooler immediately following sample collection. Samples delivered to the laboratory during the day of sample collection are acceptable if they have been placed on ice in an insulated cooler but have not yet reached a temperature of 4ºC. Soil and sediment samples do not require additional preservation. As noted above, sample containers for aqueous samples will be obtained from the laboratory containing the appropriate preservatives.

3.4 SAMPLE SHIPPING

Sample containers will wrapped in protective packing material (if appropriate). Samples will then be placed in a cooler with ice for shipment to the laboratory. The drain on the cooler shall be taped shut. Samples collected in glass containers will be packed in foam liners and/or

bubble wrap to ensure that no breakage occurs during shipment. A temperature blank will be included in each cooler. Samples will be sent to the analytical laboratory via Federal Express or equivalent. Shipping receipts should be retained for documentation and sample tracking.

A completed chain-of-custody (COC) form for each cooler will be placed in a Ziploc bag and taped to the inside of the cooler lid. Coolers will be wrapped with packing tape at two locations to secure lids. Signed and dated custody seals shall be placed on the outside of each cooler in two places in such a manner as to allow detection of tampering (e.g., the seals must be broken to open the cooler).

3.5 HOLDING TIME REQUIREMENTS

The holding time is specified as the maximum allowable time between sample collection and analysis and/or extraction, based on the analyte of interest, stability factors, and preservation methods. Allowable holding times for chemical analysis parameters are listed in the QAPP. Samples should be sent to the laboratory after collection in sufficient time to allow the laboratory to meet holding time requirements.

4.0 QUALITY CONTROL REQUIREMENTS

QC requirements relevant to analysis of environmental samples shall be followed during analytical activities to meet the quality objectives and criteria. The purpose of the QC program is to produce data of known and documented quality that satisfy the project objectives and that meet or exceed the requirements of the standard methods of analysis.

4.1 QC SAMPLES

A number of QC samples will be employed to assess various data quality parameters, such as representativeness of the environmental samples, the precision of sample collection and handling procedures, the thoroughness of the field equipment decontamination procedures, and the accuracy of laboratory analysis. Types of QC samples are discussed below.

4.1.1 Matrix Spike/Matrix Spike Duplicate

Matrix spike (MS) and matrix spike duplicate (MSD) samples are prepared by spiking additional aliquots of sample with known concentrations of all project target analytes.

The sample to be used for the MS/MSD analyses shall be designated on the chain of custody and additional sample volume shall be submitted, as necessary. The MS/MSD results are used to document the bias of a method due to sample matrix. Consequently, MSs and MSDs are not used to control the analytical process. Minimum numbers of MS and one MSD samples are indicated in the project-specific QAPP, generally one for every 20 environmental samples of a given matrix. Alternately, a laboratory may prepare and analyze a MS sample and a laboratory duplicate sample as discussed below. Analysis of a MS/MSD or MS/LD sample set to assess matrix effects on accuracy and precision is typically dependent on the analyte class (e.g., inorganic vs. organic) and the likelihood of detecting the target analyte.

4.1.2 Rinsate Blank

A rinsate blank is a sample of ASTM Type II reagent grade water poured into or over or pumped through the sampling device, collected in a sample container, and transported to the laboratory for analysis. ASTM Type II reagent grade water obtained from the laboratory is used to prepare the rinsate blank sample. Rinsate blanks are used to assess the effectiveness of equipment decontamination procedures used to prevent cross-contamination between sampling locations. The frequency of collection for rinsate blanks is indicated in the project-specific QAPP, generally a minimum of 1 rinsate blank for every 20 environmental samples collected with a given type of sampling equipment, and only for sampling equipment which is decontaminated and reused to collect environmental samples. Rinsate blanks will be prepared in a manner identical to samples and shall be analyzed for all laboratory analyses requested for the environmental samples collected at the site using the subject equipment. Rinsate blanks are not necessary for disposable or dedicated sampling equipment.

4.1.3 Trip Blank

The trip blank consists of a VOC sample vial filled in the laboratory with ASTM Type II reagent grade water, transported to the sampling site, handled in the same manner as an environmental sample and returned to the laboratory for analysis. Trip blanks are not opened in the field. Trip blanks are prepared only when VOC samples are taken and are analyzed only for VOC analytes. Trip blanks are used to assess the potential introduction of contaminants from sample containers or during the transportation and storage procedures. One trip blank shall accompany each cooler containing samples for VOC analysis that is sent to the laboratory.

4.1.4 Field Duplicates

A field duplicate sample is a second, discrete sample volume collected at the same location as the original sample (homogenization is not performed between the original sample and the field duplicate). Aqueous field duplicate samples are collected from successive volumes from the same sample source and device (e.g., bailers). Sediment and soil field duplicates are collected in succession from the same sample source and device. Individual analytes for the primary and duplicate groundwater samples are to be collected in order (e.g. VOC primary then VOC duplicate, etc.) so that a long period of time does not pass between collection of each analyte. Field duplicate samples are collected using identical recovery techniques, and treated in an identical manner during storage, transportation, and analysis. The sample containers are assigned an identification number in the field such that they cannot be identified (blind duplicate) as field duplicate samples by laboratory personnel performing the analysis.

Field duplicate sample results are used to assess precision of the sample collection process and the heterogeneity of the medium sampled. The frequency of collection for field duplicates is indicated in the project-specific QAPP, generally a minimum of one field duplicate sample from each group of 10 environmental samples of a given matrix. Specific locations for collection of field duplicate samples may be designated prior to the beginning of sample collection.

SOP No. 3 – Sample Handling and Management

5.0 DOCUMENTATION AND TRACKING

5.1 FIELD NOTES

Documentation of observations and data acquired in the field will provide information on the acquisition of samples and also provide a permanent record of field activities. The observations and data will be recorded with waterproof ink in a permanently bound weatherproof field logbook with consecutively numbered pages and, if applicable, on field sampling data sheets.

The information in the field logbook will include the following as a minimum. Unless information is recorded on a field sample collection form and that form is cross referenced in the logbook entry. Additional information is included in the specific SOPs regarding the appropriate data sheets.

- Project name
- Location of sample
- Sampler's signature
- Date and time of sample collection
- Sample identification numbers and sample depth (if applicable)
- Description of samples (matrix sampled), composite or grab sample
- Description of QA/QC samples (if collected)
- Sample methods or reference to the appropriate SOP
- Field observations
- Decontamination information
- Calibration information
- Personnel present
- Method of shipment
- Any deviations from SOPs
- Any information pertaining to the sample that is not noted of the sample form.

If samples are held for an extended period of time (i.e., inadvertently missed Fed-Ex pick up), field personnel will document all sample handling and custody in the field logbook.

5.2 CHAIN-OF-CUSTODY FORM

A record of each sample collected will be indicated on a COC form. Every sample in the coolers shall be covered by the COC form(s) accompanying the coolers. Coolers may contain a single COC covering only the samples in that cooler or may contain copies of the COC that covers all of the samples in all of the coolers. One cooler must contain the original COC. The COC form will provide an accurate written record which can be used to trace the custody of all samples from the time of collection through data analyses and reporting.

The following will be specified for each sample on the COC form as a minimum:

- Sample ID
- Sample date

- Sample time
- Requested analysis
- Number of containers
- Sampler's signature or initials
- Preservation technique
- Sample type (i.e., medium)

Also recorded on the COC is the signature of the person relinquishing custody, the date and time that custody was relinquished, the name and address of the laboratory, and the name and phone number of a contact person regarding the shipment.

A sample is considered in custody if it is:

1. In one's actual possession
2. In view, after being in physical possession
3. Locked so that no one can tamper with it, after having been in physical custody
4. In a secured area

The person responsible for custody of the sample prior to delivery of the samples to the laboratory will sign the COC form, retain the last copy of the three-part COC form, document the method of shipment, and send the original and the second copy of the COC form with the sample (taped in a Ziploc bag to inner cooler lid). Upon receipt at the laboratory, the person receiving the samples will sign the COC form and return the second copy to the Project Manager or Quality Assurance Manager or specified designee. Copies of the COC forms and all custody documentation will be received and kept in the central files. The original COC forms will remain with the samples until final disposition of the samples by the laboratory. The analytical laboratory may dispose of the samples in an appropriate manner 60 to 90 days after data reporting. After sample disposal, a copy of the original COC will be sent to the Project Manager or Quality Assurance Manager or specified designee by the analytical laboratory to be incorporated into the central files.

Standard Operating Procedure No. 4

Sampling Equipment Decontamination

SOP No. 4 – Sampling Equipment Decontamination

1.0 OBJECTIVE

Decontamination is performed as a quality assurance measure and safety precaution. It helps prevent cross-contamination among samples and helps maintain a clean working environment for the safety of field personnel.

2.0 EQUIPMENT AND MATERIALS

- Cleaning liquids such as soap or detergent solutions (Alconox or equivalent), potable water and distilled water
- Cleaning brushes
- Cleaning containers, such as plastic buckets or tubs
- Pump sprayers for dispensing rinse waters
- A high-pressure hot water sprayer for cleaning large equipment (e.g., drill rods)
- Waste containers
- Health and safety equipment as outlined in the Site-Specific Health and Safety Plan

3.0 METHODOLOGY

Small, reusable equipment is decontaminated primarily by rinsing with liquids that include soap or detergent solutions, potable water, and distilled water. Steam cleaning may he used whenever visible contamination exists on large machinery or vehicles. Following decontamination, if the equipment is not to be reused immediately, it should be stored and protected from recontamination.

3.1 PRE-SAMPLING DECONTAMINATION ACTIVITIES

1. Don the appropriate personal protective equipment, including nitrile gloves, as specified in the Site-Specific Health and Safety Plan and as required for the specific work area.
2. Assemble containers and equipment for decontamination.
3. Decontaminate new equipment or equipment not previously decontaminated before use. Disposable equipment, including polyethylene tubing and bailers, do not require decontamination prior to use.
4. Rinse equipment not previously decontaminated and appropriately protect from recontamination before the next use.

3.2 DECONTAMINATING SAMPLING EQUIPMENT

1. Remove solid particles from the equipment or material by brushing and rinsing with potable water. This will remove gross contamination.
2. Wash equipment with a brush and a phosphate-free detergent solution (Alconox or similar laboratory detergent).
3. Rinse equipment thoroughly with potable water.
4. Triple rinse the equipment with distilled water.

5. Unless the equipment is going to be used immediately protect it from recontamination before the next use.

3.3 DECONTAMINATING LARGE EQUIPMENT

Drilling equipment (rigs, drill rods, augers, rods, bits, casing, screen. etc.), downhole logging equipment, and other large pieces of field equipment may be high-pressure steam-cleaned before and after use. Steam cleaning will be performed at an appropriate decontamination area specified by the field supervisor. The decontamination area shall be capable of containing decontamination fluids and solids. The decontamination fluids shall be managed in accordance with SOP – IDW Management.

Additionally, the drilling subcontractor has the responsibility of making the drilling rig free of leaks (i.e., hydraulic fluid, oil, gas. etc.) that could contaminate the boreholes. Grease may be sparingly used on rod shoulders to ease rod breaking upon completion of a borehole. Rod joints should be wiped with a clean cloth to minimize the amount of grease on the exterior of the rod.

4.0 COMMENTS

Decontamination is critical for maintaining the integrity of the sampling program. Check equipment carefully prior to sampling, and if there is any doubt about the effectiveness of the decontamination, repeat the decontamination process as an extra precaution. Decontamination fluids will be containerized and disposed of following the procedures provided SOP – IDW Management. Decontamination procedures shall be documented in the field log book.

Standard Operating Procedure No. 5

Soil Sampling for Chemical Analysis

SOP No. 5 – Soil Sampling for Chemical Analysis

1.0 OBJECTIVE

Soil samples will be collected for field screening and chemical analysis to help characterize the source areas and to determine the nature and extent of contamination in soil. Soil samples will be collected during direct push activities and monitoring well drilling.

2.0 EQUIPMENT AND MATERIALS

- Appropriate number and types of sample containers
- Precleaned stainless steel sampling utensils (See SOP – Sampling Equipment Decontamination)
- Sample coolers and ice.
- Appropriate field documentation forms and labels and an indelible ink pen.
- Sampling equipment (split--spoons samplers).
- Decontamination equipment.
- Waste containers.
- Health and safety equipment, as specified in the Health and Safety Plan.

3.0 METHODOLOGY

Soil samples collected by direct push or split-spoon methods will be collected as follows (See SOP – Direct Push Drilling and SOP – Hollow Stem Auger Drilling):

a) Retrieve sampler from borehole and remove drive shoe and head assembly. Open the sampler carefully to avoid disturbing the sample.

b) Once the sampler has been opened, the length of the sample will be screened with a photoionization detector (PID), and the reading will be recorded on the drilling log.

c) The upper portion of soil in the sampler potentially represents material that has fallen from above or has been scraped from the sides of the auger hole; therefore, this portion is not representative of the sampling interval and should be discarded.

d) Samples for volatile organic compound (VOC) analysis will be collected first to minimize the potential for volatilization. A sufficient amount of soil will be collected and transferred directly to VOC sample containers using a stainless steel utensil. The sample will be packed to completely fill the container and reduce the amount of headspace, which will minimize the loss of volatile compounds. Teflon-lined septum lids will be immediately secured on the sample containers.

e) Collect samples for additional analysis next.

f) Complete the sample labels in accordance with SOP – Sample Handling and Management and place them on the jars.

g) Place protective packing on the sample jar.

h) Decontaminate sampling equipment in accordance with SOP – Sampling Equipment Decontamination.

i) Collect and manage wastes as specified the waste management plan.

4.0 COMMENTS

Following standard practice, the soil samples for VOC analysis will be collected before any logging or other sample handling is conducted. Due to the nature of VOCs, it is critical to collect these samples as quickly as possible and to immediately place them in a cooler with ice. VOC soil samples are collected via either TerraCore or EnCore sampling kits. Refer to the project-specific work plan for the specific sampling protocol. The TerraCore or EnCore samplers for VOC soil samples should be pre-chilled in a cooler with ice prior to filling. This practice will further reduce the potential for volatilization during sample collection. It may be necessary to advance an additional boring for the purpose of lithologic logging.

4.1 LITHOLOGIC LOGGING AND GEOTECHNICAL ANALYSIS

Lithologic logging will be performed to define the subsurface geology. Selected samples will be collected to send to the laboratory for quantitative geotechnical analysis. See SOP – Borehole Logging for further information.

Standard Operating Procedure No. 6

IDW Management

SOP No. 6 – IDW Management

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides technical guidance and methods that will be used for the handling, management, and disposal of investigation derived waste (IDW) encountered or generated during environmental investigation activities. This SOP gives descriptions of equipment, field development procedures, field data collection, and personnel responsibilities.

2.0 EQUIPMENT AND MATERIALS

The following equipment and materials may be needed for IDW management:

Personal protective equipment (PPE) as outlined in the HSP

- Decontamination equipment and supplies (e.g., wash/rinse tubs, brushes, alconox, plastic sheeting, paper towels, sponges, baby wipes, garden-type water sprayers, large plastic bags (minimum 0.55 mil), potable water, distilled water and/or deionized water)
- Department of Transportation (DOT)-rated 55-gallon drums or other approved containers for containing soil cuttings, decontamination water, and formation water
- Drum/bung wrench and drum funnel
- Heavy equipment forklift or vehicle with drum grappler (as necessary)
- Laboratory-supplied sample containers
- Photoionization detector (PID) or flame ionization detector (FID)
- Wood pallets (as necessary)
- Non-porous (e.g.. stainless steel) shovels
- Polyethylene tanks (as necessary)
- Field notebook and waterproof and permanent marking pens

3.0 PROCEDURES

It is anticipated that both non-liquid and liquid IDW will be generated or encountered during field activities. IDW generated during the field investigation is expected to include:

- Soil cuttings and other soil wastes generated during sampling
- Well development and purged water
- Wash and rinse waste from decontamination activities
- Used PPE and other non-soil solid wastes

3.1 SOIL IDW

- Soil cuttings generated during drilling and soil sampling will be initially assessed in the field to determine if the potential exists for contamination with site-related COCs. If the soils are generated outside of the original limits of the site (e.g., outside the fence line), they will be screened for volatile contaminants with a PID meter. If no readings are above background level, the soil will be spread on the ground at the point of generation. If PID readings indicate potential contamination, the soil will be

placed into DOT-rated 55-gallon drums, or appropriately sized containers at the point of generation pending further analysis. Soil cuttings generated inside the original limits of the site will be containerized pending further analysis.

- Mixing of the cuttings from several borings or sampling locations is permissible in order to fill the drums.
- When drums or containers are full, or daily activities are completed, the drum lids and rings will be fastened. Full drums or containers will be transported to the designated IDW accumulation area on a regular basis to avoid accumulation of drums or containers at individual investigation sites for extended periods of time.
- Drums will be stored on pallets at the designated IDW accumulation area. Drums will be segregated to separate soil from liquid IDW.
- Drums will be sealed and labeled with permanent markings (using paint pens or drum labels) with the following information:
    - Source: the boring(s), well, or site identification number
    - Matrix (e.g., soil, water)
    - Sample interval
    - Fill date
    - Drum identification number
    - Contractor
    - Point of contact with phone number
    - Labeled "Contents Pending Analysis"
- If large volumes of soil IDW will be generated, soil IDW will be transferred from the drums into roll-off bins (lined and covered) located within the designated IDW accumulation area.
- If no associated investigation sample results exist, a composite soil sample will be collected from the soil IDW drums by collecting a drive or hand auger sample from each of the drums. The sample material from all of the drums will be composited into a single sample that will be used to characterize and dispose of the IDW.

3.2 LIQUID IDW

- Well development, purge, abandonment, and decontamination water will be contained in DOT-rated drums, or in appropriately sized watertight containers, at the point of generation.
- When drums or tanks are full, or daily activities are completed, the containers will be sealed: for example, drum lids and rings will he fastened.
- Drums will be stored on pallets at the designated IDW accumulation area. Drums will be segregated to separate soil from liquid IDW.
- Drums will be sealed and labeled with permanent markings (using paint pens or drum labels) with the following information:
    - Source: the boring(s), well, or site identification number
    - Matrix (e.g., soil, water)
    - Sample interval
    - Fill date

  - Drum identification number
  - Contractor
  - Point of contact with phone number
  - Labeled "Contents Pending Analysis"
- If large volumes of water will be generated, the water will be transferred into an appropriately-sized polyethylene tank.
- If no associated investigation sample results exist, a composite water sample will be collected from the water IDW drums. The sample will be used to characterize and dispose of the IDW.

3.3 PPE AND DISPOSABLE INVESTIGATION EQUIPMENT

- The plan for managing used PPE and other non-soil solid waste generated during field activities (e.g., sample handling) will be segregated separately and placed into dedicated heavy duty plastic bags or containers (e.g. drums)
- Potentially contaminated PPE or disposable investigation equipment will be decontaminated prior to placement in the plastic bags or containers, if warranted.
- Decontamination procedures consist of brushing off, or using small amounts of water to scrub off, gross potential contamination.

3.4 DISPOSAL OF IDW

IDW will be disposed of in accordance with federal, state, and local regulations.

4.0 DOCUMENTATION

Project staff are responsible for thoroughly documenting IDW handling and disposal activities. Personnel will be responsible for documenting the collection, transportation, labeling (if applicable), and staging or disposition of IDW. A Waste Inventory Tracking Form is to be completed if necessary. Documentation should include the following:

- Project Name
- Names of personnel
- Site location
- Type of activities
- Date waste generated
- Boring, well, or site number(s)
- Matrix
- Type of container(s)
- Estimated volume
- Disposition of contents
- Comments (field evidence of contamination (e.g., PID reading, odors)
- Any variance to procedures described in this SOP

Standard Operating Procedure No. 7

Drilling and Lithologic Logging

SOP No. 7 – Drilling and Lithologic Logging

OBJECTIVE

Lithologic logging will be performed to define the subsurface geology. Selected samples will be collected to send to the laboratory for quantitative geotechnical analysis. Soils will be described using the Unified Soils Classification System (American Society for Testing and Materials [ASTM] Designation D 2488-09a: Standard Practice for Description and Identification of Soils [Visual-Manual Procedure]). This SOP serves as a supplement to the site-wide and investigation area specific workplans and field sampling plans (FSPs), and is intended to be used in conjunction with the other SOPs.

EQUIPMENT AND MATERIALS

- Monitoring equipment and personal protective equipment (PPE) as outlined in the site-specific HSP
- Air or fluid rotary drill rig with appropriate sized drill rods and downhole bits/casing systems for drilling in bedrock and unconsolidated materials. Hollow-stem auger (HSA) drill rig with appropriately sized augers and drill rods for drilling in unconsolidated materials (optional)
- High pressure, hot water washer for decontamination
- Decontamination equipment and supplies (e.g., wash/rinse tubs, brushes, alconox, plastic sheeting, paper towels, sponges, baby wipes, garden-type sprayers, large plastic bags, potable water, distilled water and/or deionized water)
- Sampling equipment for HSA rig (e.g., stainless steel 2-inch outer diameter split spoon sampler)
- Reclosable plastic bags for archiving samples
- 55-gallon drums or other approved containers for containing soil cuttings
- Other materials and equipment may be needed based on field conditions.

DRILLING PROCEDURES

Prior to drilling, borings will be numbered and the site cleared for utilities. Boring locations may be adjusted in the field due to the presence of underground utilities, overhead power lines, or other structures, or if access problems are encountered. Drilling locations will be approved by the Site Manager prior to initiating drilling activities.

Health and safety equipment specified in the site-specific HSP will be donned before proceeding with subsurface drilling activities. The HSP will specify action levels for various contaminants and the field monitoring required to measure ambient conditions.

All drill cuttings will be placed in labeled drums and moved to a central secured location for storage. Any water generated during drilling will be contained in labeled drums or tanks. Handling of investigation derived wastes (IDW) will be as specified in SOP - IDW Management.

Downhole equipment will be steam-cleaned prior to proceeding to the drill site and between subsequent boreholes using the procedures presented in SOP - Decontamination of Sampling Equipment. Samplers will be decontaminated at the drill site between each sample interval.

All work areas around borings will be restored to a physical condition equivalent to that of pre-drilling, as near as practical. This will include drill cuttings removal and rut repair.

At the direction of the field geologist, potable water may be introduced into boreholes. If it is necessary to introduce foaming agents into boreholes to lift cuttings during bedrock drilling, Material Safety Data Sheets (MSDS) for any products will be supplied for review and approval.

SOP No. 7 – Drilling and Lithologic Logging

For air or fluid rotary drilling, a shale-shaker and de-sanding sanding system may be used to maintain the density and viscosity of the drilling fluid. Sand content will be minimized to the extent possible to enhance recovery of cuttings.

The rig shall be free of leaks that could contaminate the boreholes (i.e., hydraulic fluid, oil, fuel, etc.). Pipe lubricants that are used should not introduce contaminants into the borehole. Lubricants that are environmentally acceptable include Green Stuff®, King Stuff®, vegetable oil, Crisco™, and some Teflon™-based lubricants. Lubricants that are not acceptable include petroleum-based and most metal-based lubricants. The Site Manager will pre-approve lubricants that will be used.

AIR/FLUID ROTARY DRILLING

The procedures below address drilling of boreholes using a, air-rotary or fluid-rotary drill rig. Rotary drilling may be conducted to install bedrock monitoring wells and injection points. Samples of drill cuttings will be collected for visual logging purposes at five-foot intervals. Samples may also be collected for testing. Drilling and sampling procedures using a rotary drill rig are as follows:

- Remove stones, vegetation, etc., from the sampling location surface.
- Use an appropriate drill bit to provide for a minimum 2-inch annulus around the groundwater monitoring well casing.
- Sampling of drill cuttings will be performed at five-foot intervals to the total depth of the borehole. Samples will be collected directly from the cyclone and placed in quart-sized baggies and labeled with the boring number and depth.
- Screen the sampled material using the instruments specified in the HSP.
- Log the sample in accordance with SOP - Borehole Logging.
- Follow sample handling procedures for collecting samples as described in SOP - Sample Management.
- Discard the unused samples and handle the waste as described in SOP - IDW Management.

HOLLOW-STEM AUGER DRILLING

The procedures below address drilling of boreholes and collection of subsurface soil samples using a HSA rig. HSA drilling may be conducted to install shallow monitoring wells in unconsolidated materials. Subsurface soil samples may be collected in some boreholes for laboratory chemical analysis at specific intervals as outlined in the project Work Plans. For boreholes where samples are not collected for chemical analysis, samples will be collected at five-foot intervals during drilling and placed in plastic bags. HSA drilling and samples procedures are as follows:

- Remove stones, vegetation, etc., from the sampling location surface.
- Use the appropriate sized augers to provide for a minimum 2-inch annulus around the groundwater monitoring well casing.

- If split-spoon sampling is used, collect a sample by driving the spilt-spoon sampler using a 140-pound hammer with a 30-inch drop. Record Standard Penetration Test blow counts for each 6-inch interval driven (for the first 18 inches).

- If a continuous sampling device is used, collect a sample by lowering the sample barrel through the augers and connecting the center rods to the drive head such that the barrel tip is slightly ahead of the HSA cutting bit.

- Bring the sampler to the surface and open the sampler.

- Screen the sampled material using the instruments specified in the HSP.

- Log the sample in accordance with the Borehole Logging procedures listed below.

- Repeat the process until the total depth of the borehole is reached.

BOREHOLE ABANDONMENT

Borehole abandonment may be necessary in some cases. The following procedures will be used to abandon boreholes:

- All downhole equipment will be removed from the borehole. Equipment will be decontaminated in accordance with SOP - Decontamination of Sampling Equipment.

- Boreholes will be grouted using bentonite pellets or cement-bentonite grout. If used, the grout mix will be in the proportions of one sack of Portland cement (94 pounds), 2 to 5 pounds of powdered bentonite, and approximately 7 to 9 gallons of water. The bentonite will be well mixed with the water prior to adding the cement.

- Grouting will be performed by placing a tremie pipe to the bottom of the borehole and pumping grout through the tremie pipe until undiluted grout flows from the ground surface.

- Twenty-four hours after grouting, the borehole will be checked for settlement and topped off to the ground surface with grout.

- Details concerning the abandonment process will be recorded on the boring log and in the field logbook.

Lithologic Logging

The following materials and equipment may be needed:

- Munsell soil color chart
- Hand lens
- Putty knife or spatula
- Dropper with 10% hydrochloric acid (HCl) for calcium carbonate test
- Drilling forms
- Sampling device (e.g., core barrel, split-spoon, Macrocore ™)
- Waste containers as specified in the IDW Management Plan

- Health and safety equipment, as specified in the Site-Specific Health and Safety Plan

PROCEDURES

A "site geologist" (geologist, hydrogeologist or geotechnical engineer) experienced in borehole drilling and soil sampling will be present at each operating drill rig. This site geologist will be responsible for logging samples, monitoring drilling operations, and preparing field boring logs.

- Boring log information will be recorded on a Boring Log Form. Depth information will be recorded to the nearest 0.1 foot. An appropriate scale will be used on the boring log form (e.g., a scale of 1 inch on the log form equaling 1 foot of boring).
- Measure entire sample core length and record the recovery on the drilling log. Mark lithologic changes on the drilling form. Lithologies will be logged directly from cores/cuttings and indirectly interpolated using professional judgment, drill cuttings, drill action, etc., between sampling intervals.
- Descriptions of intact unconsolidated soil samples will include parameters listed in Table 1. Material will be described in the following order:
  - Material type (i.e., sand [sandstone], silt [siltstone], clay [claystone], etc.)
  - Color (Munsell color chart will be used)
  - Grain size, sorting, rounding, and composition of the material (for sand or gravel)
  - Types and amounts of secondary constituents
  - Other pertinent characteristics (plasticity, hardness, bedding, etc.)
  - Moisture content
  - USCS code (for unconsolidated material)
- Unconsolidated materials will be classified in accordance with the USCS (equivalent to ASTM D 2488-09a, “Description and Identification of Soil [Visual Manual Procedure]”; Attachment B and USEPA Manual 625/12-91/002 “Description and Sampling of Contaminated Soils”). Soil classifications will be made in the field at the time of sampling by the site geologist (Table 1).
- In the field, visual estimates of the volume of secondary soil constituents will be reported by such terms as "trace" (<5 percent), "few" (5-10 percent), “little” (15-25 percent) "some30-45 percent), and "mostly" (50-100 percent) or by an estimated percentage.
- Consolidated material (e.g., igneous and metamorphic rocks) will be described by parameters listed in Table 2 and described in Tennisen (1983), ASTM D5434-97, “Standard Guide for Field Logging of Subsurface Explorations of Soil and Rock”, and ASTM C294-86(1991), “Standard Descriptive Nomenclature for Constituents of Natural Mineral Aggregates”. Material will be logged using drill cuttings and/or rock core. Material will be described in the following order:
  - Rock Type
  - Color (Munsell color chart will be used)
  - Grain size and shape
  - Texture (stratification. foliation)
  - Mineral composition
  - Weathering and alteration
  - Strength
  - Other relevant notes

- Drill cuttings will be described in terms of the appropriate parameters, to the extent practical. "Classification" will be minimally described for this material, along with a description of drilling actions for the corresponding depth. Notations will be made on the log that these descriptions are based on observations of material other than formal samples (e.g., from cuttings).
- The drilling equipment used will be described on each log. Information such as drill rod size, bit size and type, and rig manufacturer and model will be recorded.
- All special problems encountered during drilling and their resolution will be recorded on the log. This would include sudden tool drops, unrecovered tools in the borehole, and lost casing.
- The dates for the start and completion of borings will be recorded on the log.
- Stratigraphic/lithologic changes will be identified on the boring log with a solid line at the measured borehole depths at which changes occur. Gradational transitions and changes identified from cuttings or methods other than direct observation and measurement will be identified by a horizontal dashed line at the appropriate scale depth based on the best judgment of the logger.
- Logs will show borehole and sample diameters and depths at which drilling or sampling methods or equipment change.
- Logs will show total depth of penetration and sampling. The bottom of the hole will be clearly identified on the log.
- Logs will identify the depth at which water is first encountered, the depth of water at the completion of drilling, and the stabilized depth to water. The absence of water in borings will also be indicated. Stabilized water-level data will include time allowed for levels to stabilize.
- Logs will include other information relevant to a particular investigation, but not limited to:
    - Odors
    - Field screening or test results (e.g., organic vapors)
    - Any observed evidence of contamination in samples and cuttings
- Special abbreviations used on a log will be defined either in the log where used, or in a general legend.

DOCUMENTATION

Project staff are responsible for documenting sampling activities. A field boring log form will be completed summarizing field activities. Field notes will also be kept during drilling and logging activities. The following information will be recorded in a bound field log book:

- Names of personnel
- Weather conditions
- Date and time of drilling and sampling
- Location and sample station number
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Boring log information
- Other applicable information

SOP No. 7 – Drilling and Lithologic Logging

DECONTAMINATION

All tools and sampling equipment (e.g., spatula, drive sampler,etc.) will be decontaminated between sample locations and between individual samples. Decontamination will be conducted in accordance with SOP – Sampling Equipment Decontamination.

DOCUMENTATION

Project staff are responsible for documenting sampling activities. A field boring log form will be completed summarizing field activities. Field notes will also be kept during drilling and logging activities. The following information will be recorded in a bound field log book:

- Names of personnel
- Weather conditions
- Date and time of drilling and sampling
- Location and sample station number
- Times that procedures and measurements are completed
- Decontamination times
- Calibration information
- Boring log information
- Other applicable information

TABLE 1

DESCRIPTION OF UNCONSOLIDATED SOIL

Parameter	Example
Formation, (if named and if known)	Alluvium
Unified Soil Classification System	Sandy Clay
Secondary Components and Estimated Quantities either by percentages or by descriptive percentage ranges (Note: terms used to indicate ranges should be described on the log or in a general legend)	sand: fine, with trace of med.
Color	gray
Consistency (cohesive soil). Use relative term	very soft, soft, medium, stiff, very stiff, hard
Density (non-cohesive soil). Use relative term	loose, medium, dense, very dense
Moisture Content. (Use relative term. Do not express as a percentage unless a value has been measured)	dry, damp, moist, wet, saturated
Texture/Fabric/Bedding	no apparent bedding, numerous vertical iron-stained tight fractures
Grain Angularity	rounded sand grains
Sorting (sands)	poorly sorted
Structure	slickensides
Grain or fragment size	coarse
Note "Fill", "Top of Natural Ground", and "Top of Bedrock" where appropriate	

Standard Operating Procedure No. 8

Piezometer, Monitoring Well, and Injection Point Construction

SOP No. 8 – PIEZOMETER, MONITORING WELL, AND INJECTION POINT CONSTRUCTION

The following procedures describe construction of monitoring wells, piezometers, and injection points.

MONITORING WELL CONSTRUCTION

Objective

Monitoring wells will be installed to collect representative samples of the unconfined groundwater, to measure the groundwater surface elevation, and to conduct various types of aquifer testing. Monitoring wells will be constructed in a manner that complies with applicable federal, state, and local regulations. This SOP to be used in association with SOP – Lithologic Logging.

Equipment and Materials Needed

- Drill tooling (supplied by subcontractor). Drilling techniques will be selected based on the site-specific geology and judgment of the onsite geologist and drilling foreman.
- Well materials (supplied by subcontractor).
- Washed silica grade filter sand of an appropriate gradation
- Grout mixing and pumping equipment with a tremie system
- Sodium bentonite pellets
- Cement-bentonite grout
- Locking steel protective casing and guard posts or flush boxes
- Concrete
- Indelible pen
- Monitoring well construction diagrams
- Weighted measuring tape
- Electronic water level indicator
- Waste containers as specified in SOP – IDW Management
- Safety equipment as specified in the Site-Specific Health and Safety Plan

Methodology

Monitoring wells will be constructed in open boreholes or through the hollow stem augers or surface casing, depending on the stability of the borehole and materials encountered during drilling.

The annular space will be filled with a filter pack (adjacent to the well screen), a bentonite seal, and casing grout between the well string and the borehole wall. As the annular space is being filled, the well string will be centered and suspended such that it does not rest on the bottom of the hole.

Measurements made during filling of the annular space will be performed to the nearest 0.1 foot below ground surface (bgs) and will consist of the following:

- Total depth of borehole at the completion of drilling
- Total depth of the open borehole before the start of well construction

- Lengths of the end cap, screen sections, riser blank sections, and stickup of well above ground surface
- The depth to the top of the filter pack, top of the bentonite seal, and the top of the grout.

Casing and Screen Requirements

The casing requirements will be as follows:

- All casing will be new, unused, and/or decontaminated according to the specifications of SOP - Sampling Equipment Decontamination
- All PVC will conform to the ASTM Standard F-480-88A or the National Sanitation Foundation Standard 14 (Plastic Pipe System).
- The casing will be straight and plumb.

Well screen requirements include:

- All requirements for casing, except for strength requirements, apply to well screens.
- The screen material shall be new, non-contaminating, non-clogging, continuous slot design.
- Screen slot openings shall be 0.010 inches unless otherwise specified based on site-specific aquifer properties.
- The bottom of the screen will be capped with a threaded cap.

Filter Pack

The purpose of the well filter pack is to provide lateral support for the well screen, increase yield by improving the hydraulic conductivity in the immediate vicinity of the well, and retain the formation to prevent natural materials from entering the well. The filter pack material will be clean, inert, and well rounded, and will contain less than 2 percent flat particles. The filter pack will consist of 10/20 or 20/40 mix or equivalent of clean silica sand and will be placed from the bottom of the hole to at least 2 feet, but not more than 4 feet, above the top of the well screen. The size of the filter pack material used will be selected as appropriate for the well screen slot size installed so that no more than 10% of the filter pack material is smaller than the slot size.

For auger boreholes, the filter pack will be placed in the hole by pouring the sand through the augers and slowly raising the augers out of the hole. For bedrock monitoring wells installed in open boreholes (by rotary drilling), the screen and riser casing will be suspended above the bottom of the borehole as the filter pack is poured directly into the borehole. The volume of the filter pack placed in the well will be recorded.

Well Seal

The materials used to seal the annulus between the borehole wall and casing must prevent potential contaminant migration from ground surface or intermediate zones, isolate a discrete monitoring zone, preserve confining conditions, prevent intrusion of the overlying grout into

the filter pack, and prevent cross-contamination between strata. The bentonite seal will consist of at least 3 feet, but not more than 5 feet, of bentonite pellets between the filter pack and the casing grout. If the bentonite seal is placed above the water table, then the bentonite will be hydrated using potable water.

Annulus Backfill/Grout

The annular space above the filter pack and seal will be grouted with a bentonite/cement mixture. Grouting is used to minimize the vertical migration of water to the screened interval and to increase the stability and integrity of the well casing.

The cement/bentonite grout mixture shall consist of 95 to 97 percent Type V or Type II-V Portland Cement and 3 to 5 percent bentonite powder by weight (equivalent to one 94-pound bag of cement and between 3 and 5 pounds of bentonite). Approximately 8.5 gallons of water shall be used for each cement/bentonite batch. The grout mixture shall be prepared by thoroughly mixing the bentonite powder with water first and then mixing in the cement.

The casing grout requirements are as follows:

- The annular grout will extend from the top of the bentonite seal to approximately 3 feet below ground surface (bgs).
- Grout shall be placed in the well annulus using a tremie pipe located within approximately 10 feet of the top of the bentonite seal and the tremie pipe will be pulled up as the annular space is filled.
- Pumping will continue until undiluted grout has been returned to the surface.
- After grouting, the well shall not be disturbed or be developed for a minimum of 24 hours. Additional grout will be added if settling occurs.

Surface Casing

After the grout has hardened for at least 24 hours, place the protective casing or flush box in the borehole and support it at the appropriate height (3 feet above the ground surface for a stick-up completion or flush with the ground surface for a flush completion).

For aboveground completions, build a form approximately six (6) inches deep and two (2) feet square around the well completion. Pour concrete into the form and smooth the concrete so that it slopes gently away from the well completion.

If necessary, set four 4-inch-diameter protective posts at the corners of the pad. These posts will be set in postholes at least 18 inches deep and will stick up four (4) feet. Concrete will be poured into the posts and the holes. Bring the concrete no higher than 2 to 3 inches below ground level in the post holes, so the soil can be filled in over the top.

Mark the water level measuring point on the north side of the PVC casing and write the well identification number on the well cap with an indelible marker. Lock the well cap.

The well completion will be a permanent feature that will be visible and obvious. Make sure that completions are neat and the site graded to previous conditions. If well riser needs to be cut to achieve the proper height for the stick up, it should be cut with a hack saw before installation, or with a pipe cutter after installation so that the cut is even and level.

PIEZOMETER CONSTRUCTION

Piezometers will be installed and completed in the same manner as the unconfined monitoring wells except there will be no grout or surface completion. Bentonite will be placed to within one (1) foot of ground surface and soil will be used to fill in up to bring the area back to grade. Piezometers may be constructed one or one and one-half-inch diameter schedule 40 PVC.

INJECTION POINT CONSTRUCTION

Injection points will be installed to allow remediation treatment chemicals to be injected into groundwater. Injection points can be installed by several drilling methods, including direct-push, hollow-stem auger, and rotary drilling methods. Injection points are short-term features (e.g., may only be utilized 1-2 times) and will be abandoned after the remedy is complete.

Equipment and Materials Needed

- Drill tooling (supplied by subcontractor). Drilling techniques will be selected based on the site-specific geology and judgment of the onsite geologist and drilling foreman. The borehole will be of sufficient diameter to achieve a seal that will confine the injectate to the targeted interval.
- Grout mixing and pumping equipment with a tremie system
- Well materials (supplied by subcontractor)
- Grout basket and pre-packed bentonite seal above the slotted portion of the screen.
- Sodium bentonite pellets
- Cement-bentonite grout
- Injection well construction forms
- Weighted measuring tape
- Electronic water level indicator
- Waste containers as specified in SOP – IDW Management
- Safety equipment as specified in the Site-Specific Health and Safety Plan

Methodology

Injection points will be constructed in open boreholes or through the hollow stem augers or surface casing, depending on the stability of the borehole and materials encountered during drilling.

The annular space in the screened area of the casing will be left open to maximize contact with the affected portions of the saturated zone. A grout basket and a bentonite seal will be placed above of the screen, and cement-bentonite grout will be placed above the seal to focus injected materials into the target interval and prevent injectate from rising up the borehole annulus.

Injection points casing will be new, unused, and/or decontaminated according to the specifications of SOP - Sampling Equipment Decontamination. The well seal will be constructed of either a grout basket that will expand to the maximum dimensions of the borehole to prevent bentonite pellets from entering the screened internal and/or pre-packed bentonite placed on the well pipe. The bentonite seal will consist of at least 3 feet, but not more than 5 feet, of bentonite pellets. If the bentonite seal is placed above the water table, then the bentonite will be hydrated using potable water.

The annular space above the seal will be grouted with a bentonite/cement mixture. Grout will be mixed as specified in "Monitoring Well Construction" above.

Standard Operating Procedure No. 9

Monitoring Well Development

SOP No. 9 – Monitoring Well Development

1.0 OBJECTIVE

Monitoring wells installed during the remedial investigation will be developed to:

- Remove fine-grained native soil material that may have collected in the well casing during construction
- Grade the filter pack from formation to casing
- Allow for accurate chemical measurements to be made during well sampling
- Reduce potential cross contamination within the borehole

During well development, water quality parameters will be measured and pumping rates will be documented, the latter of which can be used to select sample purge rates and pumping test rates during future field activities.

2.0 EQUIPMENT AND MATERIALS

- Copies of well drilling and installation records, including lithologic logs for the well to be developed
- Electronic water level meter
- Water quality meter(s) to measure pH, temperature, electrical conductivity and turbidity and appropriate calibration standards
- Bailer and appropriate pumping equipment
- Well development log
- Decontamination equipment
- Waste management equipment
- Health and safety equipment as specified in the Site-Specific Health and Safety Plan

3.0 METHODOLOGY

1. Begin developing no sooner than 24 hours after a well has been constructed.
2. Prior to the start of development activities, clean well development equipment following proper decontamination procedures in SOP – Sampling Equipment Decontamination.
3. Before developing a well, measure the total well depth and the depth to the top of the water table (see SOP – Water Level Measurement), and record the measurements in the well development log.
4. Alternately surge and bail the well until minimal fines are produced and the purged water begins to clear.

5. Take water quality measurements for pH, temperature, specific conductivity, and turbidity frequently once the water clears, at least every half-hour; take measurements more frequently as visual observations or water quality measurements indicate that the consistency of the water is changing.
6. Turbidity will be measured during well development (see SOP – Field Parameter Measurements), with the goal being 50 nephelometric units (NTU). If 10 wetted casing volumes of water have been purged from the well, and the turbidity has not reached 50 NTU but is between 50 and 500 NTU, the well development will be considered complete.
7. Contain and handle purge water according to specifications in SOP – IDW Handling.
8. If a pump is used instead of a bailer, install a development pump (2- to 5-gallons per minute [gpm] capacity submersible pump).
9. Purge water from the well, using the pump, at a rate approximately equal to or greater than the anticipated purging/sampling rate.
10. Continue development until a minimum of five wetted casing volumes have been removed. Continue purging and monitor the pH, temperature, specific conductivity, and turbidity. Stop purging once parameters stabilized to within 10% of the previous two readings or 10 wetted casing volumes have been removed.
11. Record measurements on the well development log.
12. Continue to contain the purge water according to methods outlined in SOP – IDW Management.
13. When development is complete, remove and decontaminate the purge pump and measure and record a final total well depth and top of the water table depth.

4.0 COMMENTS

Wells at the site are expected to produce sufficient volumes of water to allow for standard development. However, slow producing wells may need to be developed using an alternative procedure. If, during the initial phases of development, a well is bailed dry, development should be performed by bailing the well, allowing it to recover to at least 90% of the initial water level, and repeating the bailing.

Standard Operating Procedure No. 10

Surface Water/Sediment Sampling for Chemical Analysis

SOP No. 10 – Surface Water/Sediment Sampling for Chemical Analysis

1.0 OBJECTIVE

Surface water and sediment samples will be collected for chemical analysis to help characterize overland migration and potential groundwater discharge to surface water from source areas, and to determine the nature and extent of contamination in intermittent or perennial water bodies.

2.0 EQUIPMENT AND MATERIALS

- Appropriate number and types of sample containers
- Pre-cleaned stainless steel sampling utensils (See SOP – Sampling Equipment Decontamination)
- Sample coolers and ice.
- Appropriate field documentation forms and labels and an indelible ink pen.
- Decontamination equipment.
- Waste containers.
- Health and safety equipment, as specified in the Health and Safety Plan.

3.0 METHODOLOGY

Surface water and sediment samples will be collected as follows:

a) The order of sample collection should be downstream to upstream.

b) Care should be taken not to disturb stream bottoms resulting in sediment suspension.

c) Water samples should be collected first by carefully immersing the sample container below the water surface, letting it fill slowly w/o losing preservative.

d) Samples bottles for volatile organic compound (VOC) analysis will be collected first to minimize the potential for volatilization. Vials should be filled with zero headspace, again taking care not to lose preservative.

e) Collect samples for all non-VOC water then sediment (location-specific) analyses next, then work your way upstream to additional sample locations as designated.

f) Complete the sample labels in accordance with SOP – Sample Handling and Management and place them on the jars.

g) Place protective packing on the sample jar.

h) Decontaminate sampling equipment in accordance with SOP – Sampling Equipment Decontamination.

i) Collect and manage wastes as specified the waste management plan.

Standard Operating Procedure

Well and Borehole Abandonment

1.0 OBJECTIVE

This Standard Operating Procedure (SOP) provides technical guidance and methods that will be used to seal and abandon boreholes created during the field investigation. This SOP serves as a supplement to Nebraska water well regulations (178 NAC 12). Personnel overseeing and performing borehole abandonment will have knowledge and experience in working with drill rigs and sealing and abandoning environmental boreholes.

2.0 EQUIPMENT AND MATERIALS

The following equipment and materials may be needed for borehole abandonment:

- Personal protective equipment (PPE) as outlined in the HSP
- Drill rig equipped with a hoist (for removal of well/piezometer casing and screen)
- Grouting mixing and pumping equipment
- Grouting materials and equipment (potable water, bentonite powder, bentonite chips, cement for grouting [portland type II or IV], large tank for mixing grout, trash pump for mixing grout, plastic or steel tremie pipe)
- Borehole Abandonment Form

Other materials and equipment may be needed based on field conditions.

3.0 METHODOLOGY

Abandonment is the procedure by which a borehole is permanently sealed in place, or a monitoring well or piezometer is sealed in place or removed. The objective of abandonment is to eliminate the potential vertical pathway to the subsurface associated with boreholes or unneeded wells. Abandonment will be performed for the following:

- Following drilling and sampling of investigation boreholes.
- To remove wells or piezometers that are no longer needed.

3.1 BOREHOLE ABANDONMENT

Investigation boreholes drilled for collection of subsurface material samples will be abandoned immediately following sampling and/or data collection. Prior to any intrusive drilling associated with abandonment, utility clearance will have been accomplished according to SOP – Staking, Utility Clearance, and Permitting.

The following steps will be performed to abandon a borehole:

- All boreholes to be abandoned will be grouted with a cement/bentonite grout.
- Grout will be injected into the borehole using a tremie pipe as the hollow-stem augers are pulled. Measurements will be taken of the grout level during the process to verify that the grout level does not fall below the bottom of the augers, thus ensuring a continuous seal is injected into the borehole.

- The cement-bentonite grout mixture will consist of the following in accordance with Nebraska Water Well Standards. The amount of bentonite shall not exceed 5 pounds bentonite per cubic foot (94 pounds) of Portland cement. The volume of water used will not exceed 6.5 gallons per 94 pound bag of cement.
- During grouting, water present in the borehole will be displaced up the borehole. Displaced borehole water cannot be discharged to the ground surface. Displaced water will be pumped from the borehole as grouting progresses and contained in drums or other approved water-tight containers for handling as investigation derived waste (IDW). IDW management is addressed in SOP -IDW Management.
- Twenty-four hours after grouting, the borehole will be checked for grout settlement and will be topped off to the ground surface with grout, if necessary.
- Any excess grout will be placed in drums for disposal in accordance with SOP -IDW Management.

3.2 MONITORING WELL AND PIEZOMETER ABANDONMENT

Monitoring wells or piezometers that are no longer needed may also be abandoned The preferred method of abandonment is to remove the surface completion (i.e., protective casing, traffic posts, and surface seal), pull or drill out the well or piezometer casing and screen, drill out the annular seal and filter pack material, and seal the borehole. State or local regulations may require alternate methods in specific circumstances.

In most cases it may not be feasible to remove the well casing. Wells that are not pulled or drilled out, will be sealed by injecting grout under pressure into the well casing. Well casings may be split prior to grouting.

If the well or piezometer cannot be pulled or drilled out, the well or piezometer will be sealed in place by injecting grout under pressure into the well casing. The PVC well casing may be split using a down-hole casing splitter prior to grouting.

All well/piezometer casing and annular materials will be handled as IDW as addressed in SOP – IDW Management.

4.0 DOCUMENTATION

The rig geologist or other responsible person will document all borehole or well/piezometer abandonment procedures on a Borehole Abandonment Form and in the field logbook.

The following information will be documented:

- Borehole or well/piezometer designation
- RFI contractor and personnel names
- Abandonment subcontractor and personnel names
- Dates of abandonment
- Measured diameters and depths of boreholes and wells/piezometers prior to initiating abandonment activities

- Detailed record of abandonment procedures
- Volume of water produced and contained during abandonment
- Grout composition and quantity used
- Casing reused or left in place
- IDW generated

Designation: D 4044 – 96 (Reapproved 2008)

Standard Test Method for (Field Procedure) for Instantaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifers[1]

This standard is issued under the fixed designation D 4044; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the field procedure for performing an in situ instantaneous change in head (slug) test.

1.2 This test method is used in conjunction with an analytical procedure such as Test Method D 4104 to determine aquifer properties.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 4043 Guide for Selection of Aquifer Test Method in Determining Hydraulic Properties by Well Techniques

D 4104 Test Method (Analytical Procedure) for Determining Transmissivity of Nonleaky Confined Aquifers by Overdamped Well Response to Instantaneous Change in Head (Slug Tests)

D 4750 Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well)

D 5785 Test Method for (Analytical Procedure) for Determining Transmissivity of Confined Nonleaky Aquifers by Underdamped Well Response to Instantaneous Change in Head (Slug Test)

D 5881 Test Method for (Analytical Procedure) Determining Transmissivity of Confined Nonleaky Aquifers by Critically Damped Well Response to Instantaneous Change in Head (Slug)

D 5912 Test Method for (Analytical Procedure) Determining Hydraulic Conductivity of an Unconfined Aquifer by Overdamped Well Response to Instantaneous Change in Head (Slug)

3. Terminology

3.1 *Definitions:*

3.1.1 *control well*—well by which the aquifer is stressed, for example, by pumping, injection, or change of head.

3.1.2 *hydraulic conductivity—(field aquifer tests)*, the volume of water at the existing kinematic viscosity that will move in a unit time under a unit hydraulic gradient through a unit area measured at right angles to the direction of flow.

3.1.3 *observation well*—a well open to all or part of an aquifer.

3.1.4 *overdamped-well response*—characterized by the water level returning to the static level in an approximately exponential manner following a sudden change in water level. (See for comparison *underdamped well*.)

3.1.5 *slug*—a volume of water or solid object used to induce a sudden change of head in a well.

3.1.6 *storage coefficient*—the volume of water an aquifer releases from or takes into storage per unit surface area of the aquifer per unit change in head. For a confined aquifer, it is equal to the product of specific storage and aquifer thickness. For an unconfined aquifer, the storage coefficient is approximately equal to the specific yield.

3.1.7 *transmissivity*—the volume of water at the existing kinematic viscosity that will move in a unit time under a unit hydraulic gradient through a unit width of the aquifer.

3.1.8 *underdamped-well response*—characterized by the water level oscillating about the static water level following a sudden change in water level. (See for comparison *overdamped well*.)

3.1.9 For definitions of other terms used in this test method, refer to Terminology D 653.

4. Summary of Test Method

4.1 This test method describes the field procedures involved in conducting an instantaneous head (slug) test. The slug test method involves causing a sudden change in head in a control well and measuring the water level response within that control well. Head change may be induced by suddenly injecting or

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.21 on Ground Water and Vadose Zone Investigations.

Current edition approved Sept. 15, 2008. Published October 2008. Originally approved in 1991. Last previous edition approved in 2002 as D 4044 – 96 (2002).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

removing a known quantity or "slug" of water into the well, rapid removal of a mechanical "slug" from below the water level, increasing or decreasing the air pressure in the well casing, or emplacement of a mechanical slug into the water column.

4.2 The water-level response in the well is a function of the mass of water in the well and the transmissivity and coefficient of storage of the aquifer. One method of analysis of the data from this field practice is described in Test Method D 4104.

5. Significance and Use

5.1 This slug test field procedure is used in conjunction with a slug test analytical procedure, such as Test Method D 4104 to provide quick and relatively inexpensive estimates of transmissivity.

5.2 The slug test provides an advantage over pumping tests in that it does not require the disposal of the large quantities of water that may be produced. This is of special importance when testing a potentially contaminated aquifer. However, slug tests reflect conditions near the well, therefore are influenced by near-well conditions, such as gravel pack, poor well development, and skin effects.

5.3 Slug tests may be made in aquifer materials of lower hydraulic conductivity than generally considered suitable for hydraulic testing with pumping tests.

5.4 The method of data analysis (analytical procedure) should be known prior to the field testing to ensure that all appropriate dimensions and measurements are properly recorded. Selection of the analytical procedure can be aided by using Guide D 4043, Test Method D 5785, Test Method D 5881, and Test Method D 5912.

6. Apparatus

6.1 *Slug-Inducing Equipment*—This test method describes the types of equipment that can be used. Because of the infinite variety of testing conditions and because similar results can be achieved with different apparatus, engineering specifications for apparatus are not appropriate. This test method specifies the results to be achieved by the equipment to satisfy the requirements of this practice.

6.2 *Water-Level Measurement Equipment*—The method of water level measurement may be dependent on the method selected for injection or withdrawal of water, and the nature of the response of the well. For an open-well test, that is, where access to the water level is open to the surface, measure water levels manually as described in Test Method D 4750, by an automatic recording device linked to a float, or with a pressure transducer linked to a data logger or display device. A pressure transducer linked to a data logger will be necessary for a test in a closed well in which water-level changes are induced by vacuum or pressure on the control well and where manual measurements do not provide measurements of adequate frequency (see 9.3).

7. Conditioning

7.1 *Pre-Test Procedure*:

7.1.1 *Measuring Pre-Test Water Levels*—Measure the water level in the control well before beginning the test for a period longer than the duration of the test to determine the pre-test water level fluctuations and to establish pre-pumping water-level trend and to determine a pre-pumping reference water level.

8. Procedure

8.1 Cause a change in water level, either a rise or decline, by one of the following methods:

8.1.1 *Water Slug*—Inject or withdraw water of a known quantity into or from the control well.

8.1.2 *Mechanical Slug*—Inject or withdraw a mechanical slug below or above the water level. The water within the control well will then rise or decline an amount equal to the volume of the mechanical slug.

8.1.3 *Release Vacuum or Pressure*—A method of simulating the injection or withdrawal of a slug of water is by the release of a vacuum or pressure on a tightly capped (shut-in) control well. Before the release, the vacuum or pressure is held constant.

NOTE 1—There is no fixed requirement for the magnitude of the change in water level. Similar results can be achieved with a wide range in induced head change. Some considerations include a magnitude of change that can be readily measured with the apparatus selected, for example the head change should be such that the method of measurement should be accurate to 1 % of the maximum head change. Generally, an induced head change of from one-third to one meter is adequate. Although the induced head change should be sufficient to allow the response curve to be defined, excessive head change should be avoided to reduce the possibility of introducing large frictional losses in well bore.

The mechanical model for the test assumes the head change is induced instantaneously. Practically, a finite time is required to effect a head change. Selection of time zero can be selected experimentally. Refer to the method of analysis (such as Test Method D 4104) to determine time zero and to evaluate the suitability of the change effected in the well.

8.2 Measure water-level response to the change in water level. The frequency of water-level measurement during the test is dependent upon the hydraulic conductivity of the material being tested. During the early portions of the test, measure water levels at closely-spaced intervals. Measurements of water level made manually with a tape should be made as frequently as possible until the water level has recovered about 60 to 80 %. Increase the length of time between measurements with increasing duration of the test. Since most methods of data analysis are curve-fitting techniques, it is essential that water levels are measured frequently enough to define the water-level response curve (see Guide D 4043, Test Methods D 4104 and D 5785).

8.2.1 In aquifer-well systems where water-level changes are rapid, it may be necessary to use a pressure transducer linked to an electronic data logger to measure and record the water levels frequently enough to adequately define the waterlevel response. The use of transducers and data loggers generally provides a greater than adequate frequency of measurements, ranging from several measurements per second in the early part of the test to a specified frequency in the later portions of a test. With such equipment, the test analysis may use a reduced data set of measurements to calculate the hydraulic properties (see Guide D 4043, Test Methods D 4104 and D 5785 for analysis of water level data).

8.3 *Post-Test Procedure*—Make preliminary analysis of data before leaving the field and evaluate the test regarding the criteria given in this test method and the method of analysis, such as Test Method D 4104 to determine if the test should be rerun.

9. Report

9.1 Include the information listed below in the report of the field procedure:

9.2 All test reports should include the following:

9.2.1 Date, time, and well identification,

9.2.2 Method of slug withdrawal or injection, as well as whether the test is a falling head (injection) or a rising head (withdrawal) test,

9.2.3 Inside diameter of well screen and well casing above screen,

9.2.4 Depth of well,

9.2.5 Length and depth setting of screen,

9.2.6 Volume of mechanical slug or pressure change imposed on water level, and

9.2.7 Pre-testing water-level trend.

9.3 Establish and record the measurement point from which all measurements of water level are made. Record date, time, and depth to water level below measurement point of all water levels.

9.4 Water levels measured during the test should be recorded with information on date, clock time, and time since test started. If the water levels are measured with a pressure transducer and recorded with an electronic data logger, record the name of the data file on the data logger.

10. Precision and Bias

10.1 *Precision*—It is not practical to specify the precision of this test method because the response of aquifer systems during aquifer tests is dependent upon ambient system stresses.

10.2 *Bias*—No statement can be made about bias because no true reference values exist.

11. Keywords

11.1 aquifer tests; aquifers; ground water; hydraulic conductivity; hydraulic properties; instantaneous head test; slug tests; storage coefficient; transmissivity

SOP NO. 13: SOIL BORING AND MONITORING WELL SURVEY REQUIREMENTS

USACE GEOLOGY SCOPE OF SERVICES (MAY 2011)

7.0 SURVEYS (GENERAL).
All sampling locations shall be staked to facilitate subsequent surveying. The Contractor shall perform all surveys required for this project and shall supply this office with the original or a legible reproducible copy of the surveys and field books. The surveys shall at least conform to the requirements stated in the following paragraphs.

7.1 Monitoring Wells.
Coordinates and elevations shall be established for each monitoring well. The coordinates shall be to the closest one foot and referenced to the State Plane Coordinate System. If the State Plane Coordinate System is not available, an existing local grid system shall be used. A ground elevation to the closest 0.1-foot and an elevation for the top of the well riser to the closest 0.01-foot shall be obtained at each well. These elevations shall be referenced to Mean Sea Level, specifically to the North American Vertical Datum (NAVD) of 1988. If the 1988 Datum is not available, the National Geodetic Vertical Datum (NGVD) of 1929 shall be used. All positions and coordinates of all permanent points within the control traverse shall be shown. If not stated in the Site Specific Scope of Services, the Contractor shall coordinate with the USACE- Omaha

7.2 Soil Borings/Sampling Points
All soil sampling locations shall be located horizontally following procedures outlined in paragraph: **8.1 Monitoring Wells**.

7.4 Documentation.
The location, identification, coordinates, and elevations of the wells and monuments shall be plotted on maps with a scale large enough to show their locations with reference to other structures at the individual sites. A tabulated list of the monitoring wells and monuments, copies of all field books, and all computations sheets shall be prepared and submitted to the USACE- COR. The tabulations shall consist of the designated number of the well or monument, the X and Y coordinates, and all the required elevations. These items shall be submitted to Omaha District no later than the Draft Project Report.

En Novative Technologies, Inc.

Recommended Use Of The Terra Core®

NOTE: The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.

Step 1

Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.

Step 2

Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

Step 3

Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial. **Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.

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En Novative Technologies, Inc.

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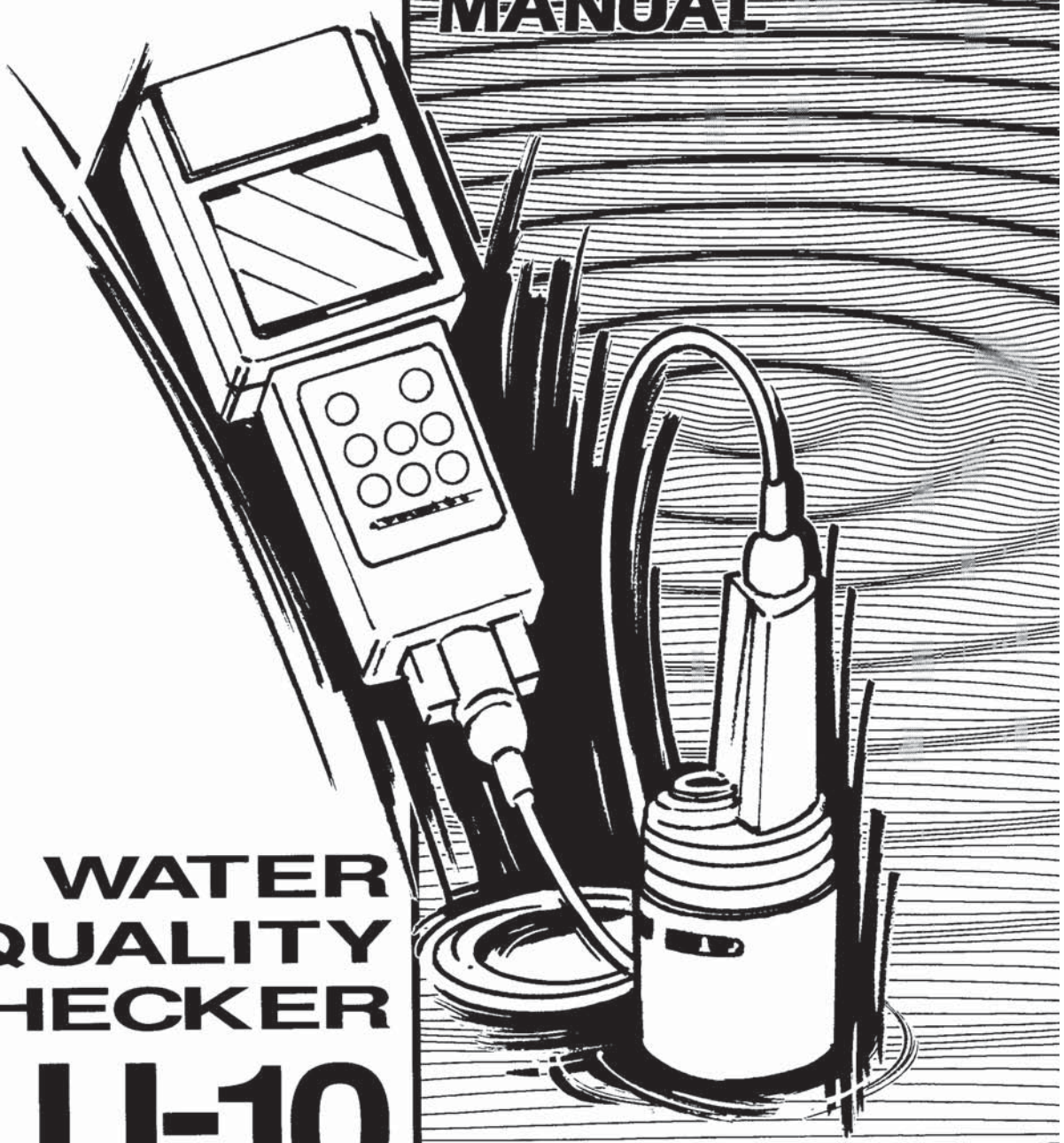
10. Governing Law. All disputes relating to the terms hereof, performance of this order or any other claim related to Seller's goods shall be governed by the laws of the State of Wisconsin; provided, however, construction shall be without regard to any rule or presumption requiring construction against the party causing this agreement to be drafted. Buyer and Seller agree that any dispute arising between them which results in either party institut ing court proceedings that such action will be maintained in the Circuit Court for Brown County, Wisconsin.

HORIBA

INSTRUCTION MANUAL



WATER QUALITY CHECKER U-10

CODE: 040801000HK-5

WARNING

The DO sensor contains a strong alkaline solution. Should any of this solution come in contact with your clothing or skin, wash it away immediately with plenty of water.

Be especially careful not to allow any of the alkaline liquid in the DO sensor to get in your eyes.

⚠CAUTION

Insert the battery with ample care to the polarity. Reverse insertion on the polarity will make damage to the inner PCB.

This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) This device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to Part 15 of the FCC Rules. These limits are designed to provede reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

The U-10 Water Quality Checker is a state-of-the-art instrument for simultaneous multiparameter measurement of water quality. The HORIBA U-10 measures six different parameters of water samples: *pH*, *conductivity*, *turbidity*, *dissolved oxygen*, *temperature*, and *salinity*.

The U-10 is compact enough to be held in one hand while taking measurements. It has a large easy-to-read LCD readout. Measurements are taken simply by immersing the probe right into the water sample.

The U-10 is extremely versatile and sophisticated, yet easy to use. You will find it a valuable addition to on-site water control operations, whatever your needs—from testing factory discharges to urban drainage, river water, lake and marsh water, aquatic culture tanks, agricultural water supplies, and sea water.

To get the most out of your U-10 Water Quality Checker, please read this *Instruction Manual* carefully before you begin to take measurements.

Note that Horiba cannot be held responsible for any equipment malfunction or failure should the U-10 Water Quality Checker be operated incorrectly or in a manner other than specified in this *Instruction Manual*.

Horiba's aim is to produce the best possible equipment and documentation for our products. We welcome comments, questions, or suggestions for improvement concerning both our products and the accompanying documentation, such as this *Instruction Manual*.

Second edition: November, 1991
First edition: July, 1991

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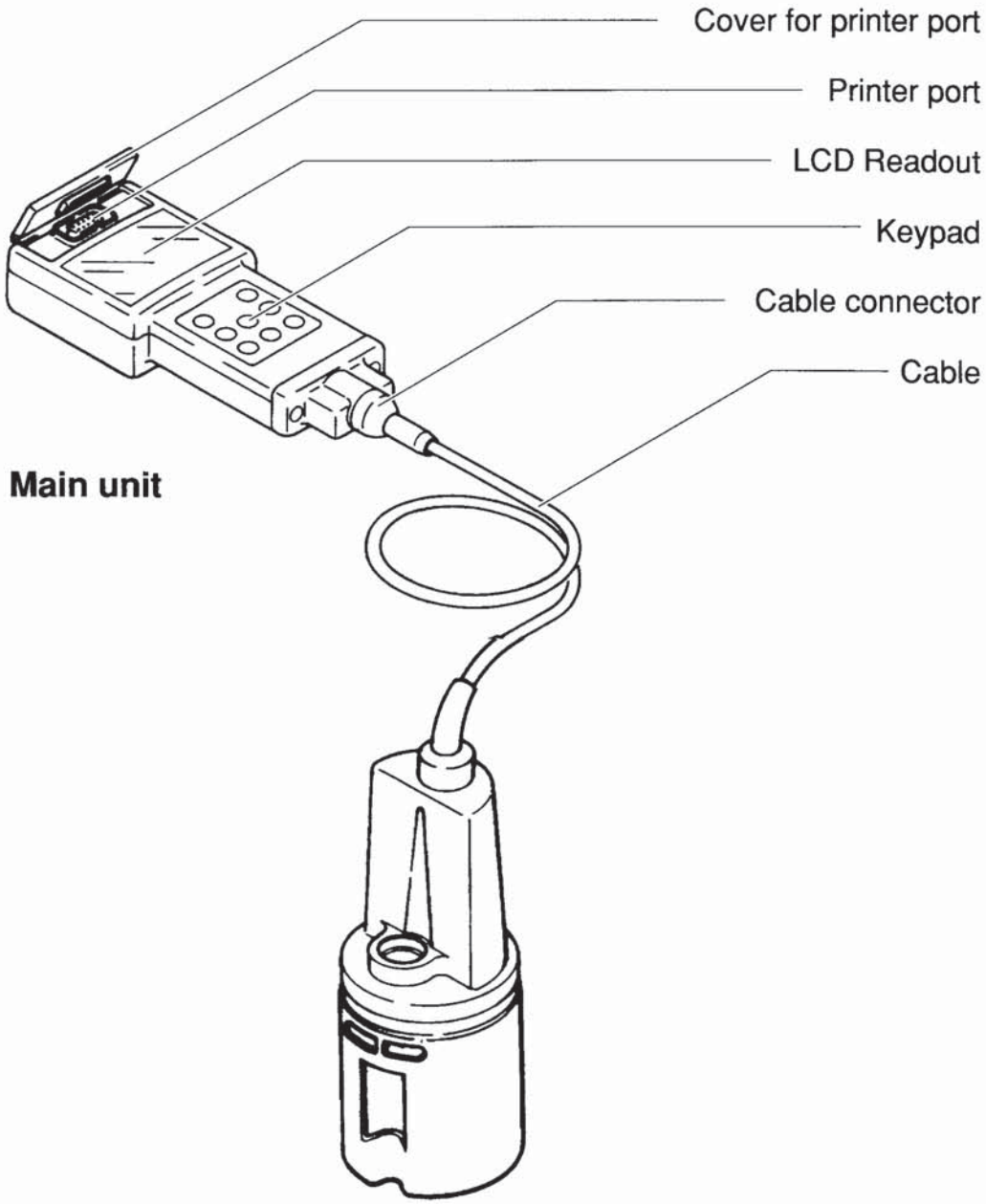
Getting Started

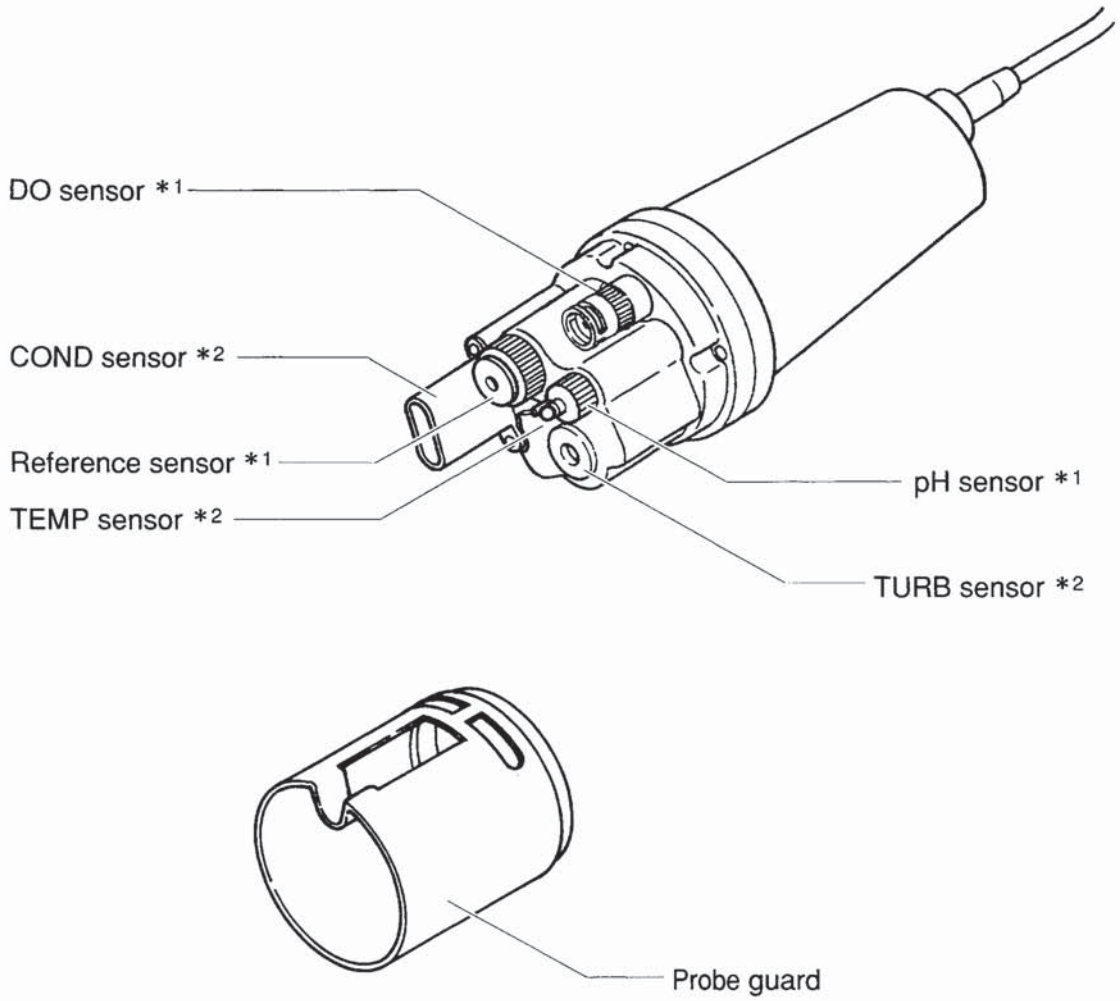
This section first gives an overview of the U-10. It then shows how to set up your U-10 by inserting the DO sensor and the battery.

Configuration of the U-10

Main unit



Probe

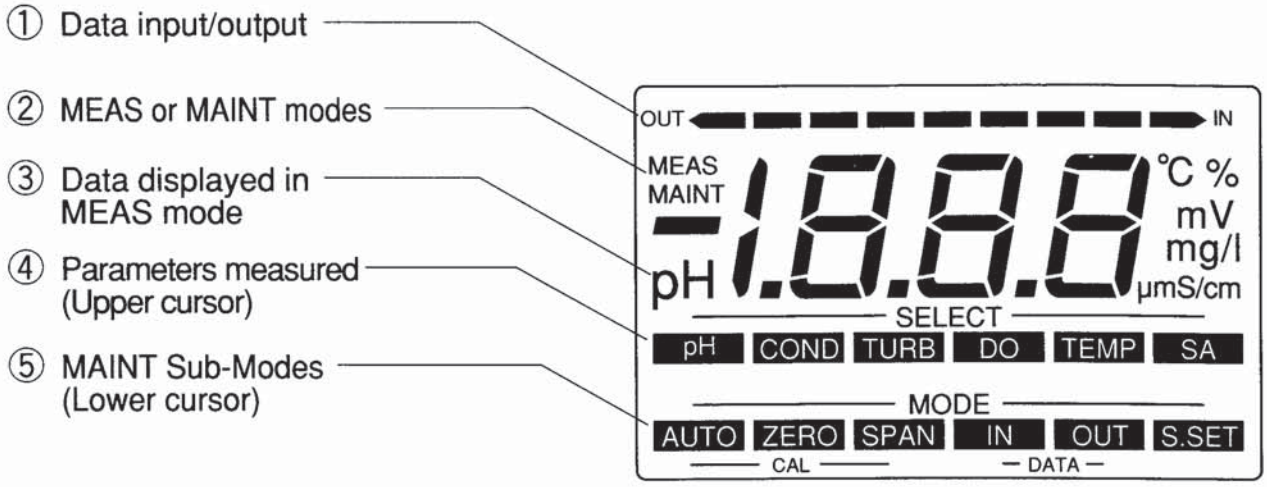


*1. Removable
DO (Dissolved oxygen)
Reference
pH

*2. Non-removable
COND (Conductivity)
TEMP (Temperature)
TURB (Turbidity)

The Readout

The readout has two main functions: (1) it displays the results of measurements, and (2) it serves as a message board to show the operating status of the U-10.



① Data input/output

OUT ▬▬► Data output

◄▬▬ IN Data input

② MEAS or MAINT modes

The U-10 may be used in one of two modes: Measurement (MEAS) mode or Maintenance mode.

MEAS the U-10 is ready to make 6-parameter measurements

MAINT the U-10 is ready for other operations, e.g., calibration, data input/recall, or salinity setting

③ Data displayed in MEAS mode

- 6-parameter results:
  pH, conductivity, turbidity, DO, temperature, and salinity
- Designated value for salinity setting
- Error codes

④ Parameters measured

Value displayed on readout is highlighted by upper cursor.

pH	pH
COND	Conductivity
TURB	Turbidity
DO	Dissolved-Oxygen
TEMP	Temperature
SAL	Salinity

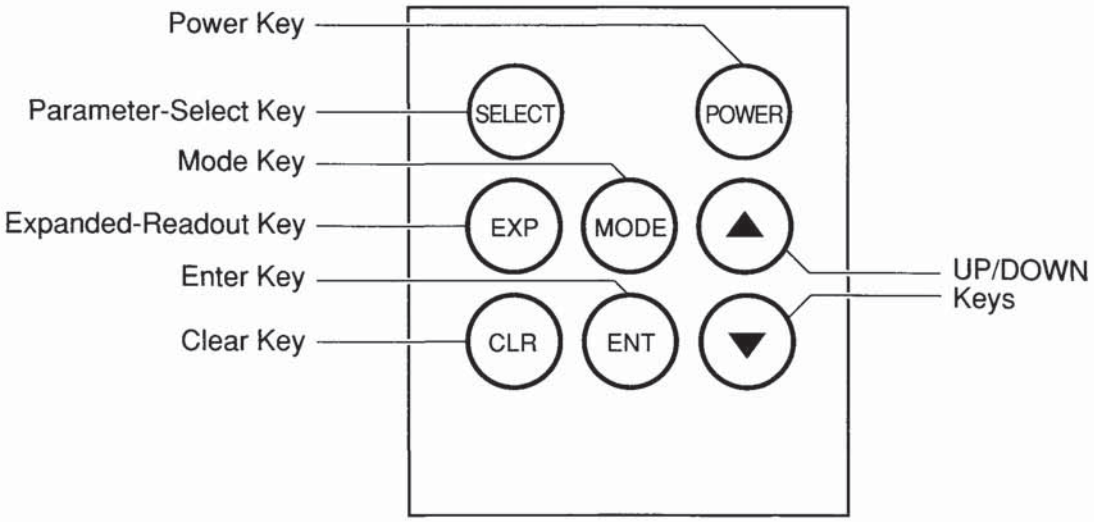
⑤ MAINT Sub-Modes

One of six Sub-Modes selected is highlighted by lower cursor.

AUTO	Automatic 1-point calibration
ZERO	Manual zero calibration
SPAN	Manual span calibration
IN	Data input
OUT	Data output (recall)
S.SET	Salinity setting correction

The Keypad

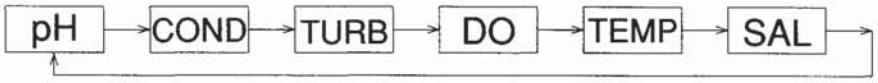
The U-10 is operated by the keypad on the main unit, which has eight surface-sealed keys, as illustrated.



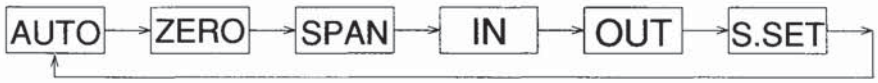
Power Key (POWER)
Turns the main unit ON/OFF.

When this key is pressed to turn the U-10 ON, the readout comes in the MEAS mode, showing the parameter last displayed in the previous measurement. If the U-10 is left with the power ON for 30 minutes without any of the keys being activated, the power will be turned OFF automatically.

Parameter-Select Key (SELECT)
Use this key to move the upper cursor to the measured parameter you want to show on the readout. It toggles through the six parameters in order:



Mode Key (MODE)
Toggles back and forth between MEAS and MAINT modes. When in the MAINT mode, this key toggles the lower cursor through the six maintenance Sub-Modes.



Expanded-Readout Key (EXP)
Toggles between (1) standard readout value and (2) expanded readout, for greater resolution, with decimal point moved one digit to the left.

Enter Key (ENT)
This acts like the RETURN Key or Enter Key on a computer keyboard. The U-10 Enter Key has four main functions, depending on which mode the unit is in.

1. In the AUTO Sub-Mode: Press this key to start automatic calibration.
2. In either the ZERO or SPAN Sub-Modes: Used in manual calibration to set the value for the standard solution being used.
3. In the IN Sub-Mode: Inputs data being measured to memory.
4. In the OUT Sub-Mode: Recalls values from one of the 20 Data-Set Nos. that is now shown on the readout. Prints data when a printer is connected.

Clear Key (CLR)
This acts like the ESCAPE Key on a computer keyboard. It has three main functions, depending on which mode the unit is in.

1. In the AUTO Sub-Mode: Aborts the auto-calibration now in progress.
2. In the IN Sub-Mode: Deletes data in memory from all 20 Data-Sets.
3. When the readout shows an error code: Clears the error code from the readout.

UP/DOWN keys
Use these keys to select values when in one of the MAINT Sub-Modes. They have two main functions.

1. In either the ZERO or SPAN Sub-Modes: Use these keys to select value for the standard solution.
2. In the OUT mode: Used to toggle through the 20 Data-Set Nos. to select the one you wish to recall.

Setting up the U-10

Preparations of the pH sensor and the reference sensor

1. Remove the protective rubber cap from the pH sensor.
2. Remove the sealing tape from the reference sensor.

Inserting the DO sensor

WARNING

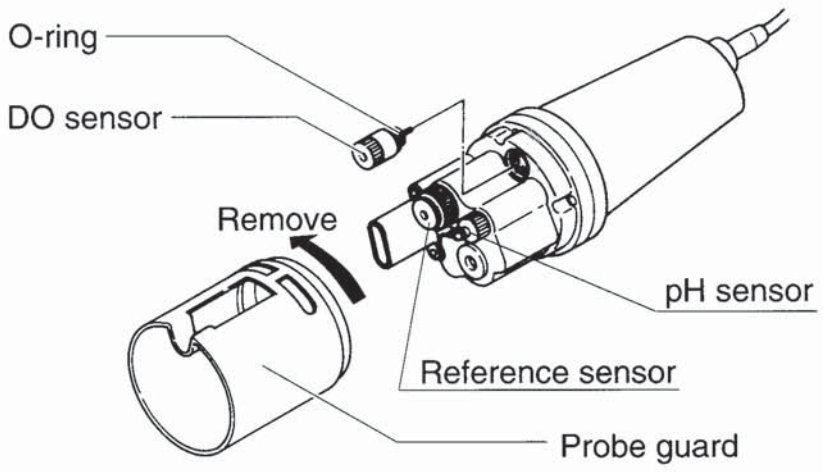
The DO sensor contains a strong alkaline solution. Should any of this solution come in contact with your clothing or skin, wash it away immediately with plenty of water. Be especially careful not to allow any of the liquid in the DO sensor to get in your eyes.

The Dissolved-Oxygen (DO) sensor has a delicate membrane that can easily be ruptured. For safety's sake, the U-10 is shipped to you with the DO sensor packed separately. You should insert the DO sensor when you unpack your U-10 unit.

1. Make sure that the DO sensor has the correct O-ring, as shown.

2. First, fit the DO sensor lightly into its socket, and then put on the probe guard to align it correctly.

3. Then, tighten the DO sensor securely to the probe body. When doing this, be especially careful not to damage the membrane, which is located in the front of the DO sensor.



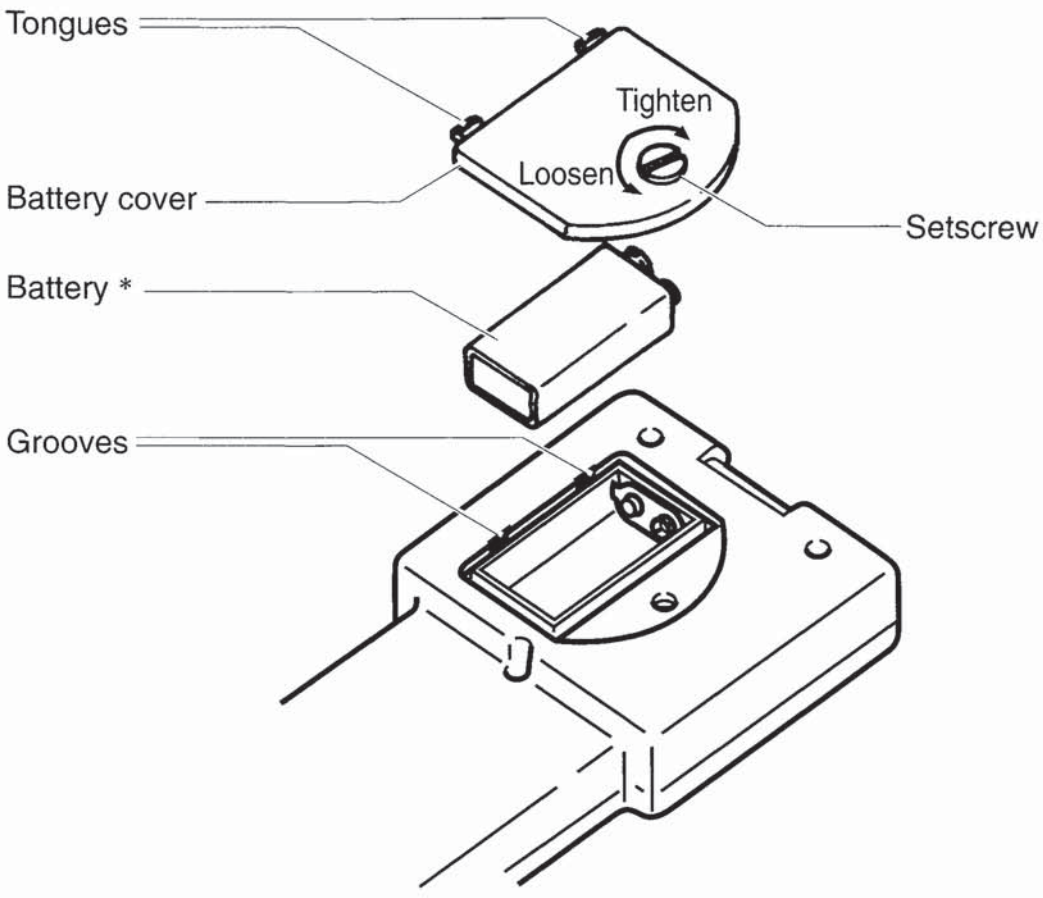
Inserting the battery

The U-10 is shipped from the factory with the battery packed separately.

The battery may be inserted by loosening the set-screw on the battery cover and pulling up the cover. Make sure that the plus and minus poles of the battery match the terminals correctly.

If the readout shows the message Er 1, it means that the battery is defective or exhausted and should be replaced.

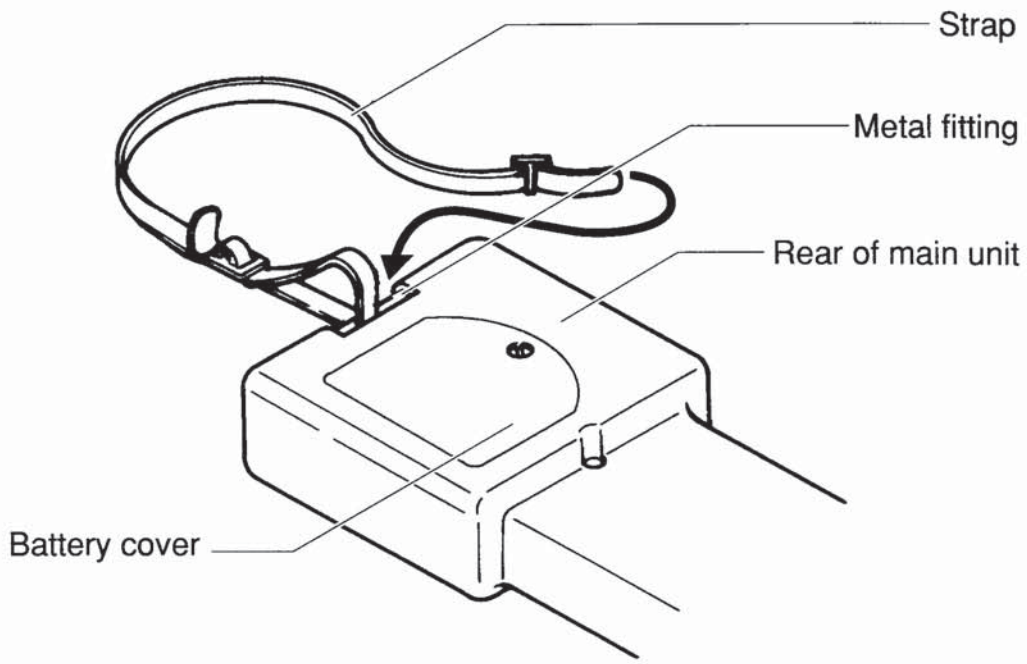
If you are replacing the battery and already have data stored in the U-10 memory that you wish to save, be sure to turn OFF the POWER Key before you remove the old battery. This will assure that data stored in memory will be maintained by the internal backup battery.



* Use a 9V-battery.

Attaching the carrying strap

Hook both ends of the strap through the metal fitting on back of the main unit, as illustrated.



Section 2

Making Measurements

Making a measurement with the U-10 Water Checker is extremely simple. Just turn on the power and place the probe in the sample of water you wish to measure.

All six parameters are measured simultaneously. These parameters may be stored in memory, printed out, or viewed one-by-one on the LCD readout. For printing and data storage, see the appropriate sections following this one. To view the parameters one-by-one on the readout, use the SELECT Key to toggle the upper cursor through them.

While the U-10 is both rugged and precise, the key to accurate measurements is cleanliness and frequent calibration. It is essential to clean the U-10 thoroughly after each measurement, and it is recommended that you re-calibrate your U-10 as frequently as possible. For best results, you should recalibrate it before each measurement session. Cleaning and calibration procedures are described below in this section and in the following one.

How to make a measurement

1 Turn the power on.

2 Gently place the probe into the water sample.

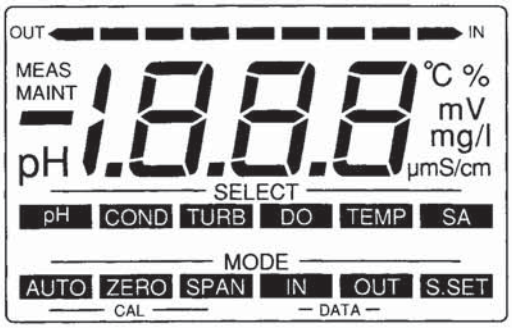
Basically, that's all there is to it: just turn it on and put the probe in the sample. Of course, the U-10 can do many sophisticated things with the sample data, and for best results, you should be careful about calibrating the unit and maintaining it in good condition. This is explained in detail below and in the next section.

Be careful!
Never drop or throw the probe into the water. It is a precision instrument containing five delicate sensors and five pre-amps; you can damage it beyond repair by unnecessary rough handling.

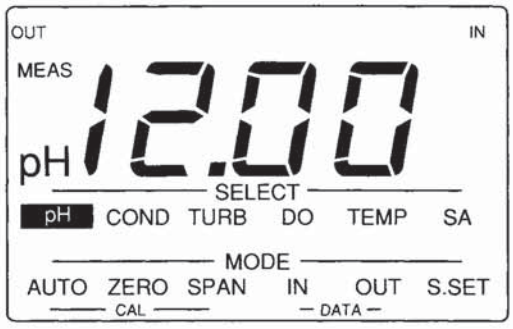
Initial readout

POWER

When you first turn the power on, the U-10 will be in the MEAS mode, the readout will look like this, with all the LCD segments activated.



After about two seconds, the readout will change to show that a new measurement is being made. The readout will show the last parameter that the upper cursor was on when the previous measurement was made, i.e., pH as illustrated here.



(Expanded readout shown)

The display of the decimal point in the readout mode will also be in the same format as was selected with the EXP Key in the previous measurement, i.e., standard or expanded (as illustrated here).

Select the parameter you want shown on the readout of the measured data

All six parameters are automatically measured at once. Use the SELECT Key to toggle the upper cursor to the parameter you want.

pH	: pH
COND	: Conductivity
TURB	: Turbidity
DO	: Dissolved oxygen
TEMP	: Temperature
SAL	: Salinity

To get a uniform reading, slowly move the probe up and down to circulate the water through it. (Move it 1 foot (30 cm) per sec.) Then wait for the readout to stabilize while doing this.

Expanded readout

Use the EXP readout mode when you wish to see the results with one additional decimal place of accuracy. The EXP Key toggles the readout back and forth between standard to expanded display. The table below shows the result of using the EXP readout mode for each of the six parameters.

Table 1. Accuracy of expanded readout

Parameter	Range of measurement	Accuracy	
		Standard readout	Expanded readout
pH	0-14 pH	0.1 pH	0.01 pH
COND	0-1 mS/cm	0.01 mS/cm	0.001 mS/cm
	1-10 mS/cm	0.1 mS/cm	0.01 mS/cm
	10-100 mS/cm	1 mS/cm	0.1 mS/cm
TURB	0-800 NTU	10 NTU	1 NTU
DO	0-19.9 mg/*l*	0.1 mg/*l*	0.01 mg/*l*
TEMP	0-50°C	1°C	0.1°C
SAL	0-4%	0.1%	0.01%

Note that the salinity parameter is the only value not measured directly with its own sensor. The U-10 obtains salinity by converting the conductivity value. If large amounts of conductive ions other than salt-water components are present in the sample, an error may occur. Be cautious when interpreting the salinity results.

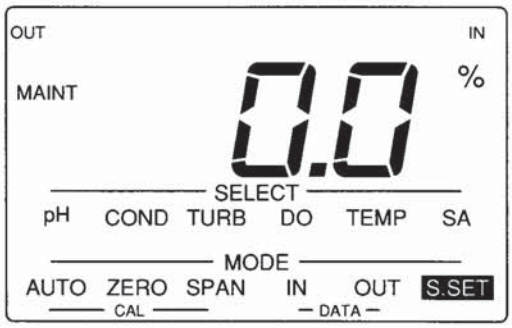
Measuring fresh water or salt water?

The U-10 can be set to the salinity for either fresh water or salt water when measuring DO. This is done by using the S.SET Sub-Mode.

Measuring fresh water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.

2. Once you are in the S.SET Sub-Mode, use the UP/DOWN Keys to select the salinity value. For fresh water, set the salinity to 0.0%.



3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.

4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

Measuring salt water

1. First, use the MODE Key to put the U-10 in the MAINT mode. Keep pressing the MODE Key to toggle the lower cursor to the S.SET Sub-Mode.

2. For salt water, set it to *A i.e.*, for auto-salinity.
   The *A* setting should be sufficient for measurements of normal sea water with a salinity value close to 3.3%. For sea water of an unusual salinity, however, and where the value is otherwise known, you may wish set the value manually to any salinity within the range of 0.0%-4.0%. (You may also possibly want to use a manual setting if, for example, the COND sensor is malfunctioning but it is still desirable to take readings of the other parameters.)

3. Finally, press the ENT Key to complete the salinity setting while in the S.SET Sub-Mode.

4. When the salinity setting has been made, switch back to the MEAS mode by pressing the the MODE Key.

After measurement: Cleaning and storing the U-10

1. Turn OFF the power.

2. Wash the probe thoroughly with tap water. Be sure to flush off all of sample solution from the probe.

Storing the U-10 for brief periods, *i.e.*, about 1 week or less:

Fill the calibration beaker with tap water and fit the probe over it.

For longer storage

The pH sensor must always be kept moist. Fill the small rubber cap with water and use it to cover the pH sensor.

The KCl internal solution in the reference sensor may seep out over time. Place vinyl tape around the O-ring portion to prevent this.

If you are going to store the U-10 for a prolonged period without using it, remove the battery from the main unit.

Section 3

Calibrating the U-10

The U-10 Water Checker may be calibrated either manually or automatically. The 4-parameter auto-calibration procedure is quite handy and should be sufficient for most measurement operations.

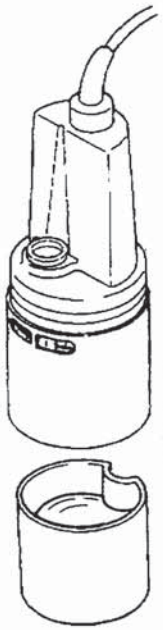
Manual calibration for each of the four parameters is more accurate but, of course, also more time-consuming. This method should be used for more precise measurement. The manual calibration procedure is explained below in detail, following the description of the auto-calibration procedure.

The auto-calibration procedure is extremely simple. The U-10 Water Checker uses just a single solution to do a simultaneous calibration of four parameters: *pH*, *COND*, *TURB*, and *DO*. Your U-10 comes with a bottle of standard phthalate pH solution and a calibration beaker for this purpose.

Auto-calibration procedure

Fill the calibration beaker to about 2/3 with the standard solution. Note the line on the beaker.

Fit the probe over the beaker, as illustrated. Note that the beaker is specially shaped to prevent the DO sensor from being immersed in the standard solution. This is because the DO auto-calibration is done using atmospheric air.



Calibration beaker

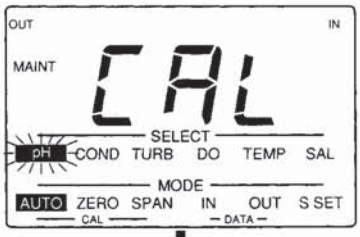
MODE

With the power on, press the MODE Key to put the unit into the MAINT mode. The lower cursor should be on the AUTO Sub-Mode; if it is not, use the MODE Key to move the lower cursor to AUTO.

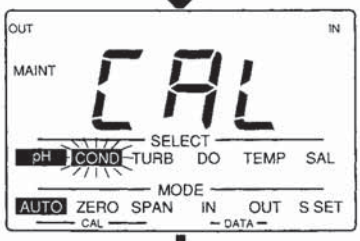
ENT

With the lower cursor on AUTO, press the ENT Key. The readout will show CAL. Wait a moment, and the upper cursor will gradually move across the four auto-calibration parameters one-by-one: *pH, COND, TURB,* and *DO*. When the calibration is complete, the readout will briefly show End and then will switch to the MEAS mode.

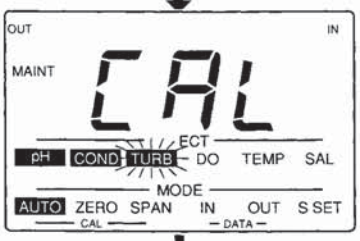
The upper cursor will blink while the auto-calibration is being made. When the auto-calibration has stabilized, the upper cursor will stop blinking.



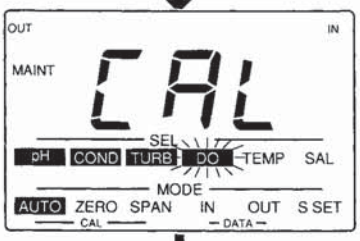
First, pH is being auto-calibrated



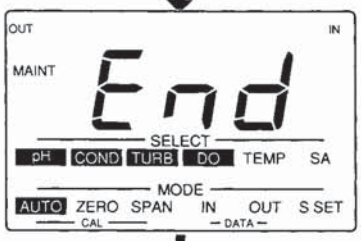
Then, COND is being auto-calibrated



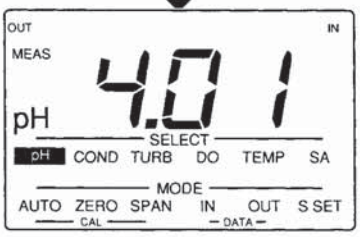
Next, TURB is being auto-calibrated



Finally, DO is being auto-calibrated



Auto-calibration now ends

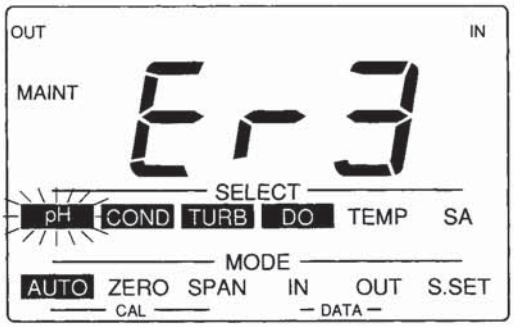


And the readout switches to the MEAS mode

Note: If you wish to abort the auto-calibration for any reason, press the CLR Key. The parameters auto-calibrated so far will be stored in memory.

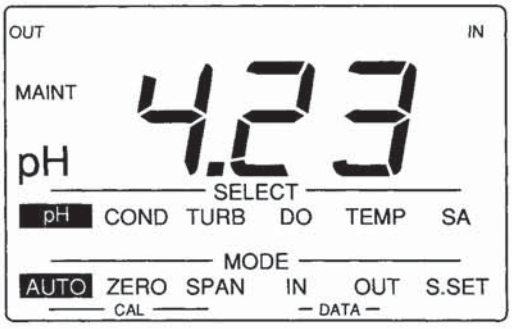
Auto-calibration error

After the DO auto-calibration, if the unit does not switch to the MEAS mode as it should, and the readout shows either Er3 or Er4, an auto-calibration error has occurred. Parameters will blink where an error occurred.



pH auto-calibration error

If this happens, re-do the auto-calibration. First, press the CLR Key to cancel the error code.



Then press the ENT Key to re-start the auto-calibration. Restart the auto-calibration beginning again with pH.

Manual (2-point) calibration procedures

For normal measurements, the 4-parameter auto-calibration described above is sufficiently accurate. However, you may wish to do a parameter-by-parameter, 2-point manual calibration of one or more of the four parameters. This is recommended either for high-accuracy measurements, especially when using the expanded readout mode. It is necessary if a new probe is being used for the *first time*.

Parameters to be calibrated manually.

pH	• Zero	(see page 24.)
	• Span	(see page 25.)
COND	• Zero	(see page 28.)
	• Span	(see page 29.)
TURB	• Zero	(see page 31.)
	• Span	(see page 31.)
DO	• Zero	(see page 32.)
	• Span	(see page 33.)

Parameters not to be calibrated.

Sample temperature
Salinity

pH calibration

pH calibration on the U-10 is done using two commercially-available standard solutions of different pH values, one for the zero calibration, the other for the span calibration. Note that the temperature characteristics of the various standard solutions that are available may differ; therefore, before using these two solutions to make the pH calibration, carefully measure the temperature and determine the temperature characteristics of each.

Preparation

Wash the probe 2-3 times, using de-ionized or distilled water. Place it in a beaker of each standard solution.

1. Zero calibration

Use a pH7 standard solution for the zero calibration.

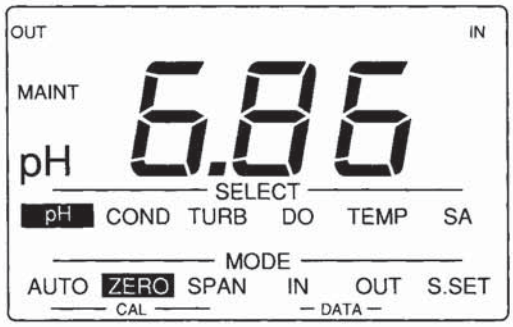
Operation

1. With the power on, press the MODE Key to put the unit into the MAINT mode.

2. Press the MODE Key again to move the lower cursor to ZERO.

3. Use the SELECT Key to move the upper cursor to pH.

4. When the readout has stabilized, use the UP/DOWN Keys to select the value of the pH 7 standard solution at the temperature of the sample. Refer to Table 2 for pH values of standard solutions at various temperatures.



5. Press the ENT Key to complete the zero calibration for pH.

2. Span calibration

Use either a pH4 or a pH9(10) standard solution for the span calibration.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.

2. As in Step 4. above in zero calibration, when the readout has stabilized, use the UP/DOWN Keys to select the value of the standard solution (i.e., either pH4 or pH9) at the temperature of the sample. Again, refer to Table 2 for pH values of standard solutions at various temperatures.

3. Press the ENT Key to complete the span calibration for pH.

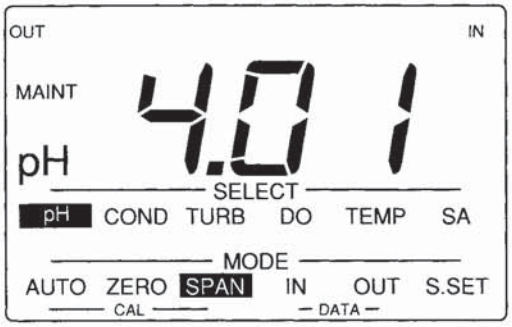


Table 2 pH values of standard solutions at various temperatures*

Temperature ℃ / °F	pH2[a]	pH4[b]	pH7[c]	pH9[d]	pH10[e]	pH12[f]
0 / 32	1.67	4.01	6.98	9.46	10.32	13.43
5 / 41	1.67	4.01	6.95	9.39	10.25	13.21
10 / 50	1.67	4.00	6.92	9.33	10.18	13.00
15 / 59	1.67	4.00	6.90	9.27	10.12	12.81
20 / 68	1.68	4.00	6.88	9.22	10.06	12.63
25 / 77	1.68	4.01	6.86	9.18	10.01	12.45
30 / 86	1.69	4.01	6.85	9.14	9.97	12.30
35 / 95	1.69	4.02	6.84	9.10	9.93	12.14
40 / 104	1.70	4.03	6.84	9.07	9.89	11.99
45 / 113	1.70	4.04	6.83	9.04	9.86	11.84
	1.71	4.06	6.83	9.01	9.83	11.70

a : oxalate, b : phthalate, c : neutral phosphate, d : borax,
e : carbonate, f : Sat.calcium hydroxide solution

* These pH valves are for Japanese standard solutions. Should you prefer to use different standard solutions, be sure to make the proper adjustments in calibration.

COND calibration

The U-10 can measure conductivity in the range of 0-100 mS/cm. Depending on the sample concentration, however, the U-10 automatically selects the proper range out of its three possible ranges of 0-1 mS/cm, 1-10 mS/cm, and 10-100 mS/cm.

Therefore, if you are doing a manual calibration for COND, this must be done for each of the three ranges. However, since the zero point is common for all three ranges, only the three one-point span calibrations need be done separately.

Preparing the standard solution for COND span calibration

This solution uses a potassium chloride as a reagent. For greater accuracy, the solution should be freshly prepared each time. If it is unavoidable to use a stored solution, be sure to keep it tightly capped in a polyethylene or hard glass bottle. The shelf life of this solution is six months. Date-stamp the bottle for reference. Never use a KCl standard solution that has been stored for more than six months: the calibration accuracy may be adversely affected.

Use potassium chloride powder of the best quality commercially available. Dry the powder for two hours at 105°C, and cool it down, in a desiccator. Weigh out an appropriate amount of dried and cooled potassium chloride powder according to the table below. Make the potassium chloride standard solution as shown.

Table 3 Making the potassium chloride standard solution

KCl standard solution	KCl weight g	Conductivity* mS/cm	Range to be calibrated mS/cm
0.005N	0.373	0.718	0-1
0.05N	3.73	6.67	1-10
0.5N	37.28	58.7	10-100

* Value at the temperature, 25°C

To prepare the standard solution, use a 1-liter volumetric flask. First, dissolve the KCl in a small amount of de-ionized or distilled water. Then fill the flask with de-ionized or distilled water up to the 1-liter line. Finally, shake the solution to mix it thoroughly.

1. Zero calibration

This calibration is carried out in atmospheric air; no solution is needed.

Preparation

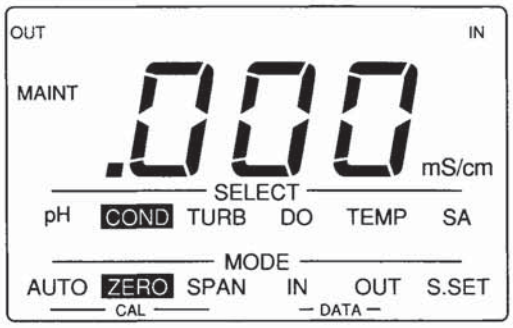
Wash the probe 2-3 times, using de-ionized or distilled water. Shake the probe to remove any water droplets from the COND sensor. Then allow it to dry by exposing it to fresh air.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to COND.

3. Use the UP/DOWN Keys to set the readout to zero.



4. Press the ENT Key. This completes the zero calibration for COND.

2. Span calibration

This procedure uses a standard solution of potassium chloride. For best results, a fresh batch of the solution should be prepared each time. See page 27 for details.

Preparation

Wash the probe 2-3 times using de-ionized or distilled water. Following this, wash it 2-3 times in the KCl standard solution you have prepared. Then place the probe in a beaker of the KCl solution maintained at a temperature of 25±5°C.

Operation

1. Use the MODE Key to move the lower cursor to SPAN.

2. After the readout stabilizes, as you did for the pH calibration, use the UP/DOWN Keys to select set the value of the KCl standard solution, referring to the KCl table.

3. Press the ENT Key to complete the span calibration for this COND range.

4. Repeat this procedure for the three ranges, using each of three values of KCl standard solutions.

TURB calibration

Use good-quality de-ionized water, which may be considered as having a turbidity of zero. If that is not readily available, distilled water may be used instead. When doing the turbidity zero calibration, it is particularly crucial that you clean the probe thoroughly. Never use a dirty probe; otherwise the calibration will be unreliable.

Preparing the standard solution for TURB span calibration

1. Weigh out 5.0 g of hydrazine sulfate.
2. Dissolve this in 400 m*l* of de-ionized or distilled water.
3. Then weigh out 50 g of hexamethylenetetramine, and dissolve it in 400 m*l* of de-ionized or distilled water.
4. Mix these two solutions, add enough de-ionized or distilled water to make 1,000 m*l*, and stir the mixed solution thoroughly.
5. Allow this solution to stand for 24 hours at a temperature of 25±3°C.

   The turbidity of this solution is equivalent to 4000 NTUs. The shelf-life of this solution is six months; i.e., this 4,000-NTU value will remain accurate for a maximum of six months.

   Each time you carry out this calibration, it is necessary to dilute the 4,000-NTU standard solution to prepare an 800-NTU standard solution for calibration. To do this, measure out 50 m*l* of the 4,000-NTU solution into a 250-m*l* measuring flask.

   It is recommended that you use a rubber pipette aspirator for this. Then add de-ionized or distilled water up to the 250-m*l* line.

   The standard solution used here for the turbidity calibration will precipitate easily. Therefore, be sure to stir the solution thoroughly before use.

1. Zero calibration

Preparation

Wash the probe thoroughly 2-3 times using de-ionized or distilled water. Shake off excess water droplets, and then place it in a beaker of de-ionized or distilled water.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to TURB.

3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.

4. Press the ENT Key to complete the zero calibration for TURB.

2. Span calibration

Preparation

Wash the probe thoroughly, using de-ionized or distilled water. Shake off excess water droplets. Then place it in a beaker of the 800-NTU solution you have prepared for this purpose.

Operation

1. Stir this 800-NTU span standard solution thoroughly.

2. Use the MODE Key to move the lower cursor to SPAN.

3. After readout has stabilized, i.e., about 60 to 90 seconds, set the readout to "800" NTU, which is the value for this standard solution.

4. Press the ENT Key to complete the span calibration for TURB

DO calibration

Unlike the other calibration procedures, the solution for the DO calibration cannot be stored for use; because the amount of dissolved oxygen in the solution is crucial, a fresh batch must be prepared each time, just before it is used in the DO calibration.

1. Zero calibration

Use a solution of sodium sulfite dissolved in either de-ionized water or tap water.

Preparation

1. Add about 50g of sodium sulfite to 1,000 ml of water (either de-ionized water or tap water will do). Stir this mixtuer to dissolve.

2. Wash the probe 2-3 times in tap water, and place it in the zero standard solution.

Operation

1. Use the MODE Key to move the lower cursor to ZERO.

2. Use the SELECT Key to move the upper cursor to DO.

3. After the readout has stabilized, set it to 0.0, using the UP/DOWN Keys.

4. Press the ENT Key. This completes the zero calibration for DO.

2. Span calibration

Use either de-ionized water or tap water that has been saturated with oxygen in air.

Preparation

1. Put 1 or 2 liters of water in a container (either de-ionized water or tap water will do). Use an air pump to bubble air through the solution until it is oxygen-saturated.

2. Wash the probe 2-3 times in tap water, and put it in the span calibration solution.

Operation

1. First, be sure the U-10 is set for fresh water readings. To do this, set the S.SET Sub-Mode to 0.0%.

2. Then, use the MODE Key to move the lower cursor to SPAN.

3. After the readout has stabilized, while slowly moving the probe up and down in the solution, set the readout value to the appropriate DO value for the temperature of this solution. For DO values at various temperatures, refer to Table 4.

4. Press the ENT Key to complete the span calibration for DO.

Table 4 Amounts of saturated dissolved oxygen in water at various temperatures, salinity = 0.0%

Temperature	DO	Temperature	DO
0 °C	14.16 mg/*l*	21 °C	8.68 mg/*l*
1	13.77	22	8.53
2	13.40	23	8.39
3	13.04	24	8.25
4	12.70	25	8.11
5	12.37	26	7.99
6	12.06	27	7.87
7	11.75	28	7.75
8	11.47	29	7.64
9	11.19	30	7.53
10	10.92	31	7.42
11	10.67	32	7.32
12	10.43	33	7.22
13	10.20	34	7.13
14	9.97	35	7.04
15	9.76	36	6.94
16	9.56	37	6.86
17	9.37	38	6.76
18	9.18	39	6.68
19	9.01	40	6.59
20	8.84		

Section 4

Data Storage and Printout

The U-10 can store up to 20 sets of data, 120 data points, of the values measured for each of the six parameters: pH, COND, TURB, DO, TEMP, and SALINITY. Values stored in memory can be recalled to the readout as desired.

If a printer is connected to the U-10 printer port, whenever a Data-Set is either stored in memory or recalled to the readout, it can also be simultaneously output to the printer.

Storing data

MODE

1. Press the MODE Key to put the U-10 in the MAINT mode.

MODE

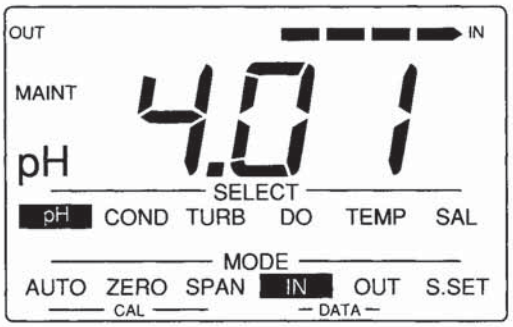
2. Continue to press the MODE Key to move the lower cursor to IN, the *Input* Sub-Mode.

SELECT

3. Use the SELECT Key to move the upper cursor to the parameter you wish to see on the readout.

ENT

4. When the readout stabilizes on a value, press the ENT Key. This will automatically input the set of six parameters for this measurement into memory.



The readout will first show the Data-Set No. for about two seconds. At the top right-hand corner, a dashed arrow points to IN, showing that data is being input. Then each parameter is automatically read into memory, one-by-one from pH to salinity. The upper cursor skips along to show this. If a printer is connected, these six values will also be printed out at the same time.

The upper cursor then returns to pH, with the U-10 still in the IN Sub-Mode.

ENT

5. You may now continue and input another set of data: simply press the ENT Key again.

The Data-Set No. will automatically advance one digit, and the next set of six parameters will be read into memory in the same manner. This procedure can be repeated for up to a total of 20 Data-Sets.

If 20 Data-Sets have been read into memory, the storage capacity is full and no more data may be input. The U-10 will beep three times to indicate the memory is full.

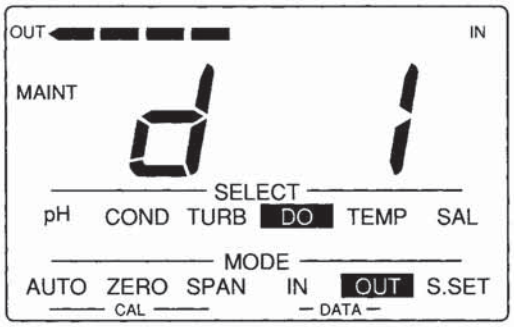
6. To return the readout to the previous setting in the MEAS mode, press the MODE Key again.

Recalling data

1. Press the MODE Key to put the U-10 in the MAINT mode.

2. Continue to press the MODE Key to move the lower cursor to OUT, the *Output* Sub-Mode. The readout will show d.1, meaning Data-Set No. 1.

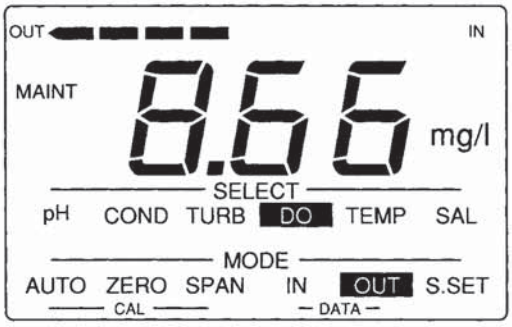
   At the top left-hand corner, a dashed arrow points to OUT, showing that data can be output now to the readout.



3. Use the UP/DOWN Keys to display the Data-Set No. of the values you wish to recall.

4. Use the SELECT Key to move the upper cursor to the parameter you wish to view.

5. Press the ENT Key to display the data on the readout.

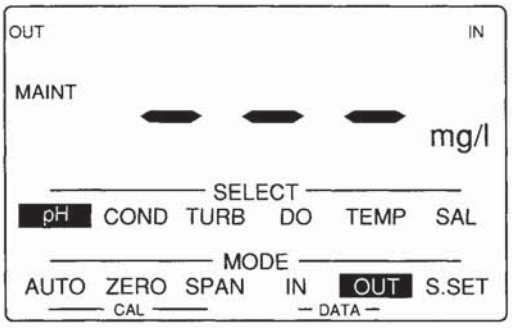


If a printer is connected, all six parameters in this Data-Set will also be printed out at the same time.

ENT

6. When the ENT Key is pressed again, the next Data-Set No. is displayed in order, i.e., d2, if two data sets are in memory. At this point, you can either press the ENT Key again to view the contents of this Data-Set, or you can use the UP/DOWN Keys to go up or down to another Data-Set No.

   If a particular Data-Set is empty, three dashes appear on the readout.



MODE

7. To return the readout to the previous setting in the MEAS mode, press the MODE Key again.

Deleting data

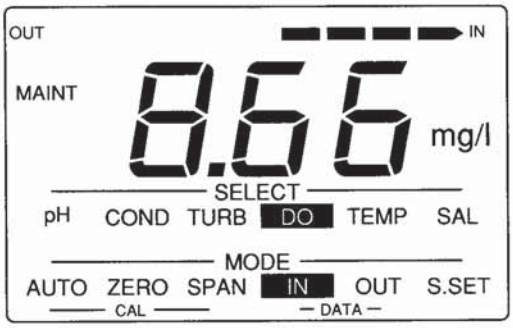
Set the U-10 as if you were going to input data:

MODE

1. Press the MODE Key to put the U-10 in the MAINT mode.

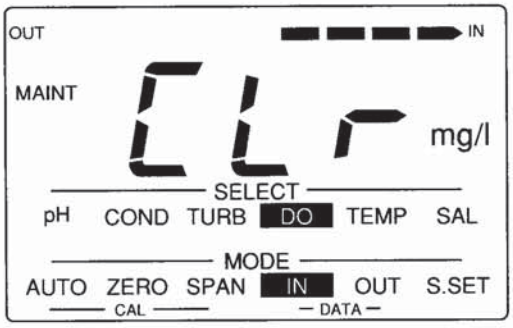
MODE

2. Continue to press the MODE Key to move the lower cursor to IN, the Input Sub-Mode.



CLR

3. Then, to erase all the data from all the Data-Sets in memory, press the CLR Key. The readout will show the message CLr for about two seconds.

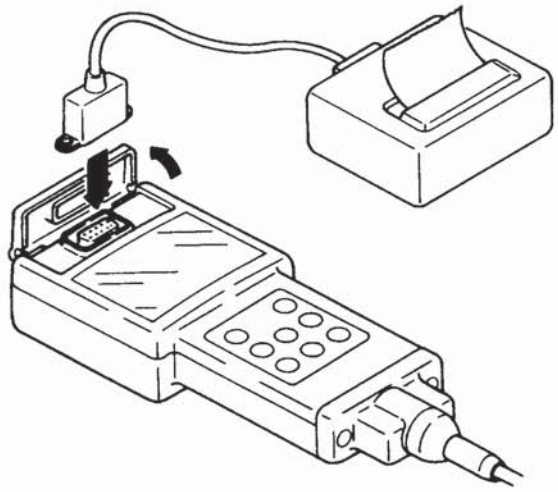


Be careful!
You cannot delete individual Data-Sets. The CLR Key always erases all data from memory.

Printing out data

If a printer is connected to the U-10 printer port, whenever a Data-Set is either stored in memory or recalled to the readout, it is also simultaneously output to the printer.

The U-10 printer port is a standard Centronics parallel port. To connect a parallel printer to the U-10: Open the rubber printer-port cover, located directly over the readout on the main unit, and connect the printer cable.



Note:
When a printer is not being used, disconnect the cable from the U-10 printer port, and close the cover tightly.

- **Sample printout**

```
NO. 1 DATE  /  /
 pH      5.0
 COND    1.5    mS/cm
 TURB  390      NTU
 DO      0.5    mg/l
 TEMP   23      °C
 SAL     3.8    %
NO. 2 DATE  /  /
 pH      3.1
 COND    1.3    mS/cm
 TURB  270      NTU
 DO      0.7    mg/l
 TEMP   25      °C
 SAL     0.1    %
NO. 3 DATE  /  /
 pH      3.1
```

Section 5

Daily Maintenance and Troubleshooting

For accurate measurements and prevention of malfunction, routine careful maintenance of the U-10 is important. In particular, failure to maintain the sensors properly can lead to serious trouble or incorrect measurements. The U-10 is provided with error-code functions for the ready detection of potential problems.

Error Codes

The U-10 has an easy-to-understand error message function so you can spot trouble readily. Error codes are displayed on the readout and the unit will beep if an error occurs.
(Note that if you press an incorrect sequence of keys, the unit will beep three times to indicate you have pushed the wrong key.)

Error Code	Cause	Action
Bad battery Er 1	• Defective or low battery	• Replace battery
Failure in main unit Er2	• Malfunction of memory backup IC	• Push POWER Key to turn the U-10 ON again. If this error code is still displayed, contact your Horiba dealer for repair or replacement.
Zero-calibration error Er3	*for all parameters*	
	• Poor connection in probe-to-main unit cable	• Connect the cable securely.
	• Water in one of the sensor sockets	• Dry out the sensor sockets.
	• Temperature of sample exceeds maximum scale of U-10	• Replace the probe.
	for pH	
	• Contaminated pH sensor.	• Clean the pH sensor.
	• Improper concentration of reference solution in reference sensor	• Replace the reference solution.
	for COND	
	• Contaminated COND sensor	• Clean the sensor, using tooth brush and neutral detergent.

Error Code	Cause	Action
	for TURB • Contaminated or defective LED sensor	• Clean out the tube containing the LED turbidity sensor, using test tube brush and neutral detergent. Never use an abrasives or cleansers for this.
	for DO • Broken DO sensor membrane.	• Check the LED turbidity sensor. If it defective, the entire probe must be replaced. Check DO sensor. If defective, replace.
Span-calibration error Er4	*for all parameters* • Poor connection in probe-to-main unit cable • Water in one of the sensor sockets • Temperature of sample exceeds maximum scale of U-10	• Connect the cable securely. • Dry out the sensor sockets. • Replace the probe.
	for pH • Contaminated pH sensor. • Improper concentration of reference solution in reference sensor	• Clean the pH sensor. • Replace the reference solution.
	for COND • Contaminated COND sensor	• Clean the sensor, using tooth brush and neutral detergent.
	for TURB • Contaminated or defective LED sensor	• Clean out the tube containing the LED turbidity sensor, using test tube brush and neutral detergent. Never use an abrasives or cleansers for this. • Check the LED turbidity sensor. If it defective, the entire probe must be replaced.

Error Code	Cause	Action
Span-calibration error Er4	*DO Auto-calibration*	
	• Broken DO sensor membrane.	• Check DO sensor membrane. If defective, replace.
	• Excessive difference between DO sensor temperature and atmospheric temperature.	• Leave DO sensor in atmosphere for 30-60 min.
	DO aqueous solution calibration	
	• Broken DO sensor membrane.	• Check DO sensor membrane. If defective, replace.
	• Contaminated electrode.	• Clean the electrode using a soft brush, taking care not to scratch membrane.
	• Insufficient agitation of solution.	• Agitate solution thoroughly.
Memory full Er5	• Data-sets for 20 samples are already in memory.	• To delete all data from memory, put the U-10 in the IN Sub-Mode mode and press the CLR Key.
Printer error Er6	• Jammed printer paper.	• Eliminate jamming of printer paper.
	• Poor cable connection .	• Replace the cable.
	• Wrong printer.	• Use proper parallel Centronics printer.
	• Defective printer.	• Replace the printer as necessary.

Normal probe maintenance

Washing the turbidity sensor

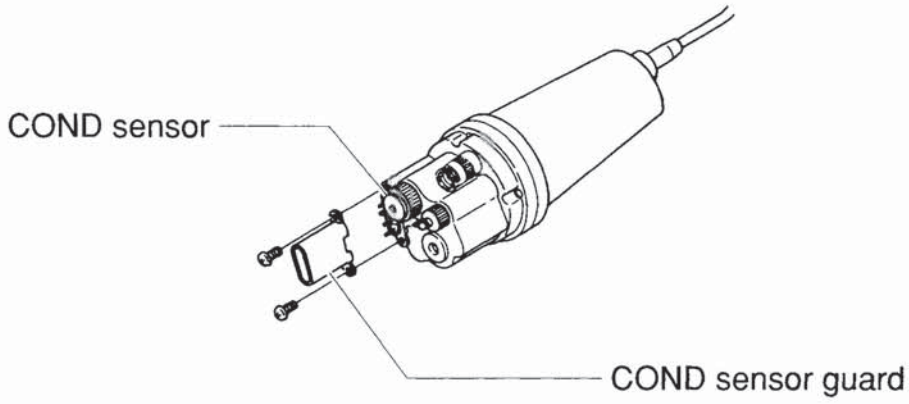
The sensor is a glass tube. Wash out the tube and remove stains carefully, using tap water and a test tube brush.

Be careful not to scratch the inside of the glass tube. Never use abrasives or cleansers.

Cleaning the conductivity sensor

Remove COND sensor guard, and carefully use a soft brush to clean off any dust from the sensor unit.

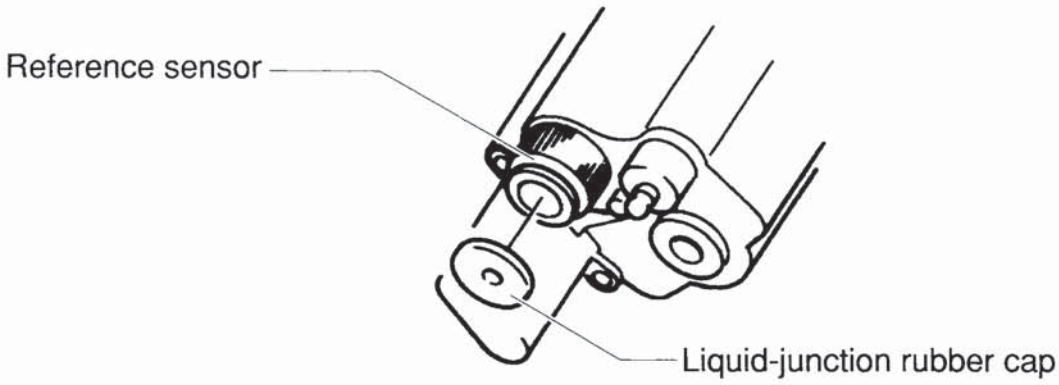
Be sure to replace the COND sensor guard before taking measurements.



Recharging the reference sensor with reference solution

Recharge the reference sensor with reference solution about once every two months, as follows.

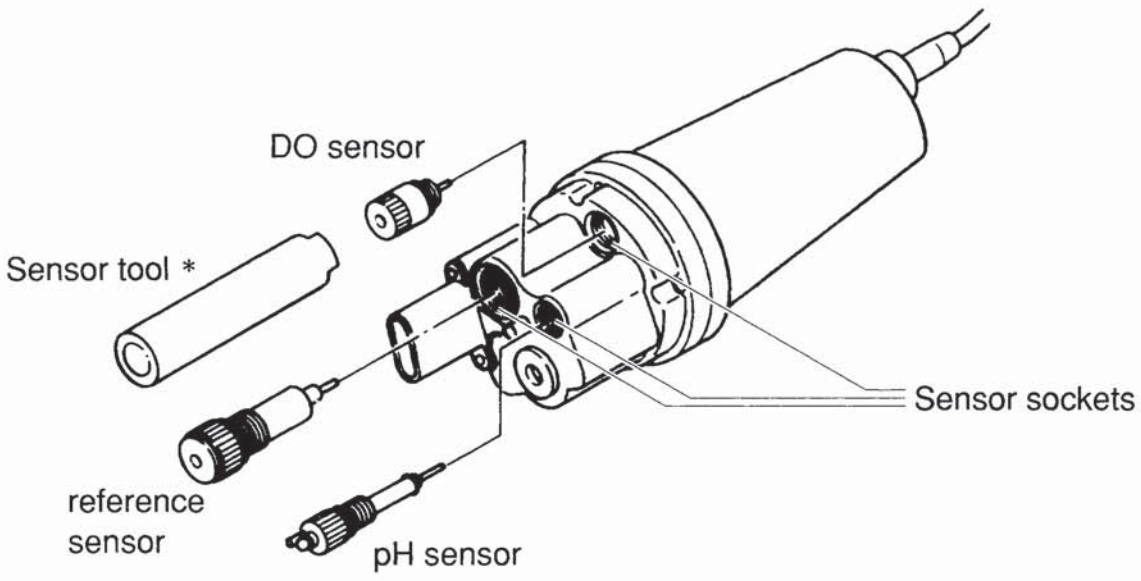
1. Remove the liquid-junction rubber cap from the reference sensor, and pour out the old solution.
2. Fill the reference sensor completely with new reference solution. Make sure there are no air bubbles.
3. Replace the liquid-junction rubber cap.
4. Carefully wash off all excess reference solution from the probe.



Replacing faulty sensors

Three of the U-10's sensors are replaceable: the *pH sensor*, the *reference sensor*, and the *DO sensor*.
These may be replaced as follows.

1. Wipe off any water droplets from the probe.
2. Remove faulty sensor.
3. Insert the new sensor carefully with your fingers.
4. Be careful not to let the sensor sockets get wet.

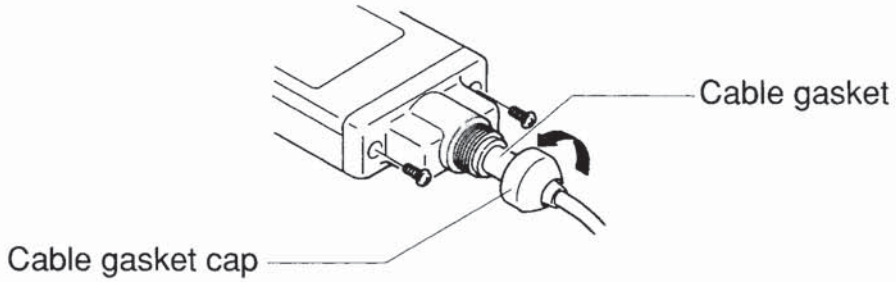


* When replacing the DO sensor, use the sensor tool provided as an accessory.

Replacing a faulty probe

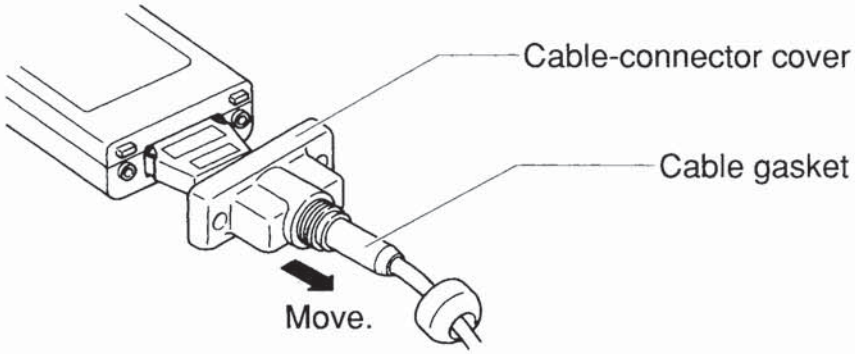
Disconnect the cable from the main unit

1. Loosen the cable gasket cap, and remove cap from gasket.



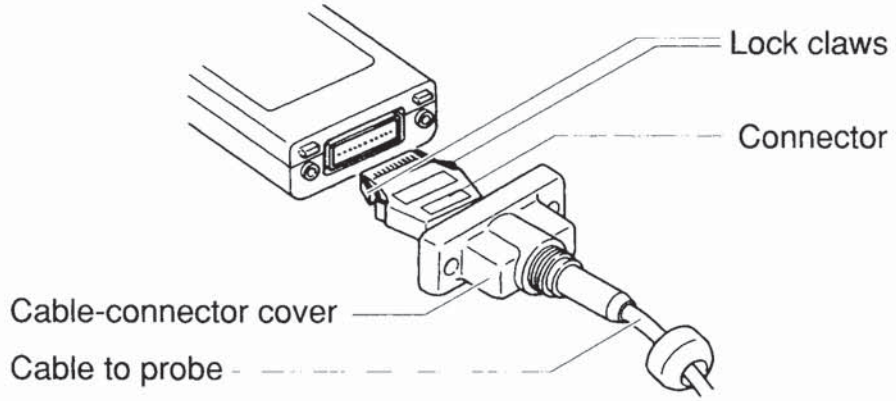
2. Slide back the gasket.

3. Back off the two screws on the cable-connector cover.



4. Slide off the cable-connector cover to expose the connector lock claws.

5. Press lock claws on both sides with your fingers to release the connector. Pull out the connector from the main unit.



Connect the new probe

1. Insert the connector until it clicks.

2. Re-attach the cable-connector cover to the main unit.

3. Slide the cable gasket toward the cable-connector cover, and screw on the cable gasket cap.

   Before you use a new probe for the first time, it is necessary to calibrate it manually for all four parameters. Refer to Section 3, "Calibrating the U-10," for instructions on manual calibration.

Section 6

Reference Materials

The following descriptive information is provided for a better understanding of the U-10 Water Checker and its functions.

Conductivity (COND)

Principle of measurement

Conductivity is an index of the flow of electrical current in a substance.

Salts dissolved in water are separated into cations and anions. Such a solution is called an electrolytic solution. An electrolytic solution has the property of allowing the flow of current according to Ohm's law. This property is referred to *ionic conductivity*, since current flow is due to ion movement in an electrolytic solution. Metals, on the other hand, allow the flow of current by means of electrons. This property is called *electronic conductivity*, which is distinguished from ionic conductivity.

A cube 1 cm on each side, as each shown in **Fig. 1**, is used to demonstrate an electrolytic solution. Two electrode plates are placed on opposite sides, and the cube is filled with a solution. If the resistance between these two electrode plates represented by r (Ω), the conductivity of the solution L ($S.cm^{-1}$) is $L=1/r$. S stands for *Siemens*, a unit of measurement of conductance.

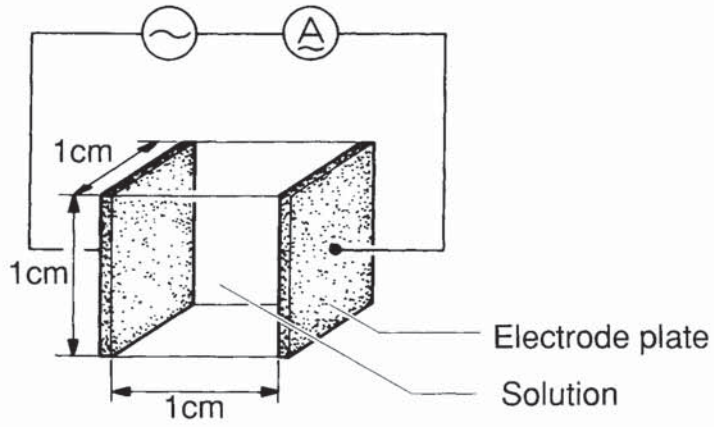


Fig. 1 Definition of conductivity

The most general method for measuring conductivity is based on the above principle, and is called the 2-electrode method. In this method, to take a measurement, it is necessary to allow flow of alternating current between the two electrode plates.

If direct current is sent between them, it will cause electroplating or decomposition, i.e., polarization; this results in inaccurate measurement of conductivity.

Even a flow of alternating current will also cause a certain amount of polarization. Measures must be taken to minimize the effect of this polarization, such as the application of platinum black plating to the electrode surfaces. In spite of such measures, however, the effect of polarization cannot be neglected in conductivity measurements of a high-conductivity solution. This makes accurate measurement difficult. Furthermore, depositions or stains on the electrode surfaces can cause a large apparent resistance, also making accurate conductivity measurement difficult.

The U-10 Water Checker has adopted the 4-electrode method to overcome these disadvantages of the the 2-electrode method. As shown in **Fig. 2**, the U-10 Water Checker uses two voltage-detecting electrodes and two voltage-applying electrodes, for a total of total four electrodes.

The voltage-detecting electrodes are for detecting AC voltage, and the voltage-applying electrodes are for applying AC voltage.

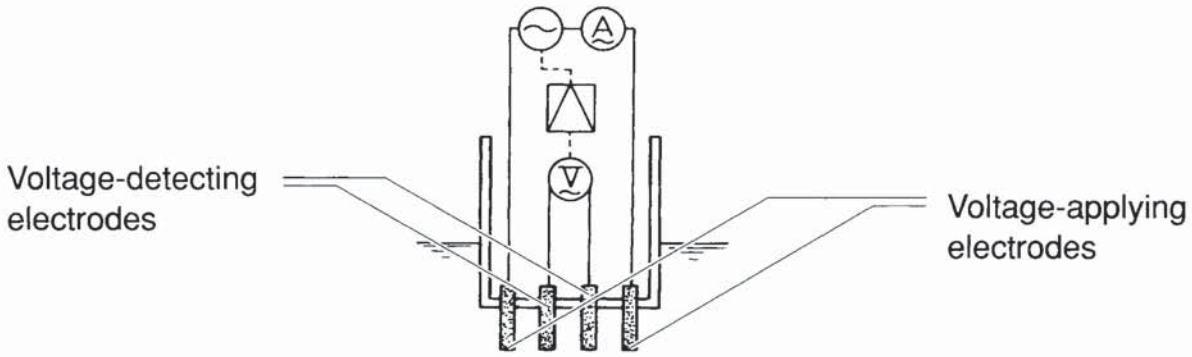


Fig. 2 Principle of the 4-electrode method

Let us assume that the current, *I(A)*, flows in a sample of conductivity *L*—under automatic control of the voltage-applying electrodes—so that the voltage at the voltage detecting-electrodes, *E(V)*, remains constant at all times. Then, the resistance of the sample, *R* (Ω), across the voltage-detecting electrodes is *R=E/I*. The resistance, *R*, of the sample is inversely proportional to its conductivity, *L*. That is, the conductivity, *L*, is proportional to the current, *I*. Accordingly, calibration of a standard solution of known conductivity, *Ls*, enables calculation of conductivity of a sample according to the formula *L=Ls(I/Is)* from the relation of *L:Ls=I:Is*.

Even in the 4-electrode method, polarization occurs, since *AC* current flows in the voltage-applying electrodes. The voltage-detecting electrodes are, however, free from the effects of polarization, since they are separated from the voltage-applying electrodes, and furthermore, current flow is negligible. Therefore, the 4-electrode method is an excellent method to enable measurement of conductivity covering a very high range.

Temperature compensation

In general, the conductivity of a solution varies largely with its temperature. The conductivity of a solution depends on ionic conductivity, described earlier. As the temperature rises, conductivity becomes higher, since ions begin to move more actively.

The temperature coefficient shows the change in % of conductivity per °C, with a certain temperature taken as the reference temperature. This is expressed in units of %/°C. The temperature coefficient assumes the premise that the conductivity of a sample changes linearly according to temperature. Strictly speaking, with actual samples, however, conductivity changes along a curve.

Furthermore, these curves form different shapes depending on the type of sample. In the ranges of smaller temperature changes, however, samples are said to have the temperature coefficient of 2%/°C; this holds for most samples, except in certain special cases. The U-10 Water Checker uses an automatic temperature conversion function to calculate conductivity at 25°C at a temperature coefficient of 2%/°C, based on the measured value of the temperature. Results are displayed on the readout. The U-10's temperature conversion function is based on the following formula.

$$\mathbf{L_{25}=L_t / \{1+0.02(t-25)\}}$$

Where,

L_{25}: Conductivity of solution converted to 25°C (value displayed on U-10)

t: Temperature of solution at time of measurement (°C)

L_t: Conductivity of solution at *t* (°C)

Turbidity (TURB)

Principle of measurement

From among several types of turbidity-measuring methods available, the U-10 uses the light-absorption-scattering method, shown in Fig. 3.
Irradiation of a beam of light onto a sample brings about separation of the beam into (1) the light transmitted by the solution and (2) the light scattered by turbidity components in the sample. In the light-absorption-scattering method, the intensity of both transmitted light and the scattered light are measured using separate receptors, and the turbidity is obtained based on the ratio of the two.

With the U-10, the light source is a pulse-lighting infrared-emission diode. The scattered light is measured at a point 30° offset from the light source. This light-absorption-scattering method has several advantages, including the fact that (1) the actual color of the sample fluid has little effect on the measurement of turbidity, (2) fluctuations in light quantity from the light source are easily compensated for, and (3) it allows the U-10 to be operated with relatively low-power consumption.

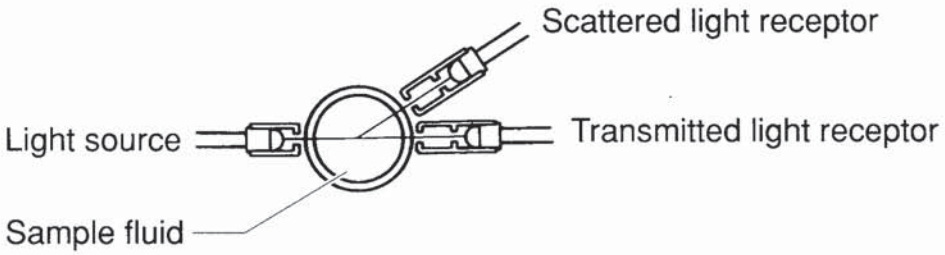


Fig. 3 Principle of the light-absorption-scattering method

NTUs (Nephelometric Turbidity Units)

For the calibration of turbidity, the U-10 uses a standard formazine solution.

Kaolin has been the conventional standard solution for many years. However, the composition of kaolin solutions often vary depending on the country of origin, and turbidity varies with the degree of purify. Furthermore, there is often individual error in preparing the solution. Kaolin is thus known for bringing about very large disparity in measurement results. As a turbidity standard solution, formazine standard solution is now increasingly being used internationally. In view of these facts, the U-10 uses the formazine standard solution for its calibration of turbidity.

In addition, the U-10 uses *NTUs* as the unit of turbidity. Other units conventionally used are formazine degrees and *FTUs*. When the measurement of turbidity is based on the phenomenon of scattering, the use of *NTUs* is preferable, and in fact, these are being used increasingly. It should be noted that *NTUs* used as turbidity units of the formazine standard solution are equivalent to formazine degrees and to *FTUs*.

Salinity (SAL)

The U-10 is designed to measure salinity as well as the other parameters.

Note that the "salinity" referred to here is the salinity of sea water. There is a constant relation between conductivity and salinity at certain temperatures.

Therefore, if data on the conductivity and temperature are available, the corresponding salinity is known. In other words, the salinity measurement of the U-10 is based on the principle of calculating the salt content, making use of the measured values of conductivity and temperature.

Note carefully, therefore, that measured results of all substances whose conductivity is detected are displayed as salinity. For example, the measured result is displayed as NaCl concentration, even if in fact the sample component is, for example, hydrochloric acid (HCl).

Temperature

Temperature changes in water have extreme biological effects on the life cycles of fish and seaweed, as well as on that of the minute organisms that cleanse the water of organic pollutants. In general, as the temperature of water increases, the amount of oxygen dissolved in the water decreases and there is a tendency for the amount of pollutants to increase.

The U-10 uses a thermistor to measure temperature. A thermistor also measures the change in electrical resistance accompany changes in temperature; these changes in resistance are measured by the thermistor and are used to calculate the temperature.

This temperature data is used by the U-10 in four different ways: (1) in pH temperature compensation, (2) in conductivity temperature conversion, (3) in the calculation of salinity, and (4) in dissolved-oxygen temperature compensation.

Dissolved-Oxygen (DO)

Principle of measurement

The "DO" referred to here means the concentration of oxygen dissolved in water.

Fig. 4 shows the principle of measurement using a DO sensor.

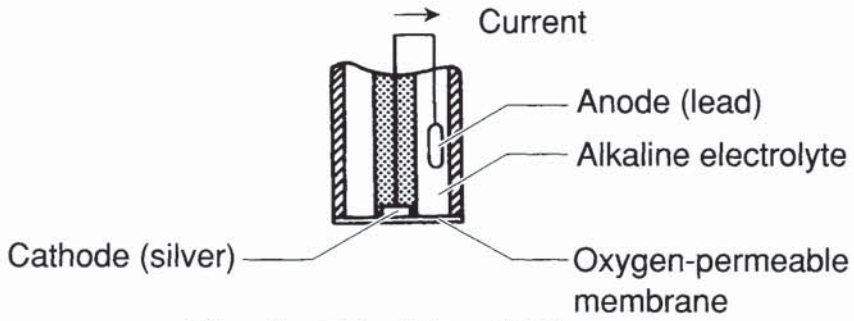


Fig. 4 Principle of DO sensor

A noble metal (silver) is fitted closely to an oxygen-permeable diaphragm to make the cathode; a base metal (lead) is used as the anode. Both are immersed in an alkaline electrolyte with the anode-to-cathode external circuit complete. Oxygen diffusing through the oxygen-permeable membrane causes a reduction reaction at the cathode; this allows flow of current in the external circuit:

$$O_2 + 2H_2O + 4e^- = 4OH^-$$

At the anode, oxidation reaction occurs as follows:

$$2Pb = 2Pb^{2+} + 4e^-$$

The current is proportional to the quantity of oxygen diffusing through the oxygen-permeable diaphragm. Accordingly, measurement of the current makes the DO in a sample known.

The DO measuring method based on this principle is called the *membrane-electrode method*. This method allows convenient measurement of DO, especially when compared with chemical-analysis methods, which need complicated pre-treatment to eliminate the effects of oxidizing or reducing substances.

DO correction for salinity

When a solution and air are in contact and in complete equilibrium (saturated), DO:C[mg/*l*] in the solution, and the oxygen partial-pressure:Ps[MPa] in air are in the following relation:

$$\mathbf{C = Ps/H}$$

H [*MPa*/(mg/*l*)] is referred to as Henry's constant, which depends on the composition of the solution. In general, *C* becomes smaller as the salinity in the solution increases, since *H* becomes larger.

A DO sensor is intended to detect *Ps* in the above expression. Therefore, the DO measurement of an aqueous solution containing salt would be in error if the DO electrode were standardized either on air-saturated pure water or on air. To settle this problem, it is necessary to correct the DO reading based on the salinity of the sample.

Conventional DO meters make this salinity correction by inputting a known salinity value. This poses no problems if the salinity of the sample is known. In practice, however, the salinity of the sample is usually not known, unless measured by a device such as the U-10. Therefore, until now, DO meters have not been practical, even if they were provided with a salinity-correcting function.

The U-10 is capable of measuring the salinity of a sample and automatically correcting the DO reading for the amount salinity measured in the sample.

pH

Principle of measurement

The following is the basic equation for obtaining pH:

$$\mathbf{pH = -\log aH^+}$$

Where,

$\mathbf{aH^+}$ **:** the activity of hydrogen ions

If a thin glass membrane is used to separate two liquids of differing pH values, an electric current will be generated in proportion to the difference between these two pH values. The value of this electrical current, *E(V)* , is shown by the folowing Nernst equation:

$$\mathbf{E = 0.0001983T\ (pH_1 - pH_0) + e}$$

Where,

T : the temperature of the liquids

$\mathbf{pH_1}$ **:** the pH of the internal liquid (i.e., inside the glass membrane)

$\mathbf{pH_0}$ **:** the pH of the sample liquid (i.e., the iliquid outside the glass membrane)

e : the assymetric potential

A conventional glass electrode for measuring pH contains a fluid inside the electrode with a pH of 7. If this is used to measure a sample that also has a pH value of 7, the assymetric potential will be close to 0V. Consequently, when a glass pH electrode is immersed in an acid solution, a positive electric current is generated; when it is immersed in an alkaline solution, a negative electric current is generated.

For actual use in a pH meter, a pair of reference electrodes with extremely stable characteristics is used. These are configured as shown in **Fig. 5**. As shown in **Fig. 5**, it can be seen that the electrical potentials generated in the internal electrodes, *E'* and *E''*, are canceled out by each other, so that the only electrical potential difference obtained is the current generated by the glass membrane, *E*, through the resistance of the membrane, *r*, and transmitted to terminals *G* and *R*.

In pH meters a readout of this voltage between the two terminals is obtained by increasing it with an amplifier. In actual practice, the pH meter is first calibrated using a standard reference solution of known pH, then the pH of the sample liquid is measured.

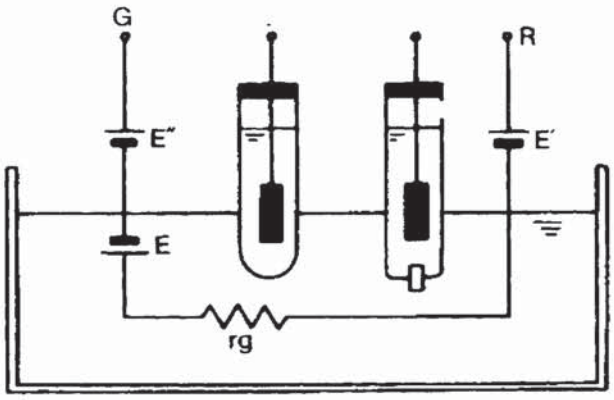


Fig. 5 Principle for Measuring pH

Specifications

pH

Principle	Glass electrode
Range	pH0-14
Resolution	Standard : 0.1pH
	Expanded : 0.01pH
Repeatability	±0.05pH
Temperature compensation	0°-50°C
Readout	LCD
Calibration	1-point auto (Zero)
	Manual 2-point

Temperature

Principle	Thermistor
Range	0°-50°C
Resolution	Standard : 1°C
	Expanded : 0.1°C
Repeatability	±0.3°C
Temperature compensation	—
Readout	LCD
Calibration	—

DO

Principle	Membrane galvanic cell
Range	0-19.9mg/*l*
Resolution	Standard : 0.1mg/*l*
	Expanded : 0.01mg/*l*
Repeatability	±0.1mg/*l*
Temperature compensation	0°-40°C
Readout	LCD
Calibration	1-point auto (Span)
	Manual 2-point

Conductivity

Principle	4-electrode
Range	0-100ms/cm
Resolution	Standard: 0-1mS/cm : 0.01mS/cm 0-10mS/cm : 0.1mS/cm 10-100mS/cm : 1mS/cm Expanded: 0-1mS/cm : 0.01mS/cm 0-10mS/cm : 0.1mS/cm 10-100mS/cm : 1mS/cm
Repeatability	±1%/F.S. within each measurement range
Temperature compensation	0°-50°C
Readout	LCD
Calibration	1-point auto (Span) Manual 2-point

Turbidity

Principle	Scattered/Transmitted light
Range	0-800 NTU
Resolution	Standard : 10 NTU Expanded : 1 NTU
Repeatability	±3%/F.S.
Temperature compensation	—
Readout	LCD
Calibration	1-point auto (Zero) Manual 2-point

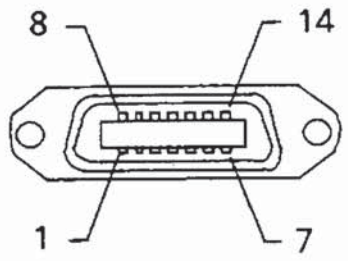
Salinity

Principle	Conversion based on conductivity
Range	0-4%
Resolution	Standard : 0.1% Expanded : 0.01%
Repeatability	±0.1%
Temperature compensation	0°-30°C
Readout	LCD
Calibration	—

Common specification

Data storage	Max. 20 samples
Printer output	Centronics specs.
Power	Battery 9V, with auto power-off function
Operating temperature	0° - 45°C
Weight	Main unit: Approx. 400g Probe, with 2-m cable: Approx. 800g

- Output connector pin layout



Pin No.	Name	Pin No.	Name
1	STB	8	DB_6
2	DB_0	9	DB_7
3	DB_1	10	Not used
4	DB_2	11	BUSY
5	DB_3	12	Not used
6	DB_4	13	Not used
7	DB_5	14	GND

This equipment is in conformity with the following directive (s) and standard (s);

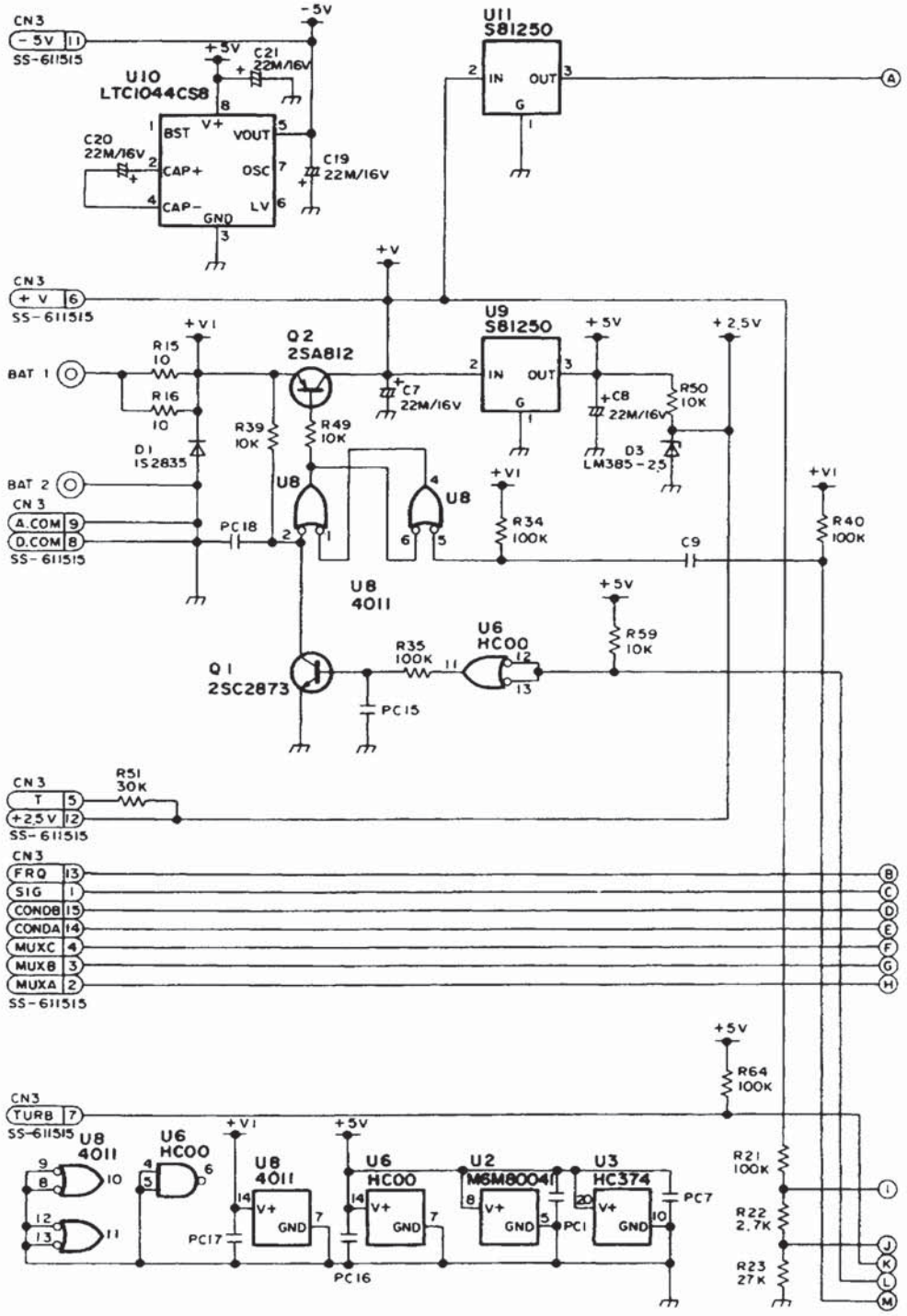
Directive (s)	the EMC Directive 89/336/EEC as amended by 91/263/EEC, 92/31/EEC and 93/68/EEC, in accordance with the Article 10 (1) of the Directive
Standard (s)	EN55011:1991 Class B Group 1 and EN50082-1:1992

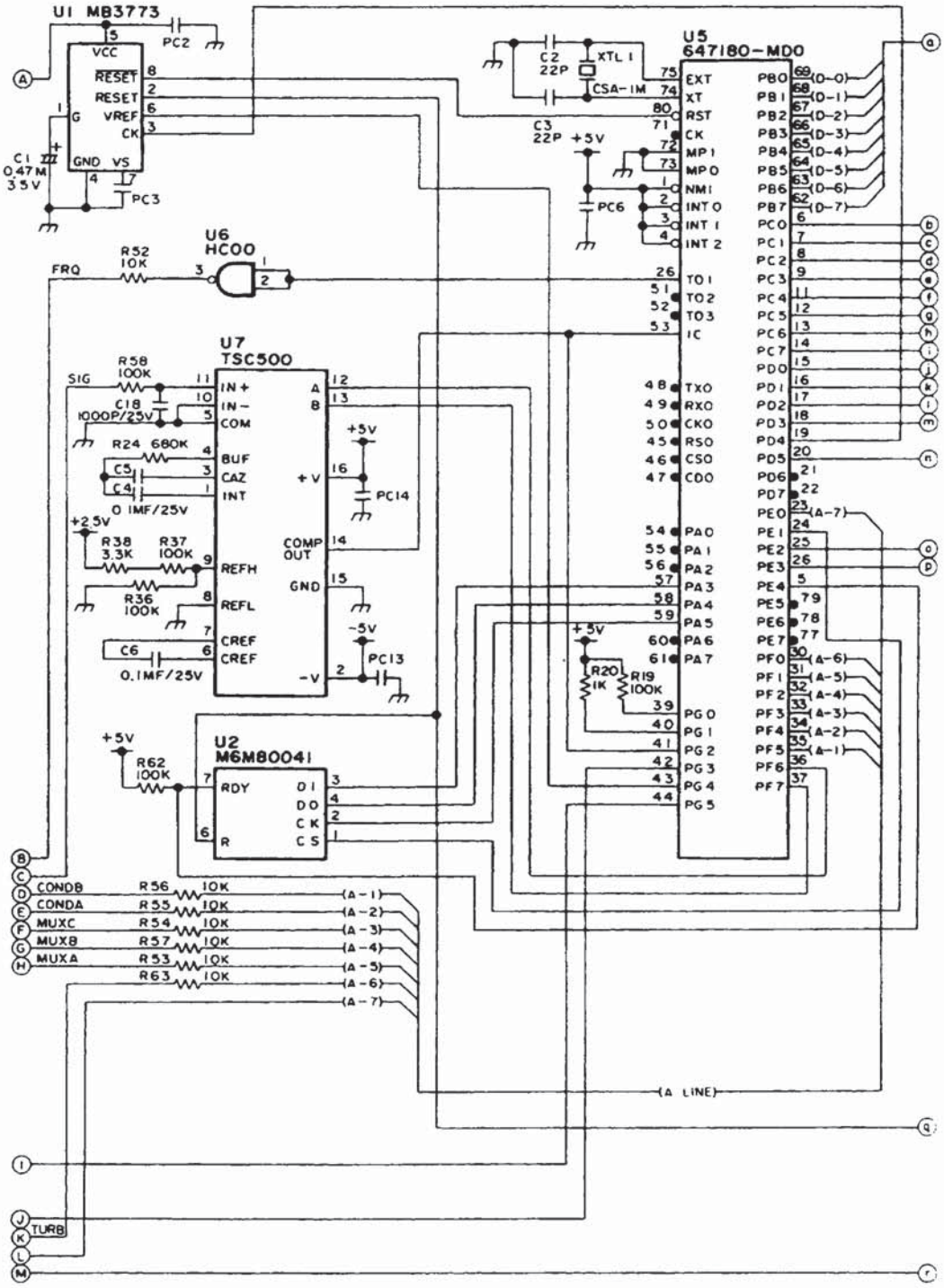
Parts List

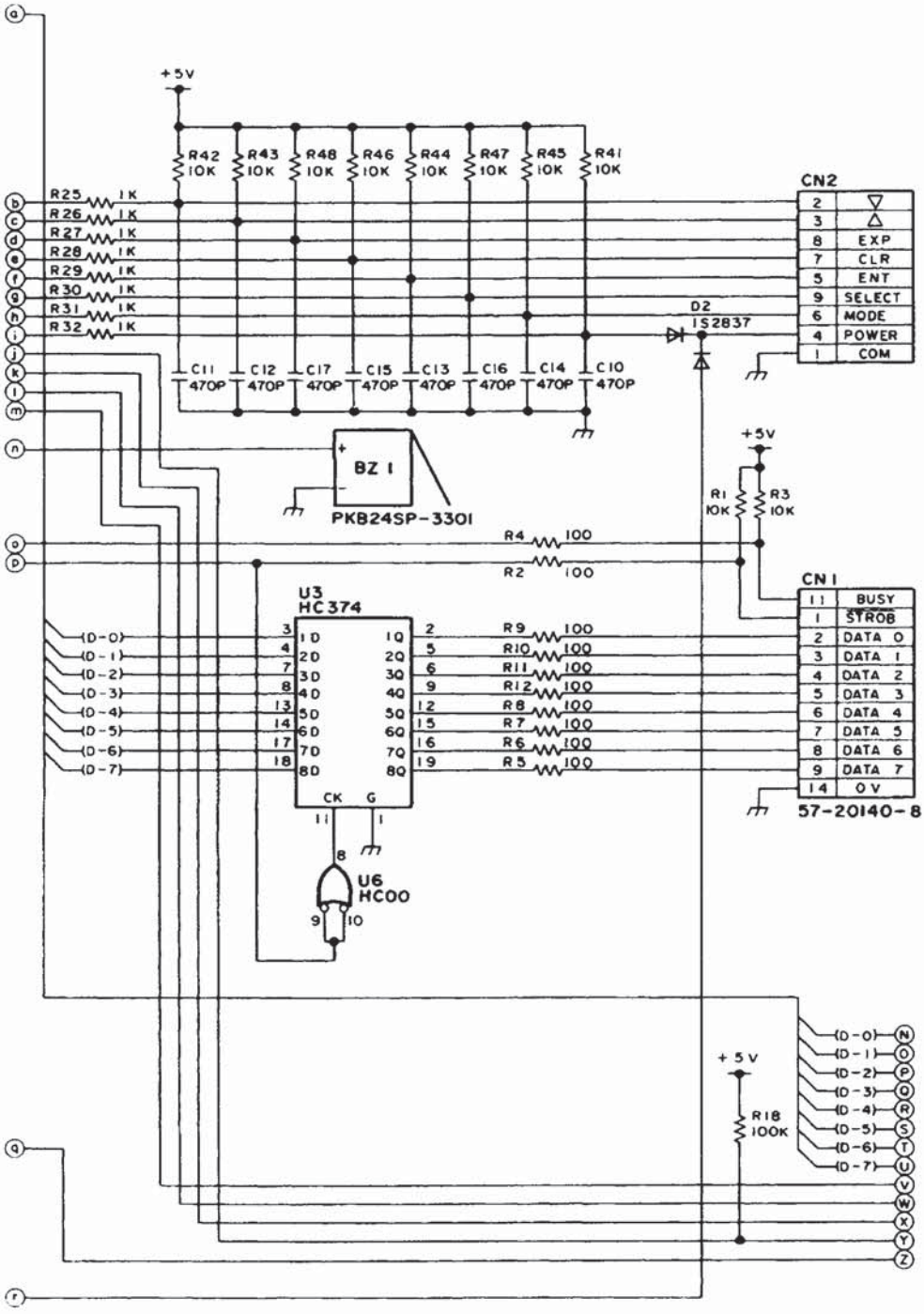
The following expendable parts are available for the U-10 Water Checker.

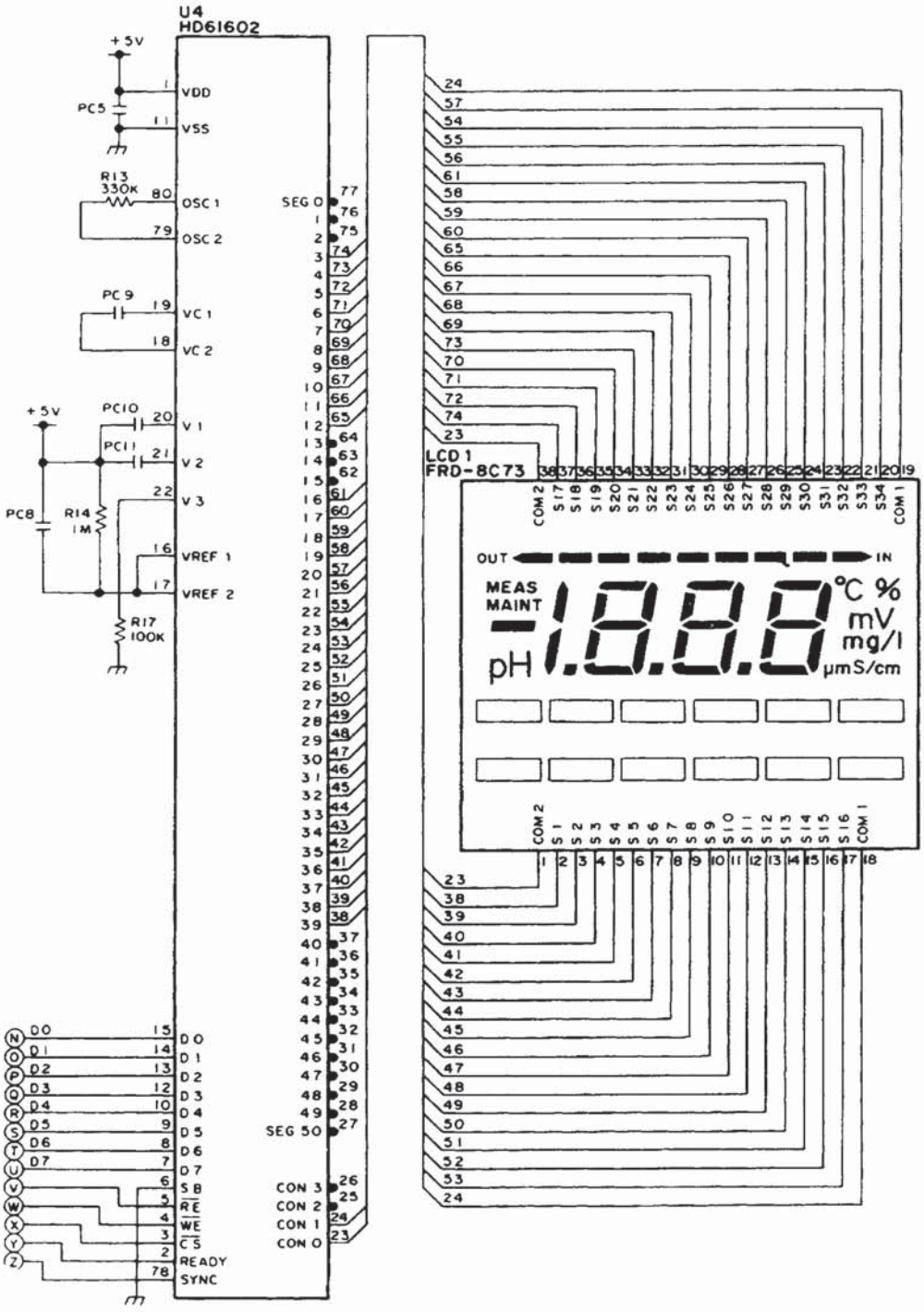
Part name	Model No.	P / N
Probe		9037-0047-00
pH sensor	#7112	9037-0048-00
DO sensor	#7542	9037-0049-00
pH reference sensor		9037-0050-00
Liquid junction (1 pair)	#7210	9037-0051-00
Reference solution	#330	9037-0052-00
pH standard solution pH2	100-2	9003-0015-00
pH standard solution pH4	100-4	9003-0016-00
pH standard solution pH7	100-7	9003-0017-00
pH standard solution pH9	100-9	9003-0018-00
Calibration beaker		9037-0053-00

Circuit Diagram

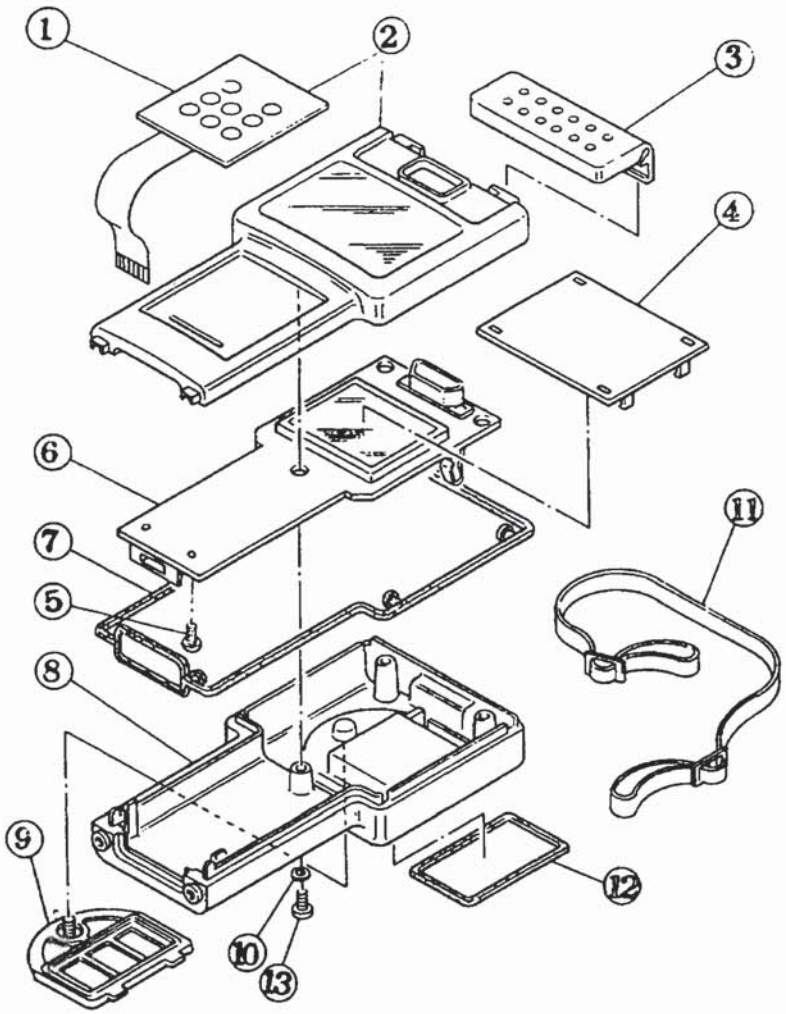



U1 MB3773
U5 647180-MD0
U6 HC00
U7 TSC500
U2 M6M80041
XTL 1
CSA-1M
C2 22P
C3 22P
PC6
PC2
PC3
C1 0.47M 35V
R52 10K
FRQ
R58 100K
SIG
C18 1000P/25V
R24 680K
C5
C4
0.1MF/25V
+2.5V
R38 3.3K
R37 100K
R36 100K
C6
0.1MF/25V
PC14
PC13
+5V
-5V
R62 100K
R20 1K
R19 100K
CONDB
CONDA
MUXC
MUXB
MUXA
R56 10K
R55 10K
R54 10K
R57 10K
R53 10K
R63 10K
TURB
A LINE


+5V
R42 10K
R43 10K
R48 10K
R46 10K
R44 10K
R47 10K
R45 10K
R41 10K
R25 1K
R26 1K
R27 1K
R28 1K
R29 1K
R30 1K
R31 1K
R32 1K
C11 470P
C12 470P
C17 470P
C15 470P
C13 470P
C16 470P
C14 470P
C10 470P
CN2
2
3
8 EXP
7 CLR
5 ENT
9 SELECT
6 MODE
4 POWER
1 COM
D2
1S2837
BZ 1
PKB24SP-3301
+5V
R1 10K
R3 10K
R4 100
R2 100
CN1
11 BUSY
1 STROB
2 DATA 0
3 DATA 1
4 DATA 2
5 DATA 3
6 DATA 4
7 DATA 5
8 DATA 6
9 DATA 7
14 0V
57-20140-8
U3
HC374
CK G
R9 100
R10 100
R11 100
R12 100
R8 100
R7 100
R6 100
R5 100
U6
HC00
+5V
R18 100K
(D-0)
(D-1)
(D-2)
(D-3)
(D-4)
(D-5)
(D-6)
(D-7)

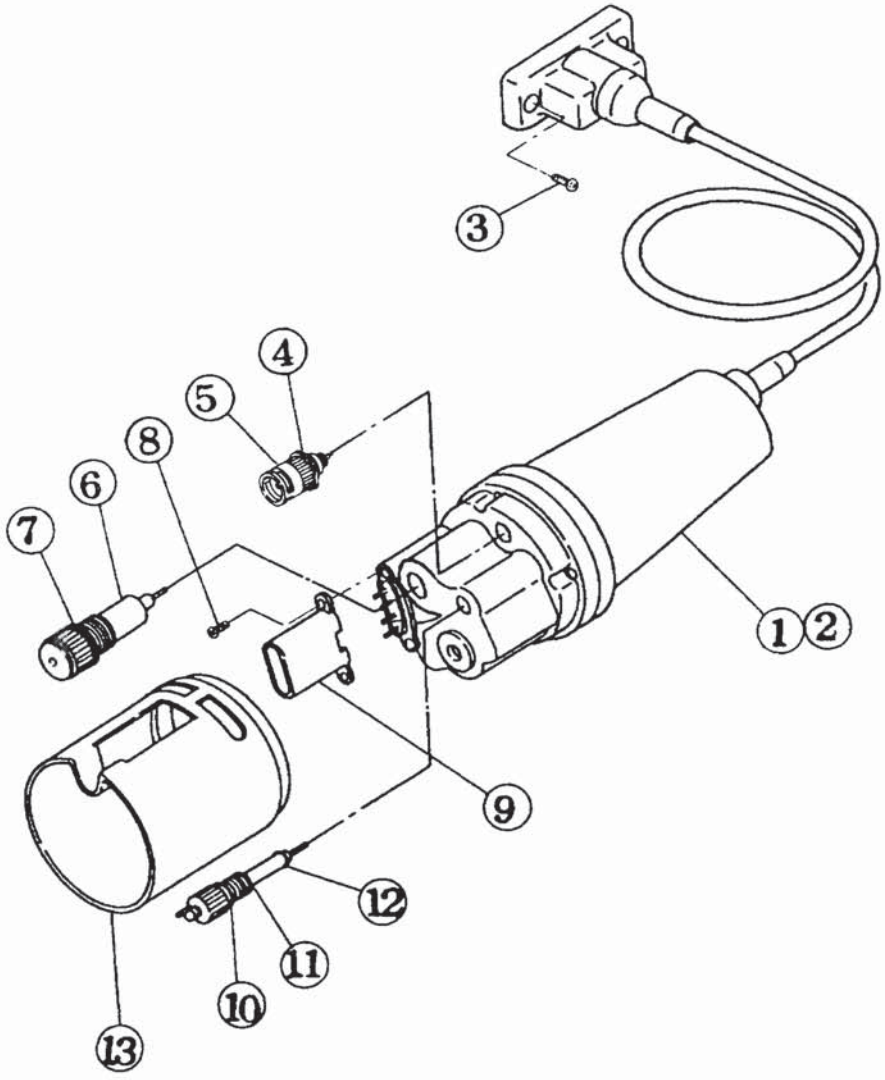

U4
HD61602
+5V
PC5
VDD
VSS
R13
330K
OSC 1
OSC 2
PC9
VC 1
VC 2
PC10
V 1
PC11
V 2
V 3
PC8
R14
1M
VREF 1
VREF 2
R17
100K
D0
D1
D2
D3
D4
D5
D6
D7
SB
RE
WE
CS
READY
SYNC
SEG 0
SEG 50
CON 3
CON 2
CON 1
CON 0
LCD 1
FRD-8C73
COM 2
COM 1
OUT
IN
MEAS
MAINT
pH
°C %
mV
mg/l
µmS/cm

Exploded Views—main unit



NO.	PARTS NO.	PARTS NAME	DESCRIPTION
1	H357911-01	SHEET SWITCH	WATER CHECKER U-10
2	U800842300	CASE ASSY, TOP	U-10 including ①
3	H357944-01	PRT COVER	U-10 METER
4	H542233-01	WINDOW, LCD	U-10 H357887-01
5	F020527500	TAPPING SCREWS	M3X6 (S-ZN3)
6	U800842400	PCB ASSY	U-10
7	H357945-01	CASE PACKING	U-10 METER
8	U800842500	CASE ASSY, BOT	U-10
9	U800842600	COVER ASSY, BAT	U-10
10	H543958-01	SEAR WASHER	U-10 METER
11	H544105-01	METER STRAP	U-10 20×1300 T=1.8
12	H542137-01	BATTERY PACKING	U-10 METER
13	F020911500	SCREW, PANHEAD	JISB1111 M3×6 (S-ZN3)

Exploded Views—prove

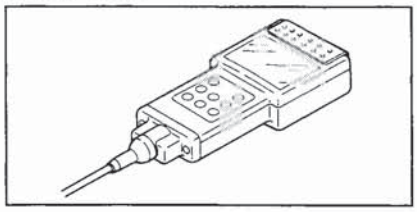


NO.	PARTS NO.	PARTS NAME	DESCRIPTION
3	F020911500	SCREW, PANHEAD	JISB1111 M3×6 (S-ZN3)
4	F020518700	O-RING	NOK S 11.2 (SI)
5	9037004900	DO SENSOR	
6	9037005000	REFERENCE	
7	F020246900	O-RING, S18	NOK S18 FPM
8	F020009500	SCREW, PANHEAD	M3-6L SUS304
9	H542141-01	COND GUARD	
10	F020058000	O-RING, P9	B2401 P9 FPM
11	9037004800	PH TIP	
12	F020058100	O-RING, P5	B2401 P5 FPM
13	H358290-01	PROTECTING TUBE	

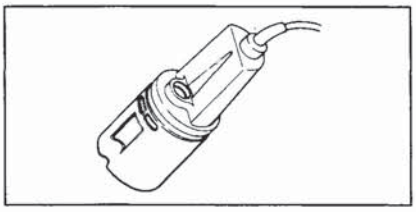
Unpacking the U-10

The following items are included with your U-10 Water Quality Checker.
When you unpack the probe and main unit, confirm that all the other accessories are included as well.

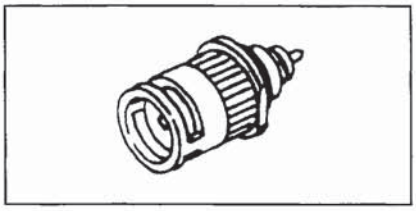
- Main unit



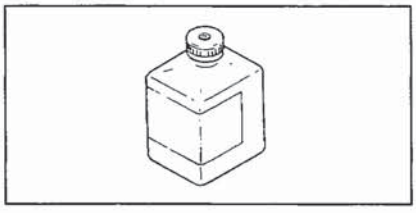
- Probe



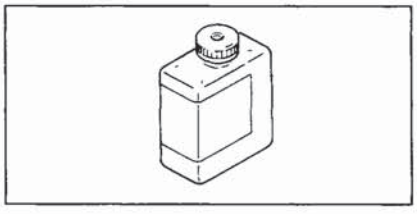
- DO sensor



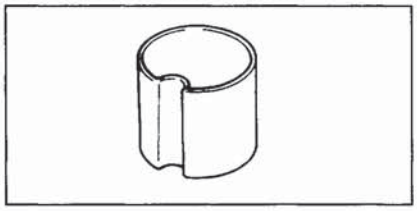
- Standard solution (pH 4 standard solution, 100-4) 500 m*l* bottle



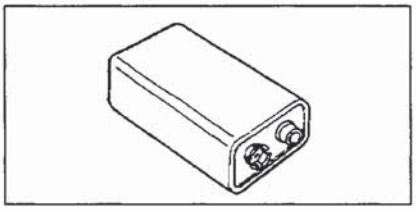
- Reference solution 250m*l* bottle



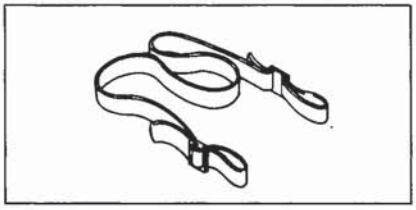
- Calibration breaker



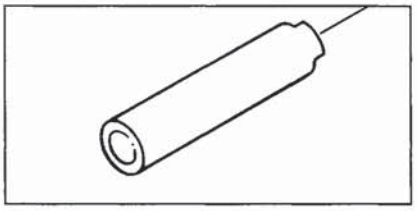
- 9V battery (6F22)



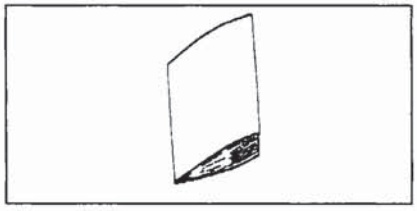
- Carrying strap for main unit



- DO sensor tool



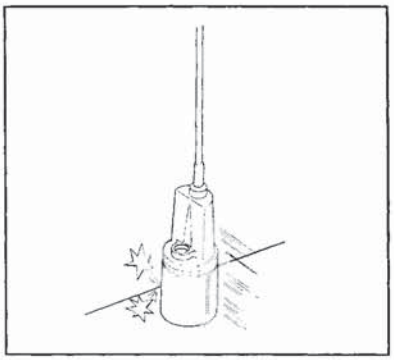
- This Instruction Manual



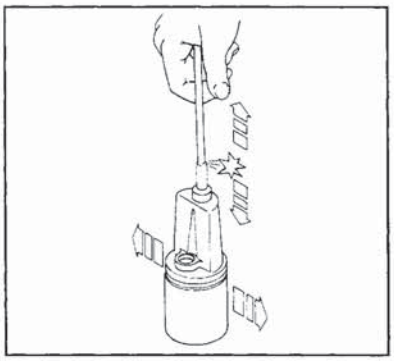
- Carrying Case

Precautions when using the U-10

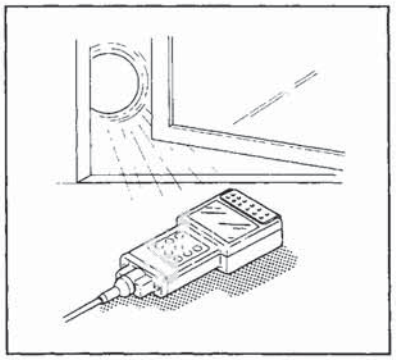
The U-10 Water Quality Checker is carefully designed for trouble-free operation. However, it is a sophisticated electronic instrument, and it can be damaged if used carelessly. Please read the following precautions and observe them when using your U-10 Water Checker.



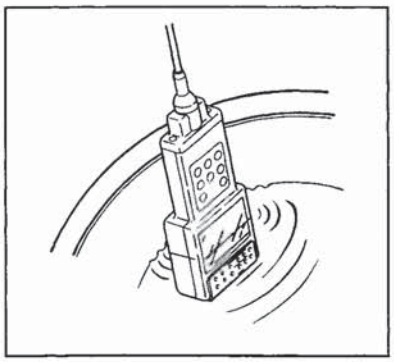
- Do not swing or jerk the probe by its cable.
- Do not subject the cable connector to stress by pulling or stretching it.



- Do not drop either the U-10 probe or main unit. Never subject either component to sudden impact.

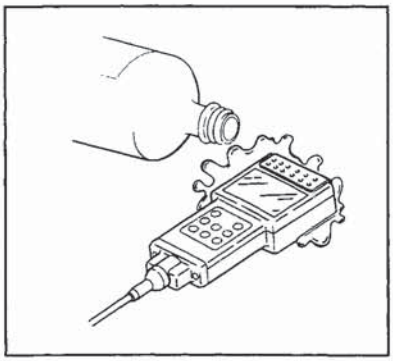


- Do not store the U-10 where it may be exposed to prolonged direct sunlight. Never leave the U-10 inside a vehicle with the windows closed.



- Never immerse the main unit directly in water.

  The main unit is water-resistant and may be safely used in the rain; however, it is not of waterproof construction. Immersing the main unit in water or any other liquid can damage the internal electronic circuits



- Never allow any organic solvent to come in contact with either the probe or the main unit. This includes such organic solvents as methylethyl ketone (MEK) and acetone.

  (The probe is made of polyphenylene ether (PPE); the main unit case is acrylic resin.)

Index

T

U

W

Z

HORIBA Worldwide Marketing Locations

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ATTACHMENT D

ANALYTICAL STANDARD OPERATING PROCEDURES AND ALS ENVIRONMENTAL/
FORT COLLINS ELAP CERTIFICATION

Scope of Accreditation
For
ALS Environmental

225 Commerce Drive
Fort Collins, CO 80524
Robert P. DiRienzo
970-490-1511

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **ALS Environmental** to perform the following tests:

Accreditation granted through: **June 1, 2016**

Testing – Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
Ion Chromatography	EPA 300.0 / EPA 9056A	Bromide
Ion Chromatography	EPA 300.0 / EPA 9056A	Chloride
Ion Chromatography	EPA 300.0 / EPA 9056A	Fluoride
Ion Chromatography	EPA 300.0 / EPA 9056A	Nitrate as N
Ion Chromatography	EPA 300.0 / EPA 9056A	Nitrite as N
Ion Chromatography	EPA 300.0 / EPA 9056A	Orthophosphate as P
Ion Chromatography	EPA 300.0 / EPA 9056A	Sulfate
Analyzer	EPA 415.1 / EPA 9060	TOC
ICP	EPA 6010B	Aluminum
ICP	EPA 6010B	Antimony
ICP	EPA 6010B	Arsenic
ICP	EPA 6010B	Barium
ICP	EPA 6010B	Beryllium
ICP	EPA 6010B	Bismuth
ICP	EPA 6010B	Boron
ICP	EPA 6010B	Cadmium
ICP	EPA 6010B	Calcium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP	EPA 6010B	Chromium
ICP	EPA 6010B	Cobalt
ICP	EPA 6010B	Copper
ICP	EPA 6010B	Iron
ICP	EPA 6010B	Lead
ICP	EPA 6010B	Lithium
ICP	EPA 6010B	Magnesium
ICP	EPA 6010B	Manganese
ICP	EPA 6010B	Molybdenum
ICP	EPA 6010B	Nickel
ICP	EPA 6010B	Phosphorus
ICP	EPA 6010B	Potassium
ICP	EPA 6010B	Selenium
ICP	EPA 6010B	Silicon
ICP	EPA 6010B	Silicon as SiO_2
ICP	EPA 6010B	Silver
ICP	EPA 6010B	Sodium
ICP	EPA 6010B	Strontium
ICP	EPA 6010B	Sulfur
ICP	EPA 6010B	Thallium
ICP	EPA 6010B	Tin
ICP	EPA 6010B	Titanium
ICP	EPA 6010B	Uranium
ICP	EPA 6010B	Vanadium
ICP	EPA 6010B	Zinc
ICP	EPA 6010B	Zirconium
ICP / MS	EPA 6020A	Aluminum
ICP / MS	EPA 6020A	Antimony
ICP / MS	EPA 6020A	Arsenic
ICP / MS	EPA 6020A	Barium
ICP / MS	EPA 6020A	Beryllium
ICP / MS	EPA 6020A	Cadmium
ICP / MS	EPA 6020A	Calcium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP / MS	EPA 6020A	Cerium
ICP / MS	EPA 6020A	Chromium
ICP / MS	EPA 6020A	Cobalt
ICP / MS	EPA 6020A	Copper
ICP / MS	EPA 6020A	Iron
ICP / MS	EPA 6020A	Lanthanum
ICP / MS	EPA 6020A	Lead
ICP / MS	EPA 6020A	Lithium
ICP / MS	EPA 6020A	Magnesium
ICP / MS	EPA 6020A	Manganese
ICP / MS	EPA 6020A	Molybdenum
ICP / MS	EPA 6020A	Neodymium
ICP / MS	EPA 6020A	Nickel
ICP / MS	EPA 6020A	Potassium
ICP / MS	EPA 6020A	Praseodymium
ICP / MS	EPA 6020A	Selenium
ICP / MS	EPA 6020A	Silver
ICP / MS	EPA 6020A	Sodium
ICP / MS	EPA 6020A	Strontium
ICP / MS	EPA 6020A	Thallium
ICP / MS	EPA 6020A	Thorium
ICP / MS	EPA 6020A	Tin
ICP / MS	EPA 6020A	Titanium
ICP / MS	EPA 6020A	U-235
ICP / MS	EPA 6020A	U-238
ICP / MS	EPA 6020A	Uranium
ICP / MS	EPA 6020A	Vanadium
ICP / MS	EPA 6020A	Yttrium
ICP / MS	EPA 6020A	Zinc
Colorimetric	EPA 335.1 SM 4500-CN C,E	Cyanide (Total and Amenable)
UV-Vis	EPA 9010C	Cyanide (Total and Amenable)
UV-Vis	EPA 7196A	Hexavalent Chromium (Cr^{VI})
Titrimetric	EPA 9013 / EPA 9014 EPA 335.2	Cyanide

Non-Potable Water		
Technology	**Method**	**Analyte**
Gravimetric	EPA 160.1 / SM 2540 C	Total Dissolved Solids
Gravimetric	EPA 160.2 / SM 2540 D	Total Suspended Solids
Gravimetric	EPA 1664A / EPA 9071B	HEM/Oil And Grease
ISE	SM 2510B EPA 120.1 / EPA 9050A	Conductivity
Titration	SM 2320B EPA 310.1	Alkalinity
UV/VIS	EPA 353.2	Nitrogen, Nitrate-Nitrite
Colorimetric	EPA 354.1 SM 4500-NO2 B	Nitrogen, Nitrite
Colorimetric	EPA 365.2 SM 4500-P E	Phosphorous, Total And Ortho
Titrimetric	EPA 376.1 SM 4500-S2 F	Sulfide
Gravimetric	EPA 9095A	Paint Filter Liquids Test
CVAA	EPA 245.1 / EPA 7470	Mercury
ISE	EPA 150.1 / EPA 9040C SM 4500-H+ B	pH
Flash Point	EPA 1010A	Ignitability
GC / ECD	EPA 8081A	4,4'-DDD
GC / ECD	EPA 8081A	4,4'-DDE
GC / ECD	EPA 8081A	4,4'-DDT
GC / ECD	EPA 8081A	Aldrin
GC / ECD	EPA 8081A	Alpha-BHC
GC / ECD	EPA 8081A	Alpha-Chlordane
GC / ECD	EPA 8081A	Beta-BHC
GC / ECD	EPA 8081A	Chlordane
GC / ECD	EPA 8081A	Delta-BHC
GC / ECD	EPA 8081A	Dieldrin
GC / ECD	EPA 8081A	Endosulfan I
GC / ECD	EPA 8081A	Endosulfan II
GC / ECD	EPA 8081A	Endosulfan Sulfate
GC / ECD	EPA 8081A	Endrin
GC / ECD	EPA 8081A	Endrin Aldehyde
GC / ECD	EPA 8081A	Endrin Ketone
GC / ECD	EPA 8081A	Gamma-BHC (Lindane)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / ECD	EPA 8081A	Gamma-Chlordane
GC / ECD	EPA 8081A	Heptachlor
GC / ECD	EPA 8081A	Heptachlor Epoxide
GC / ECD	EPA 8081A	Methoxychlor
GC / ECD	EPA 8081A	Toxaphene
GC / ECD	EPA 8082	Aroclor-1016
GC / ECD	EPA 8082	Aroclor-1221
GC / ECD	EPA 8082	Aroclor-1232
GC / ECD	EPA 8082	Aroclor-1242
GC / ECD	EPA 8082	Aroclor-1248
GC / ECD	EPA 8082	Aroclor-1254
GC / ECD	EPA 8082	Aroclor-1260
GC / ECD	EPA 8082	Aroclor-1262
GC / ECD	EPA 8082	Aroclor-1268
GC / ECD	EPA 8151A	2,4,5-T
GC / ECD	EPA 8151A	2,4-D
GC / ECD	EPA 8151A	2,4-DB
GC / ECD	EPA 8151A	Dalapon
GC / ECD	EPA 8151A	Dicamba
GC / ECD	EPA 8151A	Dichloroprop
GC / ECD	EPA 8151A	Dinoseb
GC / ECD	EPA 8151A	MCPA
GC / ECD	EPA 8151A	MCPP
GC / ECD	EPA 8151A	Silvex
GC / FPD	EPA 8141A	Chlorpyrifos
GC / FPD	EPA 8141A	Coumaphos
GC / FPD	EPA 8141A	Demeton O + S
GC / FPD	EPA 8141A	Diazinon
GC / FPD	EPA 8141A	Dichlorvos
GC / FPD	EPA 8141A	Disulfoton
GC / FPD	EPA 8141A	Ethoprop
GC / FPD	EPA 8141A	Fensulfothion
GC / FPD	EPA 8141A	Fenthion

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / FPD	EPA 8141A	Malathion
GC / FPD	EPA 8141A	Merphos A + B
GC / FPD	EPA 8141A	Methyl Azinphos
GC / FPD	EPA 8141A	Methyl Parathion
GC / FPD	EPA 8141A	Mevinphos
GC / FPD	EPA 8141A	Naled
GC / FPD	EPA 8141A	Phorate
GC / FPD	EPA 8141A	Ronnel
GC / FPD	EPA 8141A	Sulprofos
GC / FPD	EPA 8141A	Tetrachlorvinphos
GC / FPD	EPA 8141A	Tokuthion
GC / FPD	EPA 8141A	Trichloronate
GC / FPD	EPA 8141A	Triphenylphosphate
GC / FID	EPA 8015B	GRO
GC / FID	EPA 8015B	DRO
GC / FID	EPA 8015B	Ethylene Glycol
GC / FID	EPA 8015B	Propylene Glycol
GC / FID	EPA 8015B	Diethylene Glycol
GC / FID	EPA 8015B	Triethylene Glycol
GC / FID	EPA 8015B	Tetraethylene Glycol
GC / MS	EPA 8260C	Chloroacetonitrile
GC / MS	EPA 8260C	1-chlorobutane
GC / MS	EPA 8260C	Methyl acrylate
GC / MS	EPA 8260C	Pentafluorobenzene
GC / MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC / MS	EPA 8260C	1,1,1-Trichloroethane
GC / MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC / MS	EPA 8260C	1,1,2-Trichloro-1,2,2-Trifluoroethane
GC / MS	EPA 8260C	1,1,2-Trichloroethane
GC / MS	EPA 8260C	1,1-Dichloroethane
GC / MS	EPA 8260C	1,1-Dichloroethene
GC / MS	EPA 8260C	1,1-Dichloropropene
GC / MS	EPA 8260C	1,2,3-Trichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / MS	EPA 8260C	1,2,3-Trichloropropane
GC / MS	EPA 8260C	1,2,4-Trichlorobenzene
GC / MS	EPA 8260C	1,2,4-Trimethylbenzene
GC / MS	EPA 8260C	1,2-Dibromo-3-Chloropropane
GC / MS	EPA 8260C	1,2-Dibromoethane
GC / MS	EPA 8260C	1,2-Dichlorobenzene
GC / MS	EPA 8260C	1,2-Dichloroethane
GC / MS	EPA 8260C	1,2-Dichloroethene (Total)
GC / MS	EPA 8260C	1,2-Dichloropropane
GC / MS	EPA 8260C	1,3,5-Trimethylbenzene
GC / MS	EPA 8260C	1,3-Dichlorobenzene
GC / MS	EPA 8260C	1,3-Dichloropropane
GC / MS	EPA 8260C	1,4-Dichlorobenzene
GC / MS	EPA 8260C	1-Chlorohexane
GC / MS	EPA 8260C	2,2-Dichloropropane
GC / MS	EPA 8260C	2-Butanone
GC / MS	EPA 8260C	2-Chlorotoluene
GC / MS	EPA 8260C	2-Hexanone
GC / MS	EPA 8260C	4-Chlorotoluene
GC / MS	EPA 8260C	4-Methyl-2-Pentanone
GC / MS	EPA 8260C	Acetone
GC / MS	EPA 8260C	Benzene
GC / MS	EPA 8260C	Bromobenzene
GC / MS	EPA 8260C	Bromochloromethane
GC / MS	EPA 8260C	Bromodichloromethane
GC / MS	EPA 8260C	Bromoform
GC / MS	EPA 8260C	Bromomethane
GC / MS	EPA 8260C	Carbon Disulfide
GC / MS	EPA 8260C	Carbon Tetrachloride
GC / MS	EPA 8260C	Chlorobenzene
GC / MS	EPA 8260C	Chloroethane
GC / MS	EPA 8260C	Chloroform
GC / MS	EPA 8260C	Chloromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / MS	EPA 8260C	Cis-1,2-Dichloroethene
GC / MS	EPA 8260C	Cis-1,3-Dichloropropene
GC / MS	EPA 8260C	Dibromochloromethane
GC / MS	EPA 8260C	Dibromomethane
GC / MS	EPA 8260C	Dichlorodifluoromethane
GC / MS	EPA 8260C	Ethylbenzene
GC / MS	EPA 8260C	Hexachlorobutadiene
GC / MS	EPA 8260C	Iodomethane
GC / MS	EPA 8260C	Isopropylbenzene
GC / MS	EPA 8260C	M+P-Xylene
GC / MS	EPA 8260C	Methyl Tertiary Butyl Ether
GC / MS	EPA 8260C	Methylene Chloride
GC / MS	EPA 8260C	Naphthalene
GC / MS	EPA 8260C	N-Butylbenzene
GC / MS	EPA 8260C	N-Propylbenzene
GC / MS	EPA 8260C	O-Xylene
GC / MS	EPA 8260C	P-Isopropyltoluene
GC / MS	EPA 8260C	Sec-Butylbenzene
GC / MS	EPA 8260C	Styrene
GC / MS	EPA 8260C	Tert-Butylbenzene
GC / MS	EPA 8260C	Tetrachloroethene
GC / MS	EPA 8260C	Toluene
GC / MS	EPA 8260C	Total Xylenes
GC / MS	EPA 8260C	Trans-1,2-Dichloroethene
GC / MS	EPA 8260C	Trans-1,3-Dichloropropene
GC / MS	EPA 8260C	Trichloroethene
GC / MS	EPA 8260C	Trichlorofluoromethane
GC / MS	EPA 8260C	Vinyl Acetate
GC / MS	EPA 8260C	Vinyl Chloride
GC / MS	EPA 8270D	1,2,4-Trichlorobenzene
GC / MS	EPA 8270D	1,2-Dichlorobenzene
GC / MS	EPA 8270D	1,3-Dichlorobenzene
GC / MS	EPA 8270D	1,4-Dichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / MS	EPA 8270D	1,4-Dioxane
GC / MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC / MS	EPA 8270D	2,4,5-Trichlorophenol
GC / MS	EPA 8270D	2,4,6-Trichlorophenol
GC / MS	EPA 8270D	2,4-Dichlorophenol
GC / MS	EPA 8270D	2,4-Dimethylphenol
GC / MS	EPA 8270D	2,4-Dinitrophenol
GC / MS	EPA 8270D	2,4-Dinitrotoluene
GC / MS	EPA 8270D	2,6-Dinitrotoluene
GC / MS	EPA 8270D	2-Chloronaphthalene
GC / MS	EPA 8270D	2-Chlorophenol
GC / MS	EPA 8270D	2-Methylnaphthalene
GC / MS	EPA 8270D	2-Methylphenol
GC / MS	EPA 8270D	2-Nitroaniline
GC / MS	EPA 8270D	2-Nitrophenol
GC / MS	EPA 8270D	3,3'-Dichlorobenzidine
GC / MS	EPA 8270D	3+4-Methylphenol
GC / MS	EPA 8270D	4,6-Dinitro-2-Methylphenol
GC / MS	EPA 8270D	4-Aminobiphenyl
GC / MS	EPA 8270D	4-Bromophenyl Phenyl Ether
GC / MS	EPA 8270D	4-Chloro-3-Methylphenol
GC / MS	EPA 8270D	4-Chloroaniline
GC / MS	EPA 8270D	4-Chlorophenyl Phenyl Ether
GC / MS	EPA 8270D	4-Nitroaniline
GC / MS	EPA 8270D	4-Nitrophenol
GC / MS	EPA 8270D	Acenaphthene
GC / MS	EPA 8270D	Acenaphthylene
GC / MS	EPA 8270D	Aniline
GC / MS	EPA 8270D	Anthracene
GC / MS	EPA 8270D	Azobenzene
GC / MS	EPA 8270D	Benzo(A)Anthracene
GC / MS	EPA 8270D	Benzo(A)Pyrene
GC / MS	EPA 8270D	Benzo(B)Fluoranthene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC / MS	EPA 8270D	Benzo(G,H,I)Perylene
GC / MS	EPA 8270D	Benzo(K)Fluoranthene
GC / MS	EPA 8270D	Benzoic Acid
GC / MS	EPA 8270D	Benzyl Alcohol
GC / MS	EPA 8270D	Bis(2-Chloroethoxy)Methane
GC / MS	EPA 8270D	Bis(2-Chloroisopropyl)Ether
GC / MS	EPA 8270D	Bis(2-Ethylhexyl)Phthalate
GC / MS	EPA 8270D	Butyl Benzyl Phthalate
GC / MS	EPA 8270D	Carbazole
GC / MS	EPA 8270D	Chrysene
GC / MS	EPA 8270D	Dibenzo(A,H)Anthracene
GC / MS	EPA 8270D	Dibenzofuran
GC / MS	EPA 8270D	Dimethyl Phthalate
GC / MS	EPA 8270D	Di-N-Octyl Phthalate
GC / MS	EPA 8270D	Fluoranthene
GC / MS	EPA 8270D	Fluorene
GC / MS	EPA 8270D	Hexachlorobenzene
GC / MS	EPA 8270D	Hexachlorobutadiene
GC / MS	EPA 8270D	Hexachlorocyclopentadiene
GC / MS	EPA 8270D	Hexachloroethane
GC / MS	EPA 8270D	Indeno(1,2,3-Cd)Pyrene
GC / MS	EPA 8270D	Isophorone
GC / MS	EPA 8270D	Naphthalene
GC / MS	EPA 8270D	Nitrobenzene
GC / MS	EPA 8270D	N-Nitrosodimethylamine
GC / MS	EPA 8270D	N-Nitroso-Di-N-Propylamine
GC / MS	EPA 8270D	N-Nitrosodiphenylamine
GC / MS	EPA 8270D	Pentachlorophenol
GC / MS	EPA 8270D	Phenanthrene
GC / MS	EPA 8270D	Phenol
GC / MS	EPA 8270D	Pyrene
GC / MS	EPA 8270D	Pyridine
Gas Proportional Counting	EPA 900 / EPA 9310	Gross Alpha

Non-Potable Water		
Technology	**Method**	**Analyte**
Gas Proportional Counting	EPA 900 / EPA 9310	Gross Beta
Gas Proportional Counting	EPA 904 / EPA 9320	Ra228
Gas Proportional Counting	HASL 300 Sr01 HASL 300 Sr02	Strontium 90
Gas Proportional Counting	ASTM D5811	Strontium 90
Gas Proportional Counting	EPA 902.0 ALS SOP 753	Iodine-129
Liquid Scintillation Counting	EPA 906.0 SM 7500 3H	Tritium
Liquid Scintillation Counting	EPA C-01	Carbon-14
Liquid Scintillation Counting	DOE RP550 DOE RS551	Technicium-99
Liquid Scintillation Counting	Horwitz, Chiariza, Dietz 1992	Lead-210
Liquid Scintillation Counting	ALS SOP 704	Pu241, Pm147
Liquid Scintillation Counting	ALS SOP 774	Nickle-63
Emanation	EPA 903.1 SM 7500-Ra C	Radium 226
Gas Proportional Counting	EPA 903.0 / EPA 9315	Total Radium
Liquid Scintillation Counting	SM 7500-Rn B ASTM D 5072	Rn-222
Gas Proportional Counting	EPA 903.0 / EPA 9315	Radium-226
Alpha-Spec	HASL 300 U02 ASTM D 3972	Ac-227
Alpha-Spec	HASL 300 U02 ASTM D 3972	Am-241
Alpha-Spec	HASL 300 U02 ASTM D 3972	Am-242/243
Alpha-Spec	HASL 300 U02 ASTM D 3972	Am-243
Alpha-Spec	HASL 300 U02 ASTM D 3972	Cm-242
Alpha-Spec	HASL 300 U02 ASTM D 3972	Cm-243/244
Alpha-Spec	HASL 300 U02 ASTM D 3972	Cm-244
Alpha-Spec	HASL 300 U02 ASTM D 3972	Cm-245/246
Alpha-Spec	HASL 300 U02 ASTM D 3972	Np-237

Non-Potable Water		
Technology	**Method**	**Analyte**
Alpha-Spec	HASL 300 U02 ASTM D 3972	Po-210
Alpha-Spec	HASL 300 U02 ASTM D 3972	Pu-238
Alpha-Spec	HASL 300 U02 ASTM D 3972	Pu-239
Alpha-Spec	HASL 300 U02 ASTM D 3972	Pu-239/240
Alpha-Spec	HASL 300 U02 ASTM D 3972	Pu-242
Alpha-Spec	ALS-SOP 701	Ra-226
Alpha-Spec	HASL 300 U02 ASTM D 3972	Th-227
Alpha-Spec	HASL 300 U02 ASTM D 3972	Th-228
Alpha-Spec	HASL 300 U02 ASTM D 3972	Th-230
Alpha-Spec	HASL 300 U02 ASTM D 3972	Th-232
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-232
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-233/234
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-234
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-235
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-235/236
Alpha-Spec	HASL 300 U02 ASTM D 3972	U-238
Alpha-Spec	HASL 300 U02 ASTM D 3972	Uranium, Total
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ac-227
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ac-228
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ag-108m

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ag-110m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Al-26
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Am-241
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Am-243
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-72
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-73
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-74
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ba-133
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ba-140
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Be-7
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-211
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-212
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-214
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-76

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-77
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-82
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cd-109
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-139
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-141
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-144
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cf-249
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cf-251
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cl-39
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	CM-243
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-56
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-57
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-58
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-60

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cr-51
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-134
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-135
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-136
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-137
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-152
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-154
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-155
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Fe-59
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Gd-153
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ge-68
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hf-181
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hg-197m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hg-203

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	I-131
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ir-192
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	K-40
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Kr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	La-140
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Mn-54
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Na-22
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Na-24
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nb-94
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nb-95
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nd-147
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-236
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-237
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-239

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Os-191
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pa-231
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pa-234m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-210
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-211
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-212
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-214
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pm-144
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pm-146
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Po-209
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-223
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-224
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-226
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-228

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-83
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-86
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rh-101
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rh-106
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ru-103
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ru-106
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sb-124
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sb-125
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sc-46
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Se-75
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sn-113
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sn-126
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ta-182

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Tb-160
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-227
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-228
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-230
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-231
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-232
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-234
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Tl-208
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	U-235
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Uranium, Total
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	V-48
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Y-88
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Zn-65
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Zr-95

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Au-198
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cr-51
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Kr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Te-132
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-86
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Se-75
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cd-109
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	In-111
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/FID	RSK-175	Propane
Preparation	**Method**	**Type**
Preparation	EPA 3005A	Acid Digestion Total Recoverable or Dissolved Metals
Preparation	EPA 3010A	Acid Digestion for Total Metals
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Metals

Non-Potable Water		
Technology	**Method**	**Analyte**
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Semi-Volatiles
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Volatiles
Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Cleanup Procedure	EPA 3620B	Florisil Cleanup
Cleanup Procedure	EPA 3630C	Silica Gel Cleanup
Cleanup Procedure	EPA 3640A	Gel Permeation Cleanup
Cleanup Procedure	EPA 3660A	Sulfur Cleanup
Purge and Trap	EPA 5030C	Purge-and-Trap for Aqueous Samples

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Ion Chromatography	EPA 300.0 / EPA 9056A	Bromide
Ion Chromatography	EPA 300.0 / EPA 9056A	Chloride
Ion Chromatography	EPA 300.0 / EPA 9056A	Fluoride
Ion Chromatography	EPA 300.0 / EPA 9056A	Nitrate as N
Ion Chromatography	EPA 300.0 / EPA 9056A	Nitrite as N
Ion Chromatography	EPA 300.0 / EPA 9056A	Orthophosphate as P
Ion Chromatography	EPA 300.0 / EPA 9056A	Sulfate
ICP - AES	EPA 6010B	Aluminum
ICP - AES	EPA 6010B	Antimony
ICP - AES	EPA 6010B	Arsenic
ICP - AES	EPA 6010B	Barium
ICP - AES	EPA 6010B	Beryllium
ICP - AES	EPA 6010B	Bismuth
ICP - AES	EPA 6010B	Boron
ICP - AES	EPA 6010B	Cadmium
ICP - AES	EPA 6010B	Calcium
ICP - AES	EPA 6010B	Chromium
ICP - AES	EPA 6010B	Cobalt
ICP - AES	EPA 6010B	Copper
ICP - AES	EPA 6010B	Iron
ICP - AES	EPA 6010B	Lead

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP - AES	EPA 6010B	Lithium
ICP - AES	EPA 6010B	Magnesium
ICP - AES	EPA 6010B	Manganese
ICP - AES	EPA 6010B	Molybdenum
ICP - AES	EPA 6010B	Nickel
ICP - AES	EPA 6010B	Phosphorus
ICP - AES	EPA 6010B	Potassium
ICP - AES	EPA 6010B	Selenium
ICP - AES	EPA 6010B	Silicon
ICP - AES	EPA 6010B	Silicon as SiO_2
ICP - AES	EPA 6010B	Silver
ICP - AES	EPA 6010B	Sodium
ICP - AES	EPA 6010B	Strontium
ICP - AES	EPA 6010B	Sulfur
ICP - AES	EPA 6010B	Thallium
ICP - AES	EPA 6010B	Tin
ICP - AES	EPA 6010B	Titanium
ICP - AES	EPA 6010B	Uranium
ICP - AES	EPA 6010B	Vanadium
ICP - AES	EPA 6010B	Zinc
ICP - AES	EPA 6010B	Zirconium
ICP / MS	EPA 6020A	Aluminum
ICP / MS	EPA 6020A	Antimony
ICP / MS	EPA 6020A	Arsenic
ICP / MS	EPA 6020A	Barium
ICP / MS	EPA 6020A	Beryllium
ICP / MS	EPA 6020A	Cadmium
ICP / MS	EPA 6020A	Calcium
ICP / MS	EPA 6020A	Cerium
ICP / MS	EPA 6020A	Chromium
ICP / MS	EPA 6020A	Cobalt
ICP / MS	EPA 6020A	Copper
ICP / MS	EPA 6020A	Iron

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP / MS	EPA 6020A	Lanthanum
ICP / MS	EPA 6020A	Lead
ICP / MS	EPA 6020A	Lithium
ICP / MS	EPA 6020A	Magnesium
ICP / MS	EPA 6020A	Manganese
ICP / MS	EPA 6020A	Molybdenum
ICP / MS	EPA 6020A	Neodymium
ICP / MS	EPA 6020A	Nickel
ICP / MS	EPA 6020A	Potassium
ICP / MS	EPA 6020A	Praseodymium
ICP / MS	EPA 6020A	Selenium
ICP / MS	EPA 6020A	Silver
ICP / MS	EPA 6020A	Sodium
ICP / MS	EPA 6020A	Strontium
ICP / MS	EPA 6020A	Thallium
ICP / MS	EPA 6020A	Thorium
ICP / MS	EPA 6020A	Tin
ICP / MS	EPA 6020A	Titanium
ICP / MS	EPA 6020A	U-235
ICP / MS	EPA 6020A	U-238
ICP / MS	EPA 6020A	Uranium
ICP / MS	EPA 6020A	Vanadium
ICP / MS	EPA 6020A	Yttrium
ICP / MS	EPA 6020A	Zinc
CVAA	EPA 7471	Mercury
ISE	EPA 9045D	pH
Titrimetric	EPA 9013 / EPA 9014	Cyanide
UV-Vis	EPA 9010C	Cyanide (Total and Amenable)
UV-Vis	EPA 7196A	Hexavalent Chromium (Cr^{VI})
Gravimetric	EPA 9071B	HEM/Oil And Grease
Wet Chemistry	SW846 7.3.3.2 SW846 7.3.4.1	Reactivity
Flash Point Tester	EPA 1010A	Ignitability
GC / ECD	EPA 8081A	4,4'-DDD

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / ECD	EPA 8081A	4,4'-DDE
GC / ECD	EPA 8081A	4,4'-DDT
GC / ECD	EPA 8081A	Aldrin
GC / ECD	EPA 8081A	Alpha-BHC
GC / ECD	EPA 8081A	Alpha-Chlordane
GC / ECD	EPA 8081A	Beta-BHC
GC / ECD	EPA 8081A	Chlordane
GC / ECD	EPA 8081A	Delta-BHC
GC / ECD	EPA 8081A	Dieldrin
GC / ECD	EPA 8081A	Endosulfan I
GC / ECD	EPA 8081A	Endosulfan II
GC / ECD	EPA 8081A	Endosulfan Sulfate
GC / ECD	EPA 8081A	Endrin
GC / ECD	EPA 8081A	Endrin Aldehyde
GC / ECD	EPA 8081A	Endrin Ketone
GC / ECD	EPA 8081A	Gamma-BHC (Lindane)
GC / ECD	EPA 8081A	Gamma-Chlordane
GC / ECD	EPA 8081A	Heptachlor
GC / ECD	EPA 8081A	Heptachlor Epoxide
GC / ECD	EPA 8081A	Methoxychlor
GC / ECD	EPA 8081A	Toxaphene
GC / ECD	EPA 8082	Aroclor-1016
GC / ECD	EPA 8082	Aroclor-1221
GC / ECD	EPA 8082	Aroclor-1232
GC / ECD	EPA 8082	Aroclor-1242
GC / ECD	EPA 8082	Aroclor-1248
GC / ECD	EPA 8082	Aroclor-1254
GC / ECD	EPA 8082	Aroclor-1260
GC / ECD	EPA 8082	Aroclor-1262
GC / ECD	EPA 8082	Aroclor-1268
GC / ECD	EPA 8151A	2,4,5-T
GC / ECD	EPA 8151A	2,4-D
GC / ECD	EPA 8151A	2,4-DB

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / ECD	EPA 8151A	Dalapon
GC / ECD	EPA 8151A	Dicamba
GC / ECD	EPA 8151A	Dichloroprop
GC / ECD	EPA 8151A	Dinoseb
GC / ECD	EPA 8151A	MCPA
GC / ECD	EPA 8151A	MCPP
GC / ECD	EPA 8151A	Silvex
GC / FPD	EPA 8141A	Chlorpyrifos
GC / FPD	EPA 8141A	Coumaphos
GC / FPD	EPA 8141A	Demeton O + S
GC / FPD	EPA 8141A	Diazinon
GC / FPD	EPA 8141A	Dichlorvos
GC / FPD	EPA 8141A	Disulfoton
GC / FPD	EPA 8141A	Ethoprop
GC / FPD	EPA 8141A	Fensulfothion
GC / FPD	EPA 8141A	Fenthion
GC / FPD	EPA 8141A	Malathion
GC / FPD	EPA 8141A	Merphos A + B
GC / FPD	EPA 8141A	Methyl Azinphos
GC / FPD	EPA 8141A	Methyl Parathion
GC / FPD	EPA 8141A	Mevinphos
GC / FPD	EPA 8141A	Naled
GC / FPD	EPA 8141A	Phorate
GC / FPD	EPA 8141A	Ronnel
GC / FPD	EPA 8141A	Sulprofos
GC / FPD	EPA 8141A	Tetrachlorvinphos
GC / FPD	EPA 8141A	Tokuthion
GC / FPD	EPA 8141A	Trichloronate
GC / FID	EPA 8015B	GRO
GC / FID	EPA 8015B	DRO
GC / FID	EPA 8015B	Ethylene Glycol
GC / FID	EPA 8015B	Propylene Glycol
GC / FID	EPA 8015B	Diethylene Glycol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / FID	EPA 8015B	Triethylene Glycol
GC / FID	EPA 8015B	Tetraethylene Glycol
GC / MS	EPA 8260C	Chloroacetonitrile
GC / MS	EPA 8260C	1-chlorobutane
GC / MS	EPA 8260C	Methyl acrylate
GC / MS	EPA 8260C	Pentafluorobenzene
GC / MS	EPA 8260C	1,1,1,2-Tetrachloroethane
GC / MS	EPA 8260C	1,1,1-Trichloroethane
GC / MS	EPA 8260C	1,1,2,2-Tetrachloroethane
GC / MS	EPA 8260C	1,1,2-Trichloro-1,2,2-Trifluoroethane
GC / MS	EPA 8260C	1,1,2-Trichloroethane
GC / MS	EPA 8260C	1,1-Dichloroethane
GC / MS	EPA 8260C	1,1-Dichloroethene
GC / MS	EPA 8260C	1,1-Dichloropropene
GC / MS	EPA 8260C	1,2,3-Trichlorobenzene
GC / MS	EPA 8260C	1,2,3-Trichloropropane
GC / MS	EPA 8260C	1,2,4-Trichlorobenzene
GC / MS	EPA 8260C	1,2,4-Trimethylbenzene
GC / MS	EPA 8260C	1,2-Dibromo-3-Chloropropane
GC / MS	EPA 8260C	1,2-Dibromoethane
GC / MS	EPA 8260C	1,2-Dichlorobenzene
GC / MS	EPA 8260C	1,2-Dichloroethane
GC / MS	EPA 8260C	1,2-Dichloroethene (Total)
GC / MS	EPA 8260C	1,2-Dichloropropane
GC / MS	EPA 8260C	1,3,5-Trimethylbenzene
GC / MS	EPA 8260C	1,3-Dichlorobenzene
GC / MS	EPA 8260C	1,3-Dichloropropane
GC / MS	EPA 8260C	1,4-Dichlorobenzene
GC / MS	EPA 8260C	1-Chlorohexane
GC / MS	EPA 8260C	2,2-Dichloropropane
GC / MS	EPA 8260C	2-Butanone
GC / MS	EPA 8260C	2-Chlorotoluene
GC / MS	EPA 8260C	2-Hexanone

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / MS	EPA 8260C	4-Chlorotoluene
GC / MS	EPA 8260C	4-Methyl-2-Pentanone
GC / MS	EPA 8260C	Acetone
GC / MS	EPA 8260C	Benzene
GC / MS	EPA 8260C	Bromobenzene
GC / MS	EPA 8260C	Bromochloromethane
GC / MS	EPA 8260C	Bromodichloromethane
GC / MS	EPA 8260C	Bromoform
GC / MS	EPA 8260C	Bromomethane
GC / MS	EPA 8260C	Carbon Disulfide
GC / MS	EPA 8260C	Carbon Tetrachloride
GC / MS	EPA 8260C	Chlorobenzene
GC / MS	EPA 8260C	Chloroethane
GC / MS	EPA 8260C	Chloroform
GC / MS	EPA 8260C	Chloromethane
GC / MS	EPA 8260C	Cis-1,2-Dichloroethene
GC / MS	EPA 8260C	Cis-1,3-Dichloropropene
GC / MS	EPA 8260C	Dibromochloromethane
GC / MS	EPA 8260C	Dibromomethane
GC / MS	EPA 8260C	Dichlorodifluoromethane
GC / MS	EPA 8260C	Ethylbenzene
GC / MS	EPA 8260C	Hexachlorobutadiene
GC / MS	EPA 8260C	Iodomethane
GC / MS	EPA 8260C	Isopropylbenzene
GC / MS	EPA 8260C	M+P-Xylene
GC / MS	EPA 8260C	Methyl Tertiary Butyl Ether
GC / MS	EPA 8260C	Methylene Chloride
GC / MS	EPA 8260C	Naphthalene
GC / MS	EPA 8260C	N-Butanol
GC / MS	EPA 8260C	N-Butylbenzene
GC / MS	EPA 8260C	N-Propylbenzene
GC / MS	EPA 8260C	O-Xylene
GC / MS	EPA 8260C	P-Isopropyltoluene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / MS	EPA 8260C	Sec-Butylbenzene
GC / MS	EPA 8260C	Styrene
GC / MS	EPA 8260C	Tert-Butylbenzene
GC / MS	EPA 8260C	Tetrachloroethene
GC / MS	EPA 8260C	Toluene
GC / MS	EPA 8260C	Total Xylenes
GC / MS	EPA 8260C	Trans-1,2-Dichloroethene
GC / MS	EPA 8260C	Trans-1,3-Dichloropropene
GC / MS	EPA 8260C	Trichloroethene
GC / MS	EPA 8260C	Trichlorofluoromethane
GC / MS	EPA 8260C	Vinyl Acetate
GC / MS	EPA 8260C	Vinyl Chloride
GC / MS	EPA 8270D	1,2,4-Trichlorobenzene
GC / MS	EPA 8270D	1,2-Dichlorobenzene
GC / MS	EPA 8270D	1,3-Dichlorobenzene
GC / MS	EPA 8270D	1,4-Dichlorobenzene
GC / MS	EPA 8270D	1,4-Dioxane
GC / MS	EPA 8270D	2,3,4,6-Tetrachlorophenol
GC / MS	EPA 8270D	2,4,5-Trichlorophenol
GC / MS	EPA 8270D	2,4,6-Trichlorophenol
GC / MS	EPA 8270D	2,4-Dichlorophenol
GC / MS	EPA 8270D	2,4-Dimethylphenol
GC / MS	EPA 8270D	2,4-Dinitrophenol
GC / MS	EPA 8270D	2,4-Dinitrotoluene
GC / MS	EPA 8270D	2-Chloronaphthalene
GC / MS	EPA 8270D	2-Chlorophenol
GC / MS	EPA 8270D	2-Methylnaphthalene
GC / MS	EPA 8270D	2-Methylphenol
GC / MS	EPA 8270D	2-Nitroaniline
GC / MS	EPA 8270D	2-Nitrophenol
GC / MS	EPA 8270D	3,3'-Dichlorobenzidine
GC / MS	EPA 8270D	3+4-Methylphenol
GC / MS	EPA 8270D	4,6-Dinitro-2-Methylphenol

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / MS	EPA 8270D	4-Aminobiphenyl
GC / MS	EPA 8270D	4-Bromophenyl Phenyl Ether
GC / MS	EPA 8270D	4-Chloro-3-Methylphenol
GC / MS	EPA 8270D	4-Chloroaniline
GC / MS	EPA 8270D	4-Chlorophenyl Phenyl Ether
GC / MS	EPA 8270D	4-Nitroaniline
GC / MS	EPA 8270D	4-Nitrophenol
GC / MS	EPA 8270D	Acenaphthene
GC / MS	EPA 8270D	Acenaphthylene
GC / MS	EPA 8270D	Aniline
GC / MS	EPA 8270D	Anthracene
GC / MS	EPA 8270D	Azobenzene
GC / MS	EPA 8270D	Benzo(A)Anthracene
GC / MS	EPA 8270D	Benzo(A)Pyrene
GC / MS	EPA 8270D	Benzo(B)Fluoranthene
GC / MS	EPA 8270D	Benzo(G,H,I)Perylene
GC / MS	EPA 8270D	Benzo(K)Fluoranthene
GC / MS	EPA 8270D	Benzoic Acid
GC / MS	EPA 8270D	Benzyl Alcohol
GC / MS	EPA 8270D	Bis(2-Chloroethoxy)Methane
GC / MS	EPA 8270D	Bis(2-Chloroethyl)Ether
GC / MS	EPA 8270D	Bis(2-Ethylhexyl) Adipate
GC / MS	EPA 8270D	Butyl Benzyl Phthalate
GC / MS	EPA 8270D	Carbazole
GC / MS	EPA 8270D	Chrysene
GC / MS	EPA 8270D	Dibenzo(A,H)Anthracene
GC / MS	EPA 8270D	Dibenzofuran
GC / MS	EPA 8270D	Diethyl Phthalate
GC / MS	EPA 8270D	Dimethyl Phthalate
GC / MS	EPA 8270D	Di-N-Butyl Phthalate
GC / MS	EPA 8270D	Di-N-Octyl Phthalate
GC / MS	EPA 8270D	Fluoranthene
GC / MS	EPA 8270D	Fluorene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC / MS	EPA 8270D	Hexachlorobenzene
GC / MS	EPA 8270D	Hexachlorobutadiene
GC / MS	EPA 8270D	Hexachlorocyclopentadiene
GC / MS	EPA 8270D	Hexachloroethane
GC / MS	EPA 8270D	Indeno(1,2,3-Cd)Pyrene
GC / MS	EPA 8270D	Isophorone
GC / MS	EPA 8270D	Naphthalene
GC / MS	EPA 8270D	Nitrobenzene
GC / MS	EPA 8270D	N-Nitrosodimethylamine
GC / MS	EPA 8270D	N-Nitroso-Di-N-Propylamine
GC / MS	EPA 8270D	N-Nitrosodiphenylamine
GC / MS	EPA 8270D	Pentachlorophenol
GC / MS	EPA 8270D	Phenanthrene
GC / MS	EPA 8270D	Phenol
GC / MS	EPA 8270D	Pyrene
GC / MS	EPA 8270D	Pyridine
Gas Proportional Counting	EPA 900 / EPA 9310	Gross Alpha
Gas Proportional Counting	EPA 900 / EPA 9310	Gross Beta
Gas Proportional Counting	EPA 904 / EPA 9320	Ra228
Gas Proportional Counting	HASL 300 Sr01 HASL 300 Sr02 ASTM D5811	Strontium 90
Gas Proportional Counting	HASL 300 Sr01 HASL 300 Sr02 ASTM D5811	Strontium 90
Liquid Scintillation Counting	EPA 906.0 SM 7500 3H	Tritium
Liquid Scintillation Counting	EPA C-01	Carbon-14
Liquid Scintillation Counting	DOE RP550 DOE RS551	Technicium-99
Liquid Scintillation Counting	Horwitz, Chiariza, Dietz 1992	Lead-210
Liquid Scintillation Counting	ALS SOP 704	Pu241, Pm147
Liquid Scintillation Counting	ALS SOP 774	Nickle-63
Emanation	EPA 903.1 SM 7500-Ra C	Radium-226
Gas Proportional Counting	EPA 903.0 / EPA 9315	Total Radium
Gas Proportional Counting	EPA 903.0 / EPA 9315	Radium-226

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gas Proportional Counting	EPA 902.0 ALS SOP 753	Iodine-129
Alpha-Spec	HASL 300 U02 ASTM D3972	Ac-227
Alpha-Spec	HASL 300 U02 ASTM D3972	Am-241
Alpha-Spec	HASL 300 U02 ASTM D3972	Am-242/243
Alpha-Spec	HASL 300 U02 ASTM D3972	Am-243
Alpha-Spec	HASL 300 U02 ASTM D3972	Cm-242
Alpha-Spec	HASL 300 U02 ASTM D3972	Cm-243/244
Alpha-Spec	HASL 300 U02 ASTM D3972	Cm-244
Alpha-Spec	HASL 300 U02 ASTM D3972	Cm-245/246
Alpha-Spec	HASL 300 U02 ASTM D3972	Np-237
Alpha-Spec	HASL 300 U02 ASTM D3972	Po-210
Alpha-Spec	HASL 300 U02 ASTM D3972	Pu-238
Alpha-Spec	HASL 300 U02 ASTM D3972	Pu-239
Alpha-Spec	HASL 300 U02 ASTM D3972	Pu-239/240
Alpha-Spec	HASL 300 U02 ASTM D3972	Pu-242
Alpha-Spec	ALS SOP 701	Ra-226
Alpha-Spec	HASL 300 U02 ASTM D3972	Th-227
Alpha-Spec	HASL 300 U02 ASTM D3972	Th-228
Alpha-Spec	HASL 300 U02 ASTM D3972	Th-230
Alpha-Spec	HASL 300 U02 ASTM D3972	Th-232
Alpha-Spec	HASL 300 U02 ASTM D3972	U-232

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Alpha-Spec	HASL 300 U02 ASTM D3972	U-233/234
Alpha-Spec	HASL 300 U02 ASTM D3972	U-234
Alpha-Spec	HASL 300 U02 ASTM D3972	U-235
Alpha-Spec	HASL 300 U02 ASTM D3972	U-235/236
Alpha-Spec	HASL 300 U02 ASTM D3972	U-238
Alpha-Spec	HASL 300 U02 ASTM D3972	Uranium, Total
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ac-227
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ac-228
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ag-108m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ag-110m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Al-26
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Am-241
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Am-243
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-72
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-73
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	As-74

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ba-133
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ba-140
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Be-7
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-211
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-212
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Bi-214
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-76
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-77
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Br-82
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cd-109
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-139
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-141
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ce-144
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cf-249

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cf-251
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cl-39
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cm-243
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-56
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-57
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-58
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Co-60
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cr-51
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-134
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-135
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-136
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cs-137
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-152
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-154

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Eu-155
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Fe-59
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Gd-153
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ge-68
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hf-181
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hg-197m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Hg-203
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	I-131
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ir-192
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	K-40
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Kr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	La-140
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Mn-54
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Na-22

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Na-24
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nb-94
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nb-95
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Nd-147
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-236
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-237
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Np-239
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Os-191
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pa-231
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pa-234m
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-210
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-211
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-212
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pb-214

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pm-144
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Pm-146
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Po-209
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-223
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-224
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-226
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ra-228
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-83
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-86
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rh-101
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rh-106
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rn-222
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ru-103
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ru-106

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sb-124
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sb-125
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sc-46
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Se-75
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sn-113
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sn-126
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Sr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Ta-182
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Tb-160
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-227
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-228
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-230
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-231
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-232

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Th-234
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Tl-208
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	U-235
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Uranium, Total
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	V-48
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Y-88
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Zn-65
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Zr-95
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Au-198
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cr-51
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Kr-85
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Te-132
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Rb-86
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Se-75

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	Cd-109
Gamma Spec	EPA 901.1 HASL 300 Ga-01-R EML 4.5.2.3	In-111
Preparation	**Method**	**Type**
Preparation	EPA 3060 A	Alkaline Digestion For Hexavalent Chromium
Preparation	EPA 3050 B	Acid Digestion Of Sediments, Sludges And Soils
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Metals
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Semi-Volatiles
Leaching Procedure	EPA 1311	Toxicity Characteristic Leaching Procedure Volatiles
Preparation	EPA 3540C	Soxhlet Extraction
Preparation	EPA 3580A	Waste Dilution
Cleanup Procedure	EPA 3620B	Florisil Cleanup
Cleanup Procedure	EPA 3630C	Silica Gel Cleanup
Cleanup Procedure	EPA 3640A	Gel Permeation Cleanup
Cleanup Procedure	EPA 3660A	Sulfur Cleanup
Purge and Trap	EPA 5035A	Purge-And-Trap And Extraction For Volatile Organics
Preparation	EPA-3546 for EPA 8081 A	Microwave Extraction
Preparation	EPA-3546 for EPA 8082	Microwave Extraction
Preparation	EPA-3546 for EPA 8270 D	Microwave Extraction

Notes:

1) This laboratory offers commercial testing service.

Approved by: _____ Date: August 20, 2015

R. Douglas Leonard
Chief Technical Officer

Re-issued: 4/15/13 Revised: 8/29/13 Revised: 9/10/14 Revised: 8/20/15

NEW YORK
STATE OF
OPPORTUNITY.

Department
of Health

ANDREW M. CUOMO
Governor

HOWARD A. ZUCKER, M.D., J.D.
Commissioner

SALLY DRESLIN, M.S., R.N.
Executive Deputy Commissioner

LAB ID: 12036

June 16, 2015

MR. ROY FRENCH
ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

Certificate Expiration Date:
April 01, 2016

Dear Mr. French,

Enclosed are revised certificate(s) of approval issued to your environmental laboratory for the current permit year. The certificate(s) supersede(s) any previously issued one(s) and are in effect through the expiration date listed. Please carefully examine the certificate(s) to insure that the categories, subcategories, analytes, and methods for which your laboratory is approved are correct. In addition, verify that your laboratory's name, address, lead technical director, and identification number are accurate.

Pursuant to NYCRR Subpart 55-2.2, original certificates must be posted conspicuously in the laboratory and copies shall be made available to any client of the laboratory upon request.

Pursuant to NYCRR Subpart 55-2.6, any misrepresentation of the fields of accreditation (category - method - analyte) for which your laboratory is approved may result in denial, suspension, or revocation of your certification. Any use of the Environmental Laboratory Approval Program (ELAP) or National Environmental Laboratory Accreditation Program (NELAP) name, reference to the laboratory's approval status, and/or using the NELAP logo in any catalogs, advertising, business solicitations, proposals, quotations, laboratory analytical reports, or other materials must include the laboratory's ELAP identification number and distinguish between testing for which the laboratory is approved and testing for which the laboratory is not approved.

If the changes to your certificate are due to insufficient proficiency tests and/or proficiency test failures, the expired certificates (listed below) must be returned to the ELAP office within 10 days of the date of this letter. In addition, your laboratory must investigate and document the root cause for any insufficient and/or unsatisfactory proficiency tests.

If you have any questions, please contact ELAP at the New York State Department of Health (NYS DOH), Wadsworth Center, PO Box 509, Albany NY, 12201-0509; by phone at (518) 485-5570; by facsimile at (518) 485-5568; and by email at elap@health.ny.gov.

Sincerely,

Michael P. Ryan, M.T. (ASCP), Ph.D.
Director, Division of Laboratory Quality Certification
Environmental Laboratory Approval Program

NEW YORK STATE OF OPPORTUNITY. Department of Health

ANDREW M. CUOMO
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Executive Deputy Commissioner

LAB ID: 12036

June 16, 2015

MR. ROY FRENCH
ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

Dear Mr. French,

A revised certificate has been generated because of the change(s) listed below.

If your laboratory has applied for a change in the laboratory's location and/or technical director, the approved change(s) will be reflected on the certificate.

If the changes to your certification are due to insufficient proficiency tests and/or proficiency test failures, the expired certificates must be returned to the Environmental Laboratory Approval Program (ELAP) office within 10 days of the date of this letter. In addition, your laboratory must investigate the root cause for any insufficient and/or unsatisfactory proficiency tests.

In addition, your laboratory must investigate and document the root cause for any insufficient and/or unsatisfactory proficiency tests. If your lab lost accreditation due to two PT failures, you must submit the corrective action response to ELAP for review before accreditation will be re-instated.

AppCat	Analyte Name	Method Name	Comments	Date
NW - NELAC	2,2'-Oxybis(1-chloropropane)		Analyte Name Changed	06/15/2015
SW - NELAC	2,2'-Oxybis(1-chloropropane)		Analyte Name Changed	06/15/2015

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2016
Issued April 14, 2015
Revised June 16, 2015

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROY FRENCH
ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

NY Lab Id No: 12036

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1-Naphthylamine	EPA 8270D
2-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Propionitrile	EPA 8260C
Pyridine	EPA 8270D
Benzidines	
3,3'-Dichlorobenzidine	EPA 8270D
Benzidine	EPA 8270D
Chlorinated Hydrocarbon Pesticides	
4,4'-DDD	EPA 608
4,4'-DDE	EPA 608
4,4'-DDT	EPA 608

Chlorinated Hydrocarbon Pesticides	
Aldrin	EPA 608
alpha-BHC	EPA 608
beta-BHC	EPA 608
Chlordane Total	EPA 608
delta-BHC	EPA 608
Dieldrin	EPA 608
Endosulfan I	EPA 608
Endosulfan II	EPA 608
Endosulfan sulfate	EPA 608
Endrin	EPA 608
Endrin aldehyde	EPA 608
Heptachlor	EPA 608
Heptachlor epoxide	EPA 608
Methoxychlor	EPA 608
Toxaphene	EPA 608
Chlorinated Hydrocarbons	
1,2,3-Trichlorobenzene	EPA 8260C
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Hexachloropropene	EPA 8270D

Serial No.: 53132

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2016
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225 COMMERCE DRIVE
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NY Lab Id No: 12036

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Chlorinated Hydrocarbons	
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
Dissolved Gases	
Ethane	RSK-175
Ethene (Ethylene)	RSK-175
Methane	RSK-175
Propane	RSK-175
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 611
	EPA 8270D
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Metals I	
Barium, Total	EPA 200.7 Rev. 4.4

Metals I	
Barium, Total	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Calcium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Lead, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Magnesium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Potassium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Sodium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Strontium, Total	EPA 200.7 Rev. 4.4

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MR. ROY FRENCH
ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

NY Lab Id No: 12036

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Metals I	
Strontium, Total	EPA 200.8 Rev. 5.4
Metals II	
Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Arsenic, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium VI	EPA 7196A
Mercury, Total	EPA 245.1 Rev. 3.0
	EPA 7470A
Selenium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Metals III	
Cobalt, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Molybdenum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.7 Rev. 4.4

Metals III	
Thallium, Total	EPA 200.8 Rev. 5.4
Tin, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Titanium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Uranium (Mass)	EPA 200.8 Rev. 5.4
Mineral	
Chloride	EPA 300.0 Rev. 2.1
Fluoride, Total	EPA 300.0 Rev. 2.1
Sulfate (as SO4)	EPA 300.0 Rev. 2.1
Miscellaneous	
Boron, Total	EPA 200.7 Rev. 4.4
Bromide	EPA 300.0 Rev. 2.1
Cyanide, Total	EPA 9014
Oil and Grease Total Recoverable (HEM)	EPA 1664A
Organic Carbon, Total	SM 5310C-00,-11
Silica, Dissolved	EPA 200.7 Rev. 4.4
Specific Conductance	EPA 120.1 Rev. 1982
Sulfide (as S)	SM 4500-S2- F-00,-11
Nitroaromatics and Isophorone	
1,3,5-Trinitrobenzene	EPA 8270D
1,3-Dinitrobenzene	EPA 8270D
2,4-Dinitrotoluene	EPA 8270D
2,6-Dinitrotoluene	EPA 8270D

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ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

NY Lab Id No: 12036

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All approved analytes are listed below:

Nitroaromatics and Isophorone	
Isophorone	EPA 8270D
Nitrobenzene	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrient	
Ammonia (as N)	SM 4500-NH3 H-97,-11
Nitrite (as N)	SM 4500-NO2 B-00,-11
Orthophosphate (as P)	SM 4500-P E-99,-11
Phosphorus, Total	SM 4500-P H-99,-11
Organophosphate Pesticides	
Azinphos methyl	EPA 8141B
Chlorpyriphos	EPA 8141B
Demeton-O	EPA 8141B
Demeton-S	EPA 8141B
Diazinon	EPA 8141B
Disulfoton	EPA 8141B
Organophosphate Pesticides	
Malathion	EPA 8141B
Parathion ethyl	EPA 8141B
Phorate	EPA 8141B
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polychlorinated Biphenyls	
PCB-1016	EPA 608
PCB-1221	EPA 608
PCB-1232	EPA 608
PCB-1242	EPA 608
PCB-1248	EPA 608
PCB-1254	EPA 608
PCB-1260	EPA 608
Polynuclear Aromatics	
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D

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Polynuclear Aromatics	
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(ghi)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D
2,4-Dichlorophenol	EPA 8270D
2,4-Dimethylphenol	EPA 8270D
2,4-Dinitrophenol	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 8270D

Priority Pollutant Phenols	
2-Methyl-4,6-dinitrophenol	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 8270D
Pentachlorophenol	EPA 8270D
Phenol	EPA 8270D
Radiological Analytes	
Gross Alpha	EPA 900.0
Gross Beta	EPA 900.0
Radium-226	EPA 903.1
	EPA 903.0
Radium-228	EPA 904.0
Radon	SM 7500 Rn-06,-11
Strontium-89	HASL 300 1997 Sr-01,02-RC (G
Strontium-90	HASL 300 1997 Sr-01,02-RC (G
Uranium (Activity)	HASL 300 1997 U-02-RC
Residue	
Solids, Total	SM 2540 B-97,-11
Solids, Total Dissolved	SM 2540 C-97,-11
Solids, Total Suspended	SM 2540 D-97,-11

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All approved analytes are listed below:

Semi-Volatile Organics	
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
Acetophenone	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
1,4-Dichlorobenzene	EPA 8260C
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
Ethyl benzene	EPA 8260C
Isopropylbenzene	EPA 8260C
Volatile Aromatics	
m/p-Xylenes	EPA 8260C
Naphthalene, Volatile	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
o-Xylene	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
Toluene	EPA 8260C
Total Xylenes	EPA 8260C
Volatile Halocarbons	
1,1,1,2-Tetrachloroethane	EPA 8260C
1,1,1-Trichloroethane	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260C
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C
1,1-Dichloroethene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloroethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
3-Chloropropene (Allyl chloride)	EPA 8260C

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All approved analytes are listed below:

Volatile Halocarbons	
Bromochloromethane	EPA 8260C
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Carbon tetrachloride	EPA 8260C
Chloroethane	EPA 8260C
Chloroform	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 8260C
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,3-Dichloropropene	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatiles Organics	
1,4-Dioxane	EPA 8260C
2-Butanone (Methylethyl ketone)	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C

Volatiles Organics	
Acetonitrile	EPA 8260C
Carbon Disulfide	EPA 8260C
Di-ethyl ether	EPA 8260C
Isobutyl alcohol	EPA 8260C
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5030C
	EPA 3510C
	EPA 3520C
	EPA 9010C

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All approved analytes are listed below:

Acrylates	
Acrolein (Propenal)	EPA 8260C
Acrylonitrile	EPA 8260C
Ethyl methacrylate	EPA 8260C
Methyl acrylonitrile	EPA 8260C
Methyl methacrylate	EPA 8260C
Amines	
1-Naphthylamine	EPA 8270D
2-Nitroaniline	EPA 8270D
3-Nitroaniline	EPA 8270D
4-Chloroaniline	EPA 8270D
4-Nitroaniline	EPA 8270D
5-Nitro-o-toluidine	EPA 8270D
Aniline	EPA 8270D
Carbazole	EPA 8270D
Characteristic Testing	
Corrosivity	EPA 1110A
Ignitability	EPA 1010A
TCLP	EPA 1311
Chlorinated Hydrocarbons	
1,2,4,5-Tetrachlorobenzene	EPA 8270D
1,2,4-Trichlorobenzene	EPA 8270D
2-Chloronaphthalene	EPA 8270D
Hexachlorobenzene	EPA 8270D
Hexachlorobutadiene	EPA 8270D
Chlorinated Hydrocarbons	
Hexachlorocyclopentadiene	EPA 8270D
Hexachloroethane	EPA 8270D
Hexachloropropene	EPA 8270D
Pentachlorobenzene	EPA 8270D
Chlorophenoxy Acid Pesticides	
2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A
Haloethers	
2,2'-Oxybis(1-chloropropane)	EPA 8270D
4-Bromophenylphenyl ether	EPA 8270D
4-Chlorophenylphenyl ether	EPA 8270D
Bis(2-chloroethoxy)methane	EPA 8270D
Bis(2-chloroethyl)ether	EPA 8270D
Metals I	
Cadmium, Total	EPA 6020A
Calcium, Total	EPA 6020A

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Metals I	
Copper, Total	EPA 6020A
Lead, Total	EPA 6020A
Magnesium, Total	EPA 6020A
Manganese, Total	EPA 6020A
Nickel, Total	EPA 6020A
Potassium, Total	EPA 6020A
Silver, Total	EPA 6020A
Strontium, Total	EPA 6020A
Metals II	
Aluminum, Total	EPA 6020A
Antimony, Total	EPA 6020A
Chromium VI	EPA 7196A
Mercury, Total	EPA 7471B
Selenium, Total	EPA 6020A
Vanadium, Total	EPA 6020A
Metals III	
Molybdenum, Total	EPA 6020A
Thallium, Total	EPA 6020A
Minerals	
Bromide	EPA 9056A
Chloride	EPA 9056A
Fluoride, Total	EPA 9056A
Sulfate (as SO4)	EPA 9056A
Miscellaneous	
Cyanide, Total	EPA 9014
Nitroaromatics and Isophorone	
1,2-Dinitrobenzene	EPA 8270D
1,3,5-Trinitrobenzene	EPA 8270D
1,3-Dinitrobenzene	EPA 8270D
1,4-Dinitrobenzene	EPA 8270D
2,4-Dinitrotoluene	EPA 8270D
2,6-Dinitrotoluene	EPA 8270D
Isophorone	EPA 8270D
Nitrobenzene	EPA 8270D
Pyridine	EPA 8270D
Nitrosoamines	
N-Nitrosodiethylamine	EPA 8270D
N-Nitrosodimethylamine	EPA 8270D
N-Nitrosodi-n-butylamine	EPA 8270D
N-Nitrosodi-n-propylamine	EPA 8270D
N-Nitrosodiphenylamine	EPA 8270D
N-nitrosomethylethylamine	EPA 8270D
N-nitrosomorpholine	EPA 8270D
N-nitrosopiperidine	EPA 8270D
N-Nitrosopyrrolidine	EPA 8270D
Nutrients	
Nitrate (as N)	EPA 9056A
Nitrite (as N)	EPA 9056A

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Nutrients	
Orthophosphate (as P)	EPA 9056A
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270D
Bis(2-ethylhexyl) phthalate	EPA 8270D
Diethyl phthalate	EPA 8270D
Dimethyl phthalate	EPA 8270D
Di-n-butyl phthalate	EPA 8270D
Di-n-octyl phthalate	EPA 8270D
Polynuclear Aromatic Hydrocarbons	
2-Acetylaminofluorene	EPA 8270D
3-Methylcholanthrene	EPA 8270D
7,12-Dimethylbenzyl (a) anthracene	EPA 8270D
Acenaphthene	EPA 8270D
Acenaphthylene	EPA 8270D
Anthracene	EPA 8270D
Benzo(a)anthracene	EPA 8270D
Benzo(a)pyrene	EPA 8270D
Benzo(b)fluoranthene	EPA 8270D
Benzo(ghi)perylene	EPA 8270D
Benzo(k)fluoranthene	EPA 8270D
Chrysene	EPA 8270D

Polynuclear Aromatic Hydrocarbons	
Dibenzo(a,h)anthracene	EPA 8270D
Fluoranthene	EPA 8270D
Fluorene	EPA 8270D
Indeno(1,2,3-cd)pyrene	EPA 8270D
Naphthalene	EPA 8270D
Phenanthrene	EPA 8270D
Pyrene	EPA 8270D
Priority Pollutant Phenols	
2,3,4,6 Tetrachlorophenol	EPA 8270D
2,4,5-Trichlorophenol	EPA 8270D
2,4,6-Trichlorophenol	EPA 8270D
2,4-Dichlorophenol	EPA 8270D
2,4-Dimethylphenol	EPA 8270D
2,4-Dinitrophenol	EPA 8270D
2,6-Dichlorophenol	EPA 8270D
2-Chlorophenol	EPA 8270D
2-Methyl-4,6-dinitrophenol	EPA 8270D
2-Methylphenol	EPA 8270D
2-Nitrophenol	EPA 8270D
3-Methylphenol	EPA 8270D
4-Chloro-3-methylphenol	EPA 8270D
4-Methylphenol	EPA 8270D
4-Nitrophenol	EPA 8270D
Pentachlorophenol	EPA 8270D
Phenol	EPA 8270D

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All approved analytes are listed below:

Semi-Volatile Organics	
1,2-Dichlorobenzene, Semi-volatile	EPA 8270D
1,3-Dichlorobenzene, Semi-volatile	EPA 8270D
1,4-Dichlorobenzene, Semi-volatile	EPA 8270D
2-Methylnaphthalene	EPA 8270D
Acetophenone	EPA 8270D
Benzoic Acid	EPA 8270D
Benzyl alcohol	EPA 8270D
Dibenzofuran	EPA 8270D
Ethyl methanesulfonate	EPA 8270D
Isosafrole	EPA 8270D
Methyl methanesulfonate	EPA 8270D
Phenacetin	EPA 8270D
Safrole	EPA 8270D
Volatile Aromatics	
1,2,4-Trichlorobenzene, Volatile	EPA 8260C
1,2-Dichlorobenzene	EPA 8260C
1,3,5-Trimethylbenzene	EPA 8260C
1,3-Dichlorobenzene	EPA 8260C
1,4-Dichlorobenzene	EPA 8260C
2-Chlorotoluene	EPA 8260C
4-Chlorotoluene	EPA 8260C
Benzene	EPA 8260C
Bromobenzene	EPA 8260C
Chlorobenzene	EPA 8260C
Ethyl benzene	EPA 8260C

Volatile Aromatics	
Isopropylbenzene	EPA 8260C
m/p-Xylenes	EPA 8260C
n-Butylbenzene	EPA 8260C
n-Propylbenzene	EPA 8260C
o-Xylene	EPA 8260C
sec-Butylbenzene	EPA 8260C
Styrene	EPA 8260C
Toluene	EPA 8260C
Total Xylenes	EPA 8260C
Volatile Halocarbons	
1,1,1-Trichloroethane	EPA 8260C
1,1,2,2-Tetrachloroethane	EPA 8260C
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260C
1,1,2-Trichloroethane	EPA 8260C
1,1-Dichloroethane	EPA 8260C
1,1-Dichloroethene	EPA 8260C
1,2,3-Trichloropropane	EPA 8260C
1,2-Dibromo-3-chloropropane	EPA 8260C
1,2-Dibromoethane	EPA 8260C
1,2-Dichloropropane	EPA 8260C
1,3-Dichloropropane	EPA 8260C
2,2-Dichloropropane	EPA 8260C
2-Chloro-1,3-butadiene (Chloroprene)	EPA 8260C
2-Chloroethylvinyl ether	EPA 8260C
Bromochloromethane	EPA 8260C

Serial No.: 53133

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2016
Issued April 14, 2015
Revised June 16, 2015

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. ROY FRENCH
ALS ENVIRONMENTAL - FORT COLLINS
225 COMMERCE DRIVE
FORT COLLINS, CO 80526

NY Lab Id No: 12036

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2003) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Volatile Halocarbons	
Bromodichloromethane	EPA 8260C
Bromoform	EPA 8260C
Bromomethane	EPA 8260C
Carbon tetrachloride	EPA 8260C
Chloroethane	EPA 8260C
Chloroform	EPA 8260C
Chloromethane	EPA 8260C
cis-1,2-Dichloroethene	EPA 8260C
cis-1,3-Dichloropropene	EPA 8260C
Dibromomethane	EPA 8260C
Dichlorodifluoromethane	EPA 8260C
Hexachlorobutadiene, Volatile	EPA 8260C
Methylene chloride	EPA 8260C
Tetrachloroethene	EPA 8260C
trans-1,2-Dichloroethene	EPA 8260C
trans-1,4-Dichloro-2-butene	EPA 8260C
Trichloroethene	EPA 8260C
Trichlorofluoromethane	EPA 8260C
Vinyl chloride	EPA 8260C
Volatile Organics	
1,4-Dioxane	EPA 8260C
2-Hexanone	EPA 8260C
4-Methyl-2-Pentanone	EPA 8260C
Acetone	EPA 8260C
Acetonitrile	EPA 8260C

Volatile Organics	
Carbon Disulfide	EPA 8260C
Di-ethyl ether	EPA 8260C
Isobutyl alcohol	EPA 8260C
Methyl tert-butyl ether	EPA 8260C
Propionitrile	EPA 8260C
Vinyl acetate	EPA 8260C
Sample Preparation Methods	
	EPA 5035A-H
	EPA 3580A
	EPA 3010A
	EPA 3005A
	EPA 3050B
	EPA 3540C
	EPA 3060A
	EPA 9010C

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ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATON OF ORGANOCHLORIDE PESTICIDES BY GAS CHROMATOGRAPHY
REFERENCED METHOD:	SW 8081 A OR B; AND EPA 608
SOP ID:	402
REV. NUMBER:	14
EFFECTIVE DATE:	APRIL 11, 2013

ALS
STANDARD OPERATING PROCEDURE 402 REVISION 14

TITLE: DETERMINATION OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY - METHODS SW8081A OR B, AND EPA 608

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER_____DATE_____

QUALITY ASSURANCE MANAGER_____DATE_____

LABORATORY MANAGER _____DATE_____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references – SW8081A or B, and EPA 608, are used to determine the concentration of certain organochlorine pesticides in liquid (SW8081A, B and EPA 608) and solid matrices (SW8081A,B). The following compounds typically comprise ALS Laboratory Group - Fort Collins (ALS)'s target analyte list:

aldrin	4,4'-DDD	endrin
alpha(α)-BHC	4,4'-DDE	endrin aldehyde
beta(β)-BHC	4,4'-DDT	endrin ketone
gamma(γ)-BHC (lindane)	dieldrin	heptachlor
delta(δ)-BHC	endosulfan I	heptachlor epoxide
alpha(α)-chlordane	endosulfan II	methoxychlor
gamma (γ) chlordane	endosulfan sulfate	toxaphene
technical chlordane		

Other compounds may be analyzed if successful demonstration of capability (DOC) and method detection limit (MDL) studies are performed.

2. SUMMARY

Samples are extracted and the extracts concentrated and solvent exchanged using appropriate ALS SOPs (i.e., 620 [Microwave]; 625 [Soxhlet]; 626 [Separatory Funnel]; 607 [Kuderna-Danish Reduction]; and 637 [Concentration and Solvent Exchange]). The extracts are injected into a gas chromatograph (GC) containing a sample splitter and two columns of varying selectivity (i.e., dissimilar elution and retention time properties). The target analytes are separated in the columns and detected by two electron capture detectors (ECDs). This chromatography system allows tentative identification by one column and confirmation by the other column to be performed simultaneously. Quantitation is performed using the best column response yielded for each analyte. The Analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the primary quantitation column for each analyte detected and reported. The particular value

that is selected for reporting is often marked (designated) in the raw data (e.g., quantitation report, run log). If results from both columns are comparable, the highest result is reported.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Interference from phthalate esters can be minimized by using plastic-free solvent containers and scrupulously cleaned glassware (SOP 334) that has been kiln-baked or solvent-rinsed prior to use. Use of low phthalate gloves, such as nitrile, may also important. These practices are important both in the field and in the laboratory.

4.2 Method SW3620B Florisil cleanup may be used to remove polar interferences from sample extracts (SOP 648).

4.3 Elemental sulfur (particularly in sediment samples) may interfere with early-eluting pesticides. Method SW3660B sulfur cleanup may be performed (SOP 634).

NOTE: The recovery of endrin aldehyde can be drastically reduced when using the TBA procedure in Method SW3660B. For this reason, only the copper powder technique will be used.

4.4 Dilution is performed if high molecular weight organic interferences are present. Alternatively these may be removed using Method SW3640A GPC cleanup (SOP 641).

4.5 The presence of Aroclors in the sample may interfere with the recognition and quantitation of single components, such as 4,4'-DDT, or multi-component pesticides such as technical chlordane and toxaphene. Interpretive examples are provided in Appendix I of this SOP. If multi-component interference is observed, the interference is discussed in the data package narrative. Consult the LIMS program specification and ensure that the Project Manager is informed if interferences are present.

4.6 Particular caution is used in identifying the presence of interferences. It should be noted that methoxychlor may also be susceptible to interference.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH (GC), AUTOSAMPLER AND DETECTORS:

5.1.1 - Hewlett Packard (HP) 5890 Series II GC equipped with HP 7673A autosampler and electron capture detectors (ECDs) or equivalents

5.1.2 Agilent Technologies 7890A GC equipped with Agilent 7693 autosampler and electron capture detectors (U-EDS or ECD)

5.2 CHROMATOGRAPHIC DATA ACQUISITION AND PROCESSING SYSTEM - Hewlett Packard ChemStation (Enviroquant™) or equivalent

5.3 COLUMNS -

RTx-CLPesticides or equivalent (30m, 0.25 or 0.32mm ID, 0.5µm film), RTx-CLPesticides II or equivalent (30m, 0.25 or 0.32mm ID, 0.25µm film), guard column

Restek Capillary Column:	RTX-CLPesticides	#11123 (0.25mm)
Restek Capilary Column:	RTX-CLPesticides2	#11323 (0.25mm)
Restek Capilary Column:	RTX-CLPesticides	#11139 (0.32mm)
Restek Capilary Column:	RTX-CLPesticides2	#11324 (0.32mm)

5.4 GASES - ultra high purity (99.999%)
Helium - carrier gas
Nitrogen - make-up gas

5.5 MEASURING DEVICES
Syringes - 10µL-1000µL
Volumetric flasks, Class A with stoppers, 10mL-100mL

5.6 GC CONSUMABLES:

- Vials - Resolution Systems 670VT011LM-1232 or equivalent
- Caps - Resoltuion Systems 67-C141-11 or equivalent

- Inlet Seals, dual vespel ring - Restek 0.8mm #21243, or equivalent
- Septa, 11mm - Restek #20365 or equivalent
- O-ring, graphite, 6.5mm - Restek #20299, or equivalent
- O-ring, Viton - Restek #20377, or equivalent
- Liner, splitless, 4mm ID - Restek #20799-214.5, or equivalent
- Glass Wool, deactivated - Restek #20789, or equivalent
- Gold Seal - Restek #21306, or equivalent
- Universal Presstight Y - Restek #20469, or equivalent
- Vespel/Graphite Ferrule (detector) - Restek #20221, or equivalent
- Compact Vespel/Graphite Ferrule - Restek #20249, or equivalent
- Graphite Ferrules - various sizes
- Split Vent Trap (SW8081A,B) - Agilent RDT-1023, or equivalent

6. REAGENTS AND STANDARDS

6.1 SOLVENTS - **Only pesticide residue grade or equivalent may be used.**
Hexane - Burdick and Jackson 216-4, or equivalent
Methanol - Burdick and Jackson 230-4, or equivalent

6.2 STANDARDS
All standards are stored following ALS SOP 300 guidance, which is superseded by any guidance in this SOP. Generally after opening vials, the standards for this procedure are stored in the freezer (-10°C and -20°C), in PTFE-capped, or equivalent vials. Unopened stock standards in flame-sealed ampules are valid until the manufacturer's expiration date and may be stored at room temperature, if recommended by the manufacturer. Opened stock standards and intermediate standards expire six months from opening (preparation) or the manufacturer's expiration date, whichever is sooner. Standards may be replaced sooner if laboratory QC analyses or other factors indicate deterioration.

All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

6.2.1 Stock standards are generally purchased as mixes from vendors with certified concentrations. At a minimum, two independent sources of stock standards are needed for target analytes. An appropriate volume of stock standard is diluted (in hexane) to a specified volume to create intermediate standards. The intermediate standards are further diluted to volume using

an appropriate solvent to create the working standards. Working standards are prepared the day of use and documented in the analytical run log. A detailed description of the concentrations of the working standards and how they are used can be found in Section 8 of this SOP.

6.2.2 ICV Solution - Second source confirmation is required, this is accomplished by preparing an initial calibration verification (ICV) solution at a concentration near the midpoint of the calibration curve.

6.2.3 Endrin and 4,4'-DDT Standard (Breakdown Standard) - This standard is used to check inertness of injector system and columns prior to running standards or samples. Only one source is needed and primary, intermediate and working standards are created as described above and detailed in Section 8.

6.2.4 Surrogate Spike Solution - A primary standard containing tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB) is purchased from a vendor. A solution containing 500ng/mL is prepared in methanol or other suitable solvent. This solution is used by the Organics Extraction Group to spike all samples for this test prior to extraction. Other concentrations or solvents may be used as needed (i.e., as defined in the applicable LIMS Program Specification).

6.2.5 Spike Solution - A primary standard (or standards) is used to prepare a solution to be used by the Organics Extractions Group in preparing laboratory control samples (LCS/LCSD) or matrix spiked samples (MS/MSD). This standard typically contains all the single component analytes listed in Section 1 at 400ng/mL in methanol. Other concentrations and solvents may be used as appropriate. Technical chlordane and toxaphene are not included in this matrix spike solution because either would interfere with the quantification of the single component pesticides. Technical chlordane and toxaphene-spiked samples are prepared separately when required (i.e., as indicated in the LIMS program specification).

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Liquid samples are generally not chemically preserved and must be collected in amber glass containers (generally 1000mL) with Teflon-lined lids. Samples must be maintained at 4±2°C and extracted within 7 days of collection. Sodium thiosulfate ($Na_2S_2O_3$) may be used to dechlorinate liquid samples that contain residual chlorine. This should be accomplished by the client in the field. The Project Manager may designate need for residual chlorine check of the sample upon receipt. Additionally, samples for Method EPA 608 may need to have pH adjusted upon receipt.

7.3 Solid samples are collected in 250mL wide mouth glass containers with Teflon-lined

lids. Solid samples are not chemically preserved but must be maintained at 4±2°C. Solid samples must be extracted within 14 days of collection.

7.4 Extracts from solids or liquids must also be maintained at 4±2°C and analyzed within 40 days of preparation.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 TYPICAL GAS CHROMATOGRAPHIC CONDITIONS

Carrier gas (He):	1.5mL/min (constant flow mode 9.3psi)
Make-up gas (N_2):	20-40mL/min
Injector temperature:	205°C
1µl injection:	splitless mode (purge off)
Purge:	on, 0.75min
Detector temperature:	325°C

Oven Temperature Program:

Initial temperature:	110°C, hold 0.5min
Oven ramp 1:	20°C/min to 140°C
Oven ramp 2:	11°C/min to 240°C, hold 4.09min
Oven ramp 3:	20°C/min to 300°C ,hold 8.00min

8.2 ROUTINE MAINTENANCE/ENDRIN AND 4,4'-DDT BREAKDOWN CHECK

Routine injector maintenance is performed, to limit and control both endrin and DDT breakdown, approximately once per 24 hours of system use. This includes septum replacement, liner (clean or replace), new deactivated glass wool, cut column (approximately 4cm), clean injection port and rinse of the gold seal.

Endrin and 4,4'-DDT breakdown must be checked and found acceptable before calibration or sample analyses may proceed. This check of system inertness must be performed at the start of any sequence. A working standard containing endrin and 4,4'-DDT at 100ng/mL and 200ng/mL, respectively, in hexane is injected. The peak areas of endrin, endrin aldehyde, endrin ketone, 4,4'-DDT, 4,4'-DDE and 4,4'DDD are integrated. Endrin and 4,4'-DDT breakdown as a percentage are calculated using the equations below:

$$endrin\ breakdown\,(\%) = (100)\left[\frac{peak\ area\ (endrin\ aldehyde + endrin\ ketone)}{peak\ area\ (endrin + endrin\ aldehyde + endrin\ ketone)}\right]$$

$$4,4'\text{–}DDT\ breakdown\,(\%) = (100)\left[\frac{peak\ area\ (4,4'\text{–}DDD + 4,4'\text{–}DDE)}{peak\ area\ (4,4'\text{–}DDT + 4,4'\text{–}DDD + 4,4'\text{–}DDE)}\right]$$

If the calculated breakdown of either endrin or 4,4'-DDT exceeds 15% on either

column, corrective maintenance must be done before the analysis of any calibration standards or extracts may proceed. Endrin breakdown is typically indicative of injection port contamination. Other preventive maintenance such as clipping the guard column or analytical columns may be needed to bring the breakdown within criteria. Further injection port maintenance, such as cleaning the entire port and gas feeder lines, may also be needed.

ECD detector leak checks are performed semi-annually per the SOP 016.

8.3 INITIAL CALIBRATION

8.3.1 Prepare calibration standards as suitable for the requirements of the samples to be analyzed. A typical calibration sequence and preparation steps are shown below in Table 1. A one-point calibration at or below the report level for toxaphene and technical chlordane is performed unless these multi-component compounds are expected to be present in extracts. The concentrations of the single-point calibration for toxaphene and technical chlordane are typically 250 and 50ng/mL, respectively. When toxaphene or technical chlordane is known or expected to be present, a five-point calibration is performed to more accurately quantify these compounds. A typical calibration range for the single component pesticides is 5ng/mL to 75ng/mL, with the exception of methoxychlor at 15 to 375ng/mL. Calibration standards are prepared with surrogates at similar levels to target analytes. When toxaphene or technical chlordane curves are needed to quantify these compounds, a similar approach to that shown below for single component initial calibration is used.

TABLE 1
CALIBRATION STANDARDS

Working Standard	Hexane (µL)	Intermediate Std (or std specified) (µL)	Standard Concentration (ng/mL)
Breakdown Check	1000	250	DDT 0.2 Endrin 0.1
Toxaphene	975	25	250
Technical Chlordane	975	25	50
40% Standard	750	500	100
Single Component ICAL Level 7	250	750 of 40% std	75
Single Component ICAL Level 6	750	250	62.5
Single Component ICAL Level 5	1000	250	50
Single Component ICAL Level 4	500	500 of ICAL 7	37.5
Single Component ICAL Level 3	1000	250 of 40% Std	20
Single Component ICAL Level 2	500	500 of ICAL 3	10
Single Component ICAL Level 1	750	250 of ICAL 3	5
Single Component 2nd source ICV	varied over time	varied over time	typically 37.5

8.3.2 Note that because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not

been used for a day or more. Therefore, the GC column may need to be primed by injecting a pesticide standard mixture at high concentration prior to calibration. The total chlorinated organic concentration of the solution should be approximately 5-10 ug/mL. Solvent blanks are typically injected following the priming in order to avoid contamination of later injections due to the high concentration levels of the priming standard.

8.3.3 Inject 1µL of calibration standard under the GC conditions listed previously. Each data file quantitation is accomplished via the external standard method of quantitation. Analyte Calibration Factors (CFs) are calculated as follows:

$$CF = \frac{\text{integrated peak area of analyte}}{\text{analyte concentration}}$$

Since each CF represents the slope of the line between the response for that standard and the origin, then if the observed deviation between the CF's is constant (i.e., ≤20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample content.

Relative Standard Deviation (RSD) is calculated as:

$$RSD\,(\%) = \left(\frac{\text{standard deviation of the analyte's response factors}}{\text{mean response factor}}\right)(100)$$

When %RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be $\geq$0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of 6 calibration points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration.

The type of curve fit applied should be chosen to best represent the data.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

If regression criteria cannot be met, a new initial calibration must be performed.

8.4 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is run after calibration. The concentration of the ICV should be different from that of the continuing calibration verification (CCV) and varied over time. The acceptance criteria for the ICV are identical to those of CCV (described below). If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.5 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end, of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum,

and some clients' LIMS program specifications may require more frequent CCV analysis. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

Calibration is verified when all compounds are ≤20%D, when calculated as shown below:

$$\%D = \left[\frac{(\text{calculated concentration}) - (\text{expected concentration})}{\text{expected concentration}} \right] (100)$$

If a compound shows *elevated* response (> 20%D) and is not detected in any samples associated with the CCV, re-analysis of those samples are not necessary. If a compound shows *low* response (> 20%D) and was not detected in the samples associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If any CCV does not meet acceptance criteria, analyses should be halted and corrective action taken. Reanalyze the CCV. If the CCV still fails, the instrument must be recalibrated and all samples injected since the last compliant CCV must be reanalyzed.

8.6 RETENTION TIME WINDOWS

Retention Time Windows (RTWs) are established by analyzing replicates (typically three injections) of a mid-level standard containing all single and multi-component analytes, non-consecutively, over a 72-hour period each time a new column is installed. The standard deviation of these analyses is calculated based on the absolute retention time yielded for each component. Each component's RTW is defined as the mean retention time ±3σ such that the Upper Limit = +3σ and the Lower Limit = -3σ.

RTWs should be centered on the midpoint standard if applied to samples run immediately after an initial calibration, or centered on the beginning CCV when samples are not directly preceded by an initial calibration. Sample matrices may cause drift that requires further Analyst interpretation. In the chromatography data system, RTWs and integration parameters should be set to err on the side of false positives, so target compounds are not missed by the data system. See Appendix I for chromatographic interpretation examples.

8.7 SAMPLE ANALYSIS, CALCULATIONS AND REPORTING

A constant volume, generally 1µL of each standard, blank, QC or field sample extract is directly injected into the GC via the automated injector. Sample extracts are diluted to maintain response within the linear range when necessary. All prepared extracts contain the surrogate (see Section 9). Extract concentration and sample concentration are determined as discussed below.

8.7.1 Note that ALS employs a sample splitter that facilitates simultaneous dual column injections; therefore, both columns are calibrated in the same manner and either column may serve as the column for quantitation.

8.7.2 The tentative identification of an analyte occurs when a peak from a concentrated sample extract falls within the RTW of one column. If the retention time of the analyte falls within its RTW on the second column, the analyte's presence has been confirmed. Quantitation is calculated from both column responses and the best result is reported. The analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the primary quantitation column for each analyte detected and reported. If results from both columns are comparable, the higher concentration is reported.

8.7.3 Multi-response compounds (e.g., toxaphene, chlordane) are identified through pattern recognition. Because samples may contain more than one multi-component analyte of interest and/or contain significantly weathered compounds, analyst expertise is crucial to the identification and quantitation of multi-response target compounds.

For multi-component analytes, four to eight peaks are used for identification and quantitation depending upon how many peaks are clearly defined. The same selected peaks ***must*** be consistently used for quantitation between the standard and sample set.

8.7.4 Extract concentration is calculated using the following equation:

$$\text{extract conc. (ng/mL)} = \frac{(\text{area} - \text{intercept})}{\text{slope}}$$

8.7.5 Sample concentration is calculated using the following equations:

$$\text{liquid sample conc. (ng/mL)} = \frac{(\text{extract conc.})(\text{extract volume})(\text{D})}{\text{sample volume}}$$

where:

extract conc. = ng/mL
extract vol. = mL
sample volume = mL
D = dilution factor (if applicable)

$$\text{solid sample conc. (ng/g)} = \frac{(\text{extract conc.})(\text{extract volume})(\text{D})}{\text{dry weight of sample}}$$

where:

extract conc. = ng/mL
extract vol. = mL

D = dilution factor (if applicable)

dry weight of sample = g and is the product of the weight extracted multiplied by the fraction solids in the sample.

8.7.6 If the dual column results for any analyte exceed 40% Relative Percent Difference (RPD; see calculation below), then it is necessary to investigate possible high bias of the greatest result by checking the chromatogram for apparent interferences and to check both results for appropriate integration. If no interference is evident, the highest result that is properly integrated and quantitated is reported. The elevated RPD is also discussed in the data package narrative or flagged in accord with the LIMS program specification.

$$RPD = \left[\frac{(concentration\ sample - concentration\ duplicate)}{1/2(concentration\ sample + concentration\ duplicate)} \right](100)$$

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

For this method, an analysis batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specification for additional or alternative requirements.

9.2 BLANKS

Method blanks are aliquots of matrix (i.e., organic-free water for liquids analyses; Ottawa sand for solids analyses) that have been prepared and analyzed in the same manner as the associated field samples. To be acceptable, concentrations of analytes of interest detected (if any) in the MB must be below the analyte reporting limit, or as otherwise specified in the LIMS program specification. If this criterion is not met, analyses should be halted and the source of the contamination found and corrected.

A reagent or instrument blank is an injection of solvent analyzed to demonstrate that the analytical system is free from contamination. These blanks are typically analyzed following extremely contaminated samples.

9.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the analytical system. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this analysis, either a field sample containing target compound contamination may be analyzed in duplicate (DUP), or the laboratory control sample (LCSD) or matrix spike analysis (MSD) can be performed in duplicate. The results of the duplicate analyses are evaluated in terms of RPD (calculation shown previously). See QC Table for evaluation criteria.

9.5 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. See QC Table for evaluation criteria. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%R = \frac{A_{found} - A_{sample}}{A_{target}} \text{ X } 100$$

where:

A_{found} = Calculated analyte concentration in the MS or MSD sample

A_{sample} = Calculated analyte concentration in the unspiked field sample

A_{target} = The target (anticipated) concentration of the added analyte spike

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation will be made in the data package narrative.

9.6 SURROGATE RECOVERY AND ACCEPTANCE CRITERIA

The two surrogates (SUR) used for this procedure are suggested in SW8081A,B. Both surrogates are added to all field and quality control samples prior to extraction; recovery is calculated per the recovery formula shown previously for the LCS. The two surrogates used were selected because they respond in a similar manner as the target compounds respond at the detector. Additionally these surrogates are not similar enough chemically to the target compounds to co-elute with the single component targets, nor do they suffer interferences from the multiple component targets. Tetrachloro-m-xylene (TCMX) elutes before any of the target compounds, and decachloro-biphenyl (DCB) elutes after all of the target compounds. However,

because the surrogates are not deuterated analogs of targets as in GC/MS methods, they are not extracted with exactly the same efficiencies as the target compounds. Therefore, surrogate recovery problems may not be representative of target analyte recoveries.

Heavy co-extractive non-target compounds generally do not interfere with TCMX; light co-extractive non-target compounds generally do not interfere with DCB. However, some samples may produce matrix effects that cause surrogate recovery to be high or low; because of high concentrations of target and/or non-target compounds, quantification of the surrogates may even be precluded in some samples. Muddy aqueous samples, for example, generally adsorb DCB after spiking and limit recovery to a few percent. High concentrations of heavy hydrocarbons in soils oftentimes have a similar matrix effect on DCB recovery.

The extraction process itself can have an effect on surrogate recovery. An example of this process-caused effect is use of Method SW3520 for extraction of aqueous samples and associated "low" recoveries of DCB. DCB is a heavy molecule and very hydrophobic. When spiked into water, this compound tends to rapidly adsorb to particulates at the liquid-liquid interface and thus exhibits a low recovery.

For the reasons listed above, ALS does not view the evaluation of surrogate recovery in this procedure to be a straightforward process. Therefore, the following guidance for evaluating surrogate recovery is observed:

- Evaluate and report the recovery of both surrogates. When one or both surrogates are within laboratory control limits, the process is considered to be in control and no further action is taken (unless additional measures are stipulated in the LIMS program specification.

- When, due to elevated target concentrations, an extract requires a dilution of greater than 5X, the surrogate recoveries are not controlled; no further action is taken.

- When both surrogates are outside of laboratory control limits (or other limits specified in the LIMS program specification), the extract is re-injected to assure that instrument error was not the cause. If after re-injection the recoveries of both surrogates remain out of control, then re-extraction and re-analysis may be performed as directed by the client. A non-conformance report (NCR; SOP 928) to document the problems is required.

This process of evaluating surrogate recovery is based on several methods and guidance documents and has evolved in particular from Method SW8080 guidance as well as from the National Functional Guidelines for Data Review.

9.7 A METHOD DETECTION LIMIT (MDL) STUDY

A method detection limit is determined in accordance with ALS SOP 329

10. DEVIATIONS FROM THE METHODS

10.1 This SOP meets the requirements of Method SW8081A and B. Note that ALS analyzes a breakdown check every 24hrs, not every 12hrs.

10.2 Deviations from Method EPA 608

Because samples from several sites are usually batched together, only one spiking level is used for each compound. It is impractical to match each compound's spike amount with the amount of the compound in the samples chosen for spiking and also matching the spike amount to the appropriate regulatory level for each compound. This difference must be stated in the data package narrative that accompanies each batch of samples.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 3rd edition, Final Update III, "Method 8081A", Revision 2, December 1996.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8081B", Revision 2, February 2007.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12.4 40 CFR, Part 136, Appendix A, 7-1-99 Edition, "Method 608".

Analytical Method: SW8081A,B or EPA 608	**Parameter:** Organochlorine Pesticides		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point; all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, use mean RFs and CFs to quantitate. If RSD >20% , calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r^2) is ≥ 0.990 or calculate quadratic regression (minimum of six points required); use for quantitation if COD (r^2) ≥0.990	Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Endrin and DDT Breakdown Check; approx. midpoint of calibration	Run every 12 hour shift prior to calibration verification	If ≤15% analyses may proceed.	Perform GC maintenance (e.g., change liner, clip column, inject primer, etc.); recalibrate.
Initial Calibration Verification (ICV); second source	After each initial calibration	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV); first source	Brackets each set of 20 field sample analyses (standard practice is every 10 samples injections)	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re-analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.
Retention Time Window (RTW)	Whenever a new column is installed; based on at least 3 injections throughout a 72-hour period.	Column and compound specific. Window is ±3x the standard deviation of the 3-injection average for the respective column. Note that the ICV and CCV analyses are also used to monitor RTW shift.	If SD=zero, then either do additional injections or use a default SD of 0.01 minutes. Experience of analyst weighs heavily in interpretation of chromatograms (refer also to RT Shift).

Analytical Method: SW8081A,B or EPA 608	**Parameter:** Organochlorine Pesticides		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time (RT) Shift	Each CCV; RT of analytes evaluated against the ICAL	Column and compound specific	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate. Evaluate data based on a comparison with other standards run during the analytical sequence; consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question.
Method Blank (MB)	1 per each preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact PM for sample disposition.
Blank Spike; BS (Laboratory Control Sample; LCS)	1 per batch of ≤20 samples of like matrix	See laboratory limits; recoveries for the spiked compounds must be within the laboratory limits or other limits as specified in the LIMS program specification.	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria. - if the samples are outside the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.
Matrix Spike (MS)	1 per batch of samples, not to exceed 20 samples of a given matrix	See laboratory limits; recoveries for the spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Duplicate	1 per batch of samples, not to exceed 20 samples of a given matrix.	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for

Analytical Method: SW8081A,B or EPA 608	**Parameter:** Organochlorine Pesticides		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
		advisory limits.	documentable errors (e.g., calculations and spike preparation). If no errors are found and, if analyzed, LCSD RPD is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Surrogate Spike	All extractions including field and laboratory QC samples.	See laboratory limits; recoveries should be within current limits for one or both surrogates; alternative criteria as defined in the LIMS program specifications may apply.	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with both surrogate recoveries outside the recovery limits, with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery. - if both surrogate recoveries in the associated MB are not within limits, and the samples are within the holding time, then re-extract and reanalyze all associated samples. - if samples are outside the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.
Method Detection Limit (MDL) Study	As needed and at minimum, annually	Concentrations for the MDL study shall be at a level lower than that of the reporting limit	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If `criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

Appendix I
Chlorinated Pesticide Data Interpretation Examples

- The presence of multi-component target analytes, such as toxaphene and/or chlordane, or non-target analytes such as Aroclors, in a sample may interfere with proper identification of target analytes by retention time and may cause false positives on both columns. Typically, the presence of either Aroclor 1254 or Aroclor 1260 result in peaks on both columns that are RT matches (potential false positives) for 4,4'-DDT. Aroclor 1254 also results in peaks on both columns that are RT matches for dieldrin. When either of these Aroclors is observed or suspected, the analyst may need to inject the appropriate Aroclor standard in order to better interpret the data with respect to presence or absence of single component pesticides.

- The retention times of surrogates in each extract injected provide useful information about the effects of sample matrices on retention times during each analysis. The surrogates used for this procedure have very early and very late retention times compared to the target analytes. If the observed TCMX and DCB retention times in sample analysis are the same as in bracketing CCVs, then it can be assumed that there is little or no matrix effect on the RT of target analytes. If, for example, DCB is 0.01 minutes later in a sample analysis than in surrounding calibration verifications, then the analyst may assume that late-eluting targets may also be eluting slightly later, and use this observation to assist with peak identification.

- Retention time difference between columns can also be used as an aid to identification. If both surrogates in a sample have the equivalent RTs to nearby standards and, for example, heptachlor is 0.03 minutes early on column 1 and 0.02 minutes late on column 2, then the analyst may decide that heptachlor is not confirmed as present and to not report heptachlor based on the relative retention time differences.

- The retention times and peak shapes of analytes in matrix spikes may be used as an aid to the analyst in identification in similar matrix samples. If endrin, for example, is identified on both columns and is 0.03 minutes later in the MS/MSD than in nearby calibration verifications, and this is consistent with other late-eluting peak RTs in the MS/MSD, then the analyst should use this information in evaluating and identifying targets in samples of similar matrix. If a sample has a peak that is within the RT window for example at 12.01 (column 1) and 13.07 (column 2), but the matrix spikes have peaks at 11.99 and 13.04, and both show evidence of a shoulder on the main peak, and the shoulder is on the later eluting side of the main peak, the analyst may document that the RT match appears to be a false positive match and not report the target as confirmed.

- The presence of alpha- and gamma-chlordane in a sample is a primary indicator of the presence of chlordane even if little or no pattern can be observed due to low concentrations of the technical mixture. ALS does not analyze for individual components of toxaphene, so pattern recognition of toxaphene is the only mechanism of identifying this multi-component analyte. Weathered chlordane or toxaphene can produce patterns that are not good matches to fresh standards. Also, several producers manufactured multi-component pesticides, so patterns may not be identical between sources. The presence of moderate to high concentrations of single component pesticides such as 4,4'-DDT and its degradation products can also make pattern recognition of toxaphene or chlordane more difficult in real samples than in standards. Analyst experience is essential in properly identifying and quantifying such complex mixtures. For multi-component analytes, four to eight peaks are used for identification/quantitation, depending upon how many peaks are clearly defined. The same selected peaks must be consistently used for quantitation between the standard and sample set.

ALS Standard Operating Procedure

DOCUMENT TITLE:	EXTRACTABLE PETROLEUM HYDROCARBONS BY GAS CHROMATOGRAPHY (TEPH, DRO)
REFERENCED METHOD:	SW 8015D AND CAL-LUFT
SOP ID:	406
REV. NUMBER:	18
EFFECTIVE DATE:	OCTOBER 31, 2014

ALS
STANDARD OPERATING PROCEDURE 406 REVISION 18

TITLE: EXTRACTABLE PETROLEUM HYDROCARBONS ANALYSIS BY GAS CHROMATOGRAPHY (TEPH, DRO)

FORMS: APPENDIX A

PRIMARY AUTHOR: _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the determination of extractable petroleum hydrocarbons in aqueous and solid matrices, using analysis by gas chromatograph (GC) with flame ionization detection (FID). This basic GC-FID technique is comparable to analysis protocols such as SW-846 Method SW8015D and Cal LUFT.

2. SUMMARY

Concentrated sample extracts prepared via SOP 603 (e.g., SW8015) and SOP 626 (e.g., SW3510, Cal LUFT), are injected into a GC-FID chromatographic system that is temperature programmed. Detector responses are recorded by a data acquisition system to facilitate processing of data. The total peak area from the designated petroleum hydrocarbon retention time window (RTW) is measured using baseline-to-baseline integration. Quantitation is accomplished using the external standard method of quantitation. Performance of a surrogate is also monitored. Reporting limits are set at or above the concentration of the lowest standard in the initial calibration curve. Typically, both the amount and type of petroleum hydrocarbon observed are reported. Unless otherwise directed by LIMs program specifications, the retention time window for Diesel Range Organics is set to quantitate from C10 to C28. If analyzing for Motor Oil Range Organics, the range is from C20 to C32 (and the range for DRO is set from C10 to C20).

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this method. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 Final review and sign-off of the data are performed by the Department Supervisor or designee. Initialing and dating the file indicates that this review for precision accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.4 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the handling or analysis of the samples. Any discrepancies found must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Interference from phthalate esters can be minimized by using plastic-free solvent containers and scrupulously cleaned glassware (SOP 334) that has been kiln-baked or solvent-rinsed prior to use. Use of low phthalate gloves, such as nitrile, may also be important. These practices are important both in the field and in the laboratory.

4.2 Because the chromatographic conditions employed for extractable petroleum hydrocarbon analysis can result in significant column bleed and resultant variations in the baseline, it is appropriate to perform a subtraction of the column bleed from the area of the petroleum hydrocarbon chromatogram. A column comparison is performed on a dry run (no solvent injected, temperature program executed) to determine the area caused by column bleed. This area is subsequently stored by the GC or data system and used to correct the baseline for all associated standards and samples. This procedure is acceptable provided that the column bleed is consistent throughout the run. A consistent dry run or blank baseline confirms proper column compensation.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH (GC), AUTOSAMPLER, DETECTORS

Hewlett Packard (HP) 7673 Automated Injection System; HP 5890 Series II GC equipped with a flame ionization detector (FID), or equivalents

NOTE: An FID must be used because FID response is essentially proportional to the number of carbons and can, therefore, provide a useful total area for evaluation of total hydrocarbon concentration; other detectors will not produce adequate quantitative results.

5.2 COLUMN - Equivalent columns may also be used

J&W Capillary Column:	DB-5.625	# 200-0056*	30m x 0.250mm ID, 0.5µm film thickness

*J&W columns are available through Agilent, part numbers are Agilent numbers

5.3 DATA SYSTEM

Agilent EzChrome Elite, or equivalent

5.4 GASES - **Use only high purity gases!**

Helium - carrier gas
Hydrogen - to supply FID
Air - to supply FID

5.5 MEASURING DEVICES

5.5.1 Microsyringes, Hamilton Precision™ or equivalent, 1µL-1.0mL sizes

5.5.2 Top loading balance, capable of weighing to ±0.01g

5.5.3 Volumetric flasks, Class A with ground glass stoppers, 10-500mL sizes

5.6 GC CONSUMABLES

- Vials and caps, National Scientific #C4011-1 and # C4000-51B or equivalents
- Inlet seal, dual Vespel ring, 0.8mm, Restek #21243 or equivalent
- Septa, 11mm, Restek #20365 or equivalent
- O-ring, graphite, 6.5mm, Restek #20299 or equivalent

- Liner, splitless, 4mm ID, Restek #20799-214.5 or equivalent
- Glass wool, deactivated, Restek #20789 or equivalent
- Gold seal, Restek #21306 or equivalent
- Vespel/graphite ferrule (detector), Restek #20221 or equivalent
- Compact Vespel/graphite ferrule, Restek #20249 or #20264 or equivalent
- Graphite ferrules, various sizes

6. REAGENTS

6.1 SOLVENTS - **Only chromatography grade or higher quality solvents may be used**

n-hexane, Burdick & Jackson #216-4 or equivalent

methanol, Burdick & Jackson #230-4 or equivalent

methylene chloride, Burdick & Jackson #299-4 or equivalent

6.2 STANDARDS

NOTE: All standards are maintained per ALS SOP 300. Standards may be replaced sooner than required by SOP 300 if deterioration is suspected or indicated.

6.2.1 Stock standards are generally acquired through locally available commercial sources. Two independent sources (first, second) of target analyte stock standards are needed. To set correct integration windows, ALS uses a TRPH Standard (Ultra-Scientific TRPH Florida Standard or equivalent) that contains even numbered straight-chain alkanes C_8-C_{40}. Alternate standards may be used if required by the LIMS program specification.

A non-target analyte surrogate stock standard is also purchased. The surrogate used in this procedure is o-terphenyl.

Unopened stock standards are valid until the manufacturer's expiration date, and may be stored at room temperature in flame-sealed ampules, if recommended by the manufacturer. After ampules are opened, stock standards are transferred to Teflon™-lined septum seal vials for storage.

6.2.2 Intermediate standards are made by diluting an appropriate aliquot of stock standard to a specific volume using the appropriate solvent (hexane or dichloromethane), and are stored in Teflon™-sealed vials. First source target

analyte materials are used to create calibration, continuing calibration verification (CCV) and quality control (QC) sample spike standards (used by the Organics Extraction Group). Second source target analyte materials are used to create the initial calibration verification (ICV) solution.

6.2.3 Working standards are made by diluting an appropriate aliquot of intermediate standard to a specific volume using the appropriate solvent, and are stored in Teflon™-sealed vials. These working calibration/check standards (ICAL, ICV, CCV) are prepared on the day of use and documented in the analytical run log (Form 531). They must contain all target analytes and the surrogate. A detailed description of the concentration of the calibration standards and how they are used can be found in Section 8 of this SOP.

6.2.4 Section 9 of this SOP gives definitions and uses of surrogates and QC (i.e., LCS/LCSD, MS/MSD) samples.

6.2.5 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials, and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the Quality Assurance Office.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Aqueous samples are collected in 40 mL VOA Vials with screw-top caps equipped with Teflon™ liners. Aqueous samples are usually preserved in the field immediately after sampling with the addition of enough hydrochloric acid (HCl) to adjust to pH<2. The samples must be maintained chilled (4±2°C).

7.3 Soil samples are collected in wide-mouth glass containers with Teflon™-lined lids. Samples are not chemically preserved and must be maintained chilled (4±2°C).

7.4 The holding time to extraction for aqueous extractable petroleum hydrocarbon samples is 7 days (unpreserved) or 14 days (preserved with HCl) from collection. The hold time to extraction for solid matrix extractable petroleum hydrocarbon samples is 14 days from collection. Consult the LIMS program specification as other hold times may be specified.

7.5 Extracts, from liquid or solid samples, must be maintained chilled (4±2°C) and analyzed within 40 days of preparation.

8. PROCEDURES

8.1 TYPICAL GC CONDITIONS

Note that conditions may be altered to improve resolution, or simplified if a single product, such as motor oil, is to be analyzed.

Carrier Gas Flow Rate:	3-5mL/min
FID H_2 Flow Rate:	30mL/min
FID Air Flow Rate:	300mL/min
Injector Temperature:	320°C
Initial Oven Temperature:	60°C for 3min
Oven Ramp:	17°C/min to 320°C
Hold:	320°C for 15min
FID Temperature:	320°C

8.2 CHROMATOGRAPHIC MAINTENANCE

8.2.1 Reagent blanks are an injection of solvent analyzed to show that the analytical system is free from contamination. They may be injected following samples of unusually high concentration to check the status of the analytical system and to facilitate re-equilibration of the system. Reagent blank results should be below the RL.

8.2.2 Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Oxidized columns will exhibit baselines that rise rapidly during temperature programming.

8.2.3 Peak tailing for all components will be exacerbated by a contaminated injection port.

8.2.4 If the instrument has sat idle for a period of time, it may be equilibrated with one or more injections of solvent prior to initiation of the acquisition sequence.

8.3 INITIAL CALIBRATION

8.3.1 A minimum of five concentration levels are required, typically six standards are prepared, as shown below:

Level	Petroleum Hydrocarbon Concentration (µg/mL)	Surrogate Concentration (µg/mL)
6	5000	250
5	2000	100
4	500	25
3	100	5
2	25	1.25
1	16.5	0.825
ICV	400	(na)
CCV	500	25

8.3.2 Typically 1 to 2 µL of standard or field or QC sample extract is injected by automated injector for analysis. TEPH is identified by pattern recognition.

8.3.3 For each data file, quantitation is accomplished via the external standard method of quantitation using baseline-to-baseline integration across the hydrocarbon range of interest. The surrogate area is subtracted from the total area integrated in order to obtain the area associated with the TEPH fuel product.

8.3.4 A calibration factor (CF) for each standard is calculated as follows:

$$CF = \frac{\text{TEPH Area Total}}{\text{Concentration of TEPH Injected (mg/mL)}}$$

Since each CF represents the slope of the line between the response for that standard and the origin, then if the observed deviation between the CF's is constant (i.e., ≤20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample concentrations. Percent Relative Standard Deviation (%RSD) is calculated as:

$$\text{RSD (\%)} = \frac{\text{Standard Deviation (SD)}}{\text{Average (mean) CF}} \times 100$$

When %RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be $\geq$0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of 6 calibration points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration.

The type of curve fit applied should be chosen to best represent the data.

If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

If regression criteria cannot be met, a new initial calibration must be performed.

8.4 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is analyzed after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time. The acceptance criteria for the ICV are identical to those of CCV (described below). If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated

8.5 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum, and some clients' LIMS program specifications may require more frequent analyses of CCV's. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

The percent difference (%D, drift) must be calculated for each CCV (see equation below):

$$\%D = \left[\frac{(\text{calculate d concentrat ion}) - (\text{expected concentrat ion})}{\text{expected concentrat ion}} \right] (100)$$

Calibration is verified when target compound recovery is ≤20%D of the expected value.

NOTE: If target recovery shows *elevated* response (> 20%D) and is not detected in any samples associated with the CCV, re-analyses of those samples are not necessary. If target recovery shows *low* response (> 20%D) and was not detected in the samples associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If a CCV does not meet acceptance criteria, corrective action should be taken and the CCV may be reanalyzed. If the re-analyzed CCV still fails, the instrument must be recalibrated.

NOTE: When analyzing samples by the Cal LUFT method, for California compliance reporting, the CCV criteria of ±10%D **must** be observed (electronically controlled via the specific test code used in association with ALS's LIMS program specification system).

8.6 RETENTION TIME WINDOWS

8.6.1 Evaluate bracketing standards and set the retention time window (RTW) to include the designated petroleum hydrocarbon pattern. The standard contains the even numbered aliphatic hydrocarbons from C_8 –C_{40}. The default window is typically set from C_{10} through C28, but can be adjusted to meet project requirements.

8.6.2 RTWs are used to define the integration envelope for quantifying various products in samples. Peak area integration will begin immediately prior to elution of the first chromatographic peak of interest and will stop after the last peak of interest.

8.6.3 The experience of the analyst weighs heavily in interpretation of the chromatogram. Environmental samples may contain more than one type of product, and loss of light end components may indicate weathering or poor extraction techniques. Additionally, the baseline-to-baseline integration used with this analysis can make it prone to false positives in the case of a wandering or unstable baseline. The Analyst's judgement is crucial in determining whether a low-level (>mdl, <RL) quantitation is due to actual (i.e. extracted) TEPH present or is in fact instrument-related. Analyst's notes and flagging on quant reports provide further information.

8.7 SAMPLE ANALYSIS, IDENTIFICATION, CALCULATIONS, REPORTING

8.7.1 Prior to initiating acquisition, ensure that that there is an adequate supply of gases and rinse solvents to complete the run.

8.7.2 When necessary, sample extracts are diluted to maintain response within the linear range.

8.7.3 If the average CF is used for quantitation, the sample concentration is calculated using the following equation:

$$\text{Concentration}_{(\text{ug/L, ug/kg})} = \frac{(A_x)(V_t)(DF)}{(\text{Average CF})(V_s \text{ or } W_s)}$$

where:

A_x = analyte response (area units)

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

Average CF = average calibration factor (area/concentration in mg/mL)

V_s or W_s = volume or weight of sample extracted (L or kg)

8.7.4 Sample concentration, in ppm (mg/L or mg/kg), is calculated using the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)(V_t)(DF)}{m(V_s \text{ or } W_s)}$$

where:

x = concentration of the analyte (TEPH, ppm)
y = intercept for analyte instrument response (area)
b = calculated intercept (area)
m = calculated slope of the line (area/concentration in mg/mL)
V_t = total volume of concentrated extract (mL)
DF = Dilution Factor (if applicable); if no dilution, then DF = 1
V_s or W_s = volume or weight of sample extracted (L or kg)

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All QC samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specification for additional or alternative requirements.

9.2 BLANKS

MBs are aliquots of matrix (i.e., organic-free water for liquids, Ottawa sand for solids) that have been prepared and analyzed in the same manner as the associated field samples. MBs are analyzed to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Concentrations of target analytes, if any, must be less than the RL, or as otherwise prescribed in the LIMS program specification. If this criterion is not achieved, then analyses must be halted and the source of the contamination found and corrected.

9.3 LABORATORY CONTROL SAMPLE

The LCS is analyzed to measure the accuracy of the analytical system. The LCS is similar to the matrix spike in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample and/or matrix spike sample is performed in duplicate (LCSD, MSD). The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\ (\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

MSs consist of field samples into which known concentrations of target analytes are spiked and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. See QC Table for evaluation criteria. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%\text{R} = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right)(100)$$

NOTE: Typically, one MS/MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate the MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation is made in the data package narrative.

9.6 SURROGATE RECOVERY

The %R of the surrogate is calculated (see equation shown for LCS recovery above) for all field and QC samples. See QC Table for evaluation criteria.

9.7 METHOD DETECTION LIMIT STUDY

Method detection limits for this analysis are established according to the guidance provided by SOP329..

10. DEVIATIONS FROM METHOD

This SOP meets the requirements of SW8015D; there are no known deviations from the method. Cal LUFT method criteria are also met with the following exception: Cal LUFT specifies a %D criteria of ±10% for daily calibration verification. ALS defaults to the criteria listed in SW8015D of ±20% D, unless client or regulatory contractual needs dictate otherwise. ALS notes that the SW-846 suggested carbon range for diesel (C_{10} to C_{28}) may be modified to meet our client's requirements.

11. SAFETY HAZARDS AND WASTE

11.1 SAFETY HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans and Job Safety Analysis (Appendix B):

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 EPA SW-846, Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, 3rd edition, Final Update III, "Method 8015D", June 2003.

12.2 EPA SW-846, Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, 3rd edition, Final Update III, "Method 8000C", Revision 3. March 2003.

12.3 California LUFT Field Manual, October 1989 update.

12.4 EPA SW-846, Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, 3rd edition, Final Update III, "Method 3510C", Revision 3, December 1996.

12.5 EPA SW-846, Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, 3rd edition, Final Update III, "Method 3540C", Revision 3, December 1996.

12.6 EPA SW-846, Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, 3rd edition, Final Update III, "Method 3550B", Revision 2, December 1996.

Analytical Method: Extractable Petroleum Hydrocarbons by GC-FID	**Parameter:** Total Extractable Petroleum Hydrocarbons Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL); minimum 5-point, all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, may use mean RF to quantitate Calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r^2) ≥0.99 or Calculate quadratic regression (minimum of six points required); use for quantitation if COD ≥0.99	Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Initial Calibration Verification (ICV); conc. not equal conc. to midpoint of calibration curve; second source	After each ICAL	≤20%D of each compound	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.
Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Run at start of sequence if ICAL not performed; brackets each set of 20 sample analyses; more frequent analyses recommended; standard practice is CCV every 10 samples.	≤20%D of each compound **NOTE:** Cal LUFT method requirements state that analyses may continue only when CCV is ±10%D. Check the LIMS Project Specification to ensure that appropriate criteria are applied.	Evaluate/correct instrument malfunction as needed (e.g., rinse or change liner, remove some of the guard column); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. - Samples analyzed before and after a failed CCV (bracketing with acceptable calibration fails) must be reanalyzed. - If a failed CCV for an autosampler analysis returns to acceptable

Analytical Method: Extractable Petroleum Hydrocarbons by GC-FID	**Parameter:** Total Extractable Petroleum Hydrocarbons Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			calibration later in the sequence, samples following the acceptable CCV (bracketed by acceptable CCVs) will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition..
Retention Time Window (RTW); set to include the designated extractable petroleum hydrocarbon reference standard	Whenever a new column is installed or if a new extractable petroleum hydrocarbon range is required	Brackets appropriate hydrocarbon elution range Note that the ICV and CCV analyses are also used to monitor RT drift	Perform system maintenance to correct drift. Experience of analyst weighs heavily in interpretation of chromatograms.
Retention Time Shift; RT of pattern in CCV is evaluated against the midpoint of the ICAL or the preceding CCV	Each CCV; RT of analytes evaluated against the ICAL or preceding CCV to ensure that the designated extractable petroleum hydrocarbon range has not significantly shifted	Instrument performance supports accurate quantitation of TEPH	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate Evaluate data based on comparison with other standards run during sequence, consider RTs for the surrogates and spiked compounds analyzed before and after the sample in question.
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective

Analytical Method: Extractable Petroleum Hydrocarbons by GC-FID	**Parameter:** Total Extractable Petroleum Hydrocarbons Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
		per other criteria as specified in the applicable LIMS program specification	action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition.
Blank Spike (BS); Laboratory Control Sample (LCS)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds must be within laboratory limits or other limits as specified in the LIMS program specification	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed) - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be

Analytical Method: Extractable Petroleum Hydrocarbons by GC-FID	**Parameter:** Total Extractable Petroleum Hydrocarbons Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			extracted outside of the holding time and the data will be submitted with appropriate narration
Matrix Spike (MS)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; see Matrix Spike information above for MSD recoveries RPDs should be within advisory limits	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.
Surrogate Spike	All field and laboratory QC samples	See laboratory limits; recoveries should be within current limits alternative criteria as defined in the LIMS program specifications may apply	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with

Analytical Method: Extractable Petroleum Hydrocarbons by GC-FID	**Parameter:** Total Extractable Petroleum Hydrocarbons Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery. - if surrogate recovery in the associated MB is not within limits and the samples are within the holding time, then re-extract and reanalyze all associated samples - if samples are beyond the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.
Method Detection Limit (MDL) Study	As needed, at minimum, annually	Concentrations for the MDL study shall be at a level lower than that of the reporting limit	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

APPENDIX A
EXAMPLE

ALS LABORATORY
DRO RUN LOG

Instrument Name _____ Date Analyzed _____ Operator_____
Hexane Lot _____ Dichloromethane Lot _____
GC Method _____ Analytical Method _____

Run Information	Comments	rr?

Note: Each page is copied and included with the run documentation; reviewed subsequently. Form 531r3.xls (5/26/09)

ALS Standard Operating Procedure

DOCUMENT TITLE:	ORGANOPHOSPHOROUS COMPOUNDS BY GAS CHROMATOGRAPHY
REFERENCED METHOD:	EPA 8141 A OR B, AND EPA 614
SOP ID:	407
REV. NUMBER:	10
EFFECTIVE DATE:	AUGUST 16, 2011

STANDARD OPERATING PROCEDURE 407 REVISION 10

TITLE: ORGANOPHOSPHOROUS COMPOUNDS BY GAS CHROMATOGRAPHY -- METHODS SW8141A or B, AND EPA 614

FORMS: NONE (instrument printout used as run log)

APPROVED BY:

TECHNICAL MANAGER_____ DATE _____

QUALITY ASSURANCE MANAGER_____ DATE _____

LABORATORY MANAGER_____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- SW8141A or B and EPA 614, are used to determine the concentrations of organophosphorous pesticides in extracts from liquid or solid matrices. Method SW8141A,B addresses liquid and solid matrices and Method EPA 614 addresses liquid matrices (municipal and industrial wastewater). Currently, ALS Laboratory Group - Fort Collins (ALS) analyzes the following compounds using this SOP:

Azinphos methyl	Merphos (including its degradation product)
Chlorpyrifos	Methyl parathion
Coumaphos	Mevinphos
Demeton (total o- and s-)	Naled
Diazinon	Phorate
Dichlorovos	Ronnel
Disulfoton	Tetrachlovinphos
Ethoprop	Tokuthion
Fensulfothion	Trichloronate
Fenthion	Sulprofos
Malathion	

Other compounds may be analyzed if successful method detection limit (MDL) and demonstration of capability (DOC) studies are performed.

2. SUMMARY

Samples are extracted and the extracts concentrated and solvent exchanged using appropriate ALS SOPs (i.e., 617, 625, 622, 607, 637). The extracts are injected into a gas chromatograph (GC) containing a sample splitter and two columns of varying selectivity (i.e., dissimilar RT elution properties). The target analytes are separated in the columns and detected by two flame photometric detectors (FPDs). This chromatography system allows tentative identification by one column and confirmation by the other column to be performed simultaneously. Quantitation is performed using the best column for each

analyte. The analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the primary quantitation column for each analyte detected and reported. If results from both columns are comparable, the highest result is reported.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the benchsheet indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work, and documentation of measures taken to remediate the data.

3.4 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.5 It is the responsibility of all personnel who work with samples involving this procedure to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Use of flame photometric detectors in phosphorous mode will minimize interferences from materials that do not contain phosphorous. Method SW8141A,B states that elemental sulfur may interfere with the determination of certain organophosphorous compounds by flame photometric gas chromatography.

4.2 If a sulfur cleanup is employed, only the tetrabutylammonium (TBA)-sulfite option should be chosen, because copper may destroy organophosphorous pesticides. The stability of each analyte must be tested to ensure that the recovery from the TBA-sulfite cleanup step is not less than 85%.

4.3 Analytical difficulties encountered for target analytes include:

4.3.1 The water solubility of dichlorvos is 10g/L at 20°C and recovery may be poor from aqueous solutions.

4.3.2 Naled is converted to dichlorvos in water or by injection on column by debromination. This reaction may also occur during sample extraction and preparation. The extent of debromination will depend upon the nature of the matrix being analyzed. The analyst must consider the potential for debromination when naled is to be determined or when dichlorovos is detected.

4.3.3 Trichlorfon can rearrange by hydrodechlorination in acidic, neutral, or basic media to form dichlorvos and hydrochloric acid. If this method is to be used for the determination of organophosphates and the presence of trichlorfon is known, the analyst should be aware of the possibility of rearrangement to dichlorvos if dichlorovos is detected.

4.3.4 Demeton (Systox) is a mixture of two compounds - O, O-diethyl O- [2-(ethylthio) ethyl] phosphorothioate (demeton-O) and O, O-diethyl S- [2-(ethylthio) ethyl] phosphorothioate (demeton-S). Two peaks are observed in demeton standards. The two peaks correspond to the two isomers.

4.3.5 Merphos (tributyl phosphorotrithioite) is readily oxidized to its phosphorotrithioate (merphos oxone). Chromatographic analysis of merphos almost always results in two peaks (unoxidized Merphos elutes first). As the relative amounts of oxidation of the sample and the standard are probably different, quantitation based on the sum of both peaks is most appropriate.

4.3.6 Many analytes will degrade on reactive sites in the chromatographic system. Analysts must ensure that injectors and splitters are free from contamination. Columns should be installed and maintained properly.

4.4 Interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that leads to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the condition of the analysis by analyzing reagent blanks with every batch of 20 or fewer field samples. Because the FPD with P filter is an element-specific detector, the probability of non-target contamination is minimal.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH (GC), AUTOSAMPLER AND DETECTORS Hewlett Packard (HP) 5890 Series II GC equipped with HP 7673A autosampler and dual FPD detectors (complete with accessories for on-column or split/splitless injection), or equivalents

5.2 CHROMATOGRAPHIC DATA ACQUISITION AND PROCESSING SYSTEM

Hewlett Packard ChemStation (Enviroquant™), or equivalent

5.3 COLUMNS - Equivalent columns may also be used

Restek Column:	RTX-1 #10139 (30m x 0.32mm ID, 0.5µm film thickness)
Restek Column:	RTX-OP Pesticides #11239 (30m x 0.32mm ID, 0.5µm film thickness)

5.4 GASES - ultra high purity (99.999%)
Helium - carrier gas
Nitrogen - make-up
Air -detector
Hydrogen -detector

5.5 MEASURING DEVICES
Syringes - 10µL-1000µL
Volumetric flasks, Class A with stoppers, 10mL-100mL

5.6 GC CONSUMABLES

- Vials - National Scientific C4011-1, or equivalent
- Caps - National Scientific C4000-51B, or equivalent
- Inlet Seals, dual Vespel ring - Restek 0.8mm #21243, or equivalent
- Septa, 11mm - Restek #20365 or equivalent
- O-ring, graphite, 6.5mm - Restek #20299, or equivalent
- O-ring, Viton - Restek #20377, or equivalent
- Liner, splitless, 4mm ID - Restek #20799-214.5, or equivalent
- Glass Wool, deactivated - Restek #20789, or equivalent
- Gold Seal - Restek #21306, or equivalent
- Universal Presstight Y - Restek #20469, or equivalent
- Vespel/Graphite Ferrule (detector) - Restek #20221, or equivalent
- Compact Vespel/Graphite Ferrule - Restek #20249, or equivalent
- Graphite Ferrules - various sizes
- Split Vent Trap (SW8081) - Agilent RDT-1023, or equivalent

6. REAGENTS AND STANDARDS

6.1 SOLVENTS - Only pesticide residue grade or equivalent may be used!
Hexane (C_6H_{14}), Burdick and Jackson #216-4, or equivalent
Methanol (CH_3OH, MeOH), Burdick and Jackson #230-4, or equivalent

6.2 STANDARDS

6.2.1 All standards are maintained per SOP 300, which is superceded by any guidance in this SOP. Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampules, if recommended by the manufacturer. Generally

after opening ampules, the standards for this procedure are stored in the freezer (-10--20°C), in PTFE-capped, or equivalent, vials. Opened stock standards and intermediate standards expire six months from opening (preparation) or per the manufacturer's expiration date (whichever is sooner). Standards may be replaced sooner if laboratory quality control (QC) analyses or other factors indicate deterioration.

6.2.2 Two independent sources of commercial stock standards are required for target analytes. These certified stock standards are purchased from suitable vendors. First source materials are used to create calibration, continuing calibration verification (CCV) and QC sample spike standards. Second source materials are used to create the initial calibration verification (ICV) solution. A (non-target analyte) surrogate stock standard is also purchased. The surrogate used for this method is triphenylphosphate. Section 9 of this SOP gives definitions and uses of surrogates and QC (i.e., LCS/LCSD, MS/MSD) samples.

An appropriate volume of stock standard is diluted (in hexane) to a specific volume to create intermediate standards (the QC sample and surrogate spike standards, used by the Organics Extraction Group, are intermediate standards). The intermediate calibration standards are further diluted to volume using an appropriate solvent to create working standards. Working standards are prepared on the day of use and documented in the analytical run log (Form 530). A detailed description of the concentration of the calibration standards and how they are used can be found in Section 8 of this SOP.

6.2.3 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Aqueous samples must be collected in amber glass containers (generally 1000mL) with Teflon™-lined lids. Samples must be maintained at 4±2°C and extracted within 7 days of collection. Additionally, samples for EPA Method 614 may need to have pH adjusted to be between 5 and 8, using sodium hydroxide or sulfuric acid solution, upon receipt.

7.3 Solid samples are collected in wide-mouth glass containers with Teflon™-lined lids. Solid samples are not chemically preserved and must be maintained at 4±2°C. Solid samples must be extracted within 14 days of collection.

7.4 Extracts, from liquid or solid samples, must be refrigerated and analyzed within 40 days of preparation.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 TYPICAL GC OPERATING CONDITIONS

Carrier gas:	Helium
Hydrogen (detector fuel gas)	75mL/min
Air (detector oxidizing gas)	100mL/min
Injection port temperature	210°C
Injection volume	3µL
Detector temperatures	275°C
Initial oven temperature	105°C, hold 1min
Purge	On, 1min
Initial oven ramp	20°C/min to 185°C, hold 5min
2nd oven ramp	2°C/min to 205°C, hold 2min
3rd ramp	20°C/min to 300°C, hold 8.25min

8.2 CHROMATOGRAPHIC MAINTENANCE

8.2.1 Dual columns are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector. Reattach the columns after cleanly cutting off at least two loops from the injection port side of the column using a capillary cutting tool or scribe. The accumulation of high boiling residues may change split ratios between dual columns and thereby change calibration factors. Clip a loop from the guard column or replace as necessary.

8.2.2 Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Polar columns (including DB-210, DB-1701 and DB-608) are more prone to oxidation. Oxidized columns will exhibit baselines that rise rapidly during temperature programming.

8.2.3 Peak tailing for all components will be exacerbated by dirty injectors, dirty guard columns, and dirty glass Y's. Components such as fensulfothion, naled, methyl azinphos, dimethoate, and triphenyl phosphate are good indicators of system performance.

8.2.4 FPDs may be susceptible to stray light in the photomultiplier tube compartment. This stray light will decrease the sensitivity and the linearity of the detector. Analysts may check for leaks by initiating an

analysis is a dark room and turning on the lights. A shift in the baseline indicates that light may be leaking into the photomultiplier tube compartment. Additional shielding should be applied to eliminate light leaks and minimize stray light interference.

8.2.5 FPDs use a flame to generate a response. Flow rates of air and hydrogen should be optimized to give a sensitive, but linear detector response for target analytes.

8.2.6 If the instrument has sat idle for a period of time, it may be primed with one or more injections of the CCV prior to analysis of the CCV for the acquisition sequence.

8.3 INITIAL CALIBRATION

8.3.1 Prepare a minimum of 5 concentrations of calibration standards (generally 8 or 9 are used), defining the linear range of the detector. Create calibration standards by diluting aliquots of the intermediate calibration standard using hexane. Each ICAL standard must include all target analytes and the surrogate (at a level similar to the target analytes). A typical range for the calibration is 50ng/mL to 10000ng/mL. The lowest concentration standard shall be at a level at or below the reporting limit (RL) of each analyte. Not all compounds can be detected at the lowest levels of the calibration. Reporting limits are higher for compounds that do not respond well and cannot be detected in the lowest calibration standards. The mid-range calibration standard is used for continuing calibration verification (CCV). A typical calibration sequence and preparation steps are shown below in Table 1.

TABLE 1
CALIBRATION STANDARDS

Working Standard	Hexane (µL)	10,000 PPB Primary Standard (µL)	Standard Concentration (µg/L)
ICAL Level 1 (100%)	0	500	10,000
ICAL Level 2 (80%)	100	400	8,000
ICAL Level 3 (60%)	200	300	6,000
ICAL Level 4 (50%)	250	250	5,000
ICAL Level 5 (40%)	300	200	4,000
ICAL Level 6 (20%)	400	100	2,000
ICAL Level 7 (10%)	450	50	1,000
ICAL Level 8 (5%)	475	25	500
ICAL Level 9 (0.5%) (diazinon only)	995	5	50
2nd source ICV (40%)	300	200	4,000

CCV (50%)	250	250	5,000

8.3.2 Inject and analyze 1µL of each calibration standard. Each data file quantitation is accomplished via the external standard method of quantitation. Analyte Calibration Factors (CFs) are calculated as follows:

$$CF = \frac{\text{integrated peak area of analyte}}{\text{analyte concentration}}$$

Since each CF represents the slope of the line between the response for that standard and the origin, then if the observed deviation between the CF's is constant (i.e., ≤20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample content.

Relative Standard Deviation (RSD) is calculated as:

$$\text{RSD}\,(\%) = \left(\frac{\text{standard deviation of the analyte's response factors}}{\text{mean response factor}}\right)(100)$$

When %RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be ≥0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of 6 calibration points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration.

The type of curve fit applied should be chosen to best represent the data.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among

calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

If regression criteria cannot be met, a new initial calibration must be performed.

8.4 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is analyzed after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time. The acceptance criteria for the ICV are identical to those of CCV (described below). If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.5 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end, of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum, and some clients' LIMS program specifications may require more frequent CCV analysis. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

Calibration is verified when all compounds are ≤20%D, when calculated as shown below:

$$\%D = \left[\frac{(\text{calculated concentration}) - (\text{expected concentration})}{\text{expected concentration}} \right] (100)$$

If a compound shows *elevated* response (> 20%D) and is not detected in any samples associated with the CCV, re-analysis of those samples are not necessary. If a compound shows *low* response (> 20%D) and was not detected in the samples associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If any CCV does not meet acceptance criteria, analyses should be halted and corrective action taken. Reanalyze the CCV. If the CCV still fails, the instrument must be recalibrated and all samples injected since the last compliant CCV must be reanalyzed.

8.6 RETENTION TIME WINDOWS

For GC methods, retention times are used for analyte identification. Retention Time Windows (RTWs) are established each time a new column is installed, and are used to compensate for minor RT shifts. It is important to establish valid RTWs. If too tight, false negatives may result. If too loose, false positives may occur. Determine RTWs by analyzing replicates (typically three injections), of a mid-level standard containing all analytes, non-consecutively, over a 72-hour period (this approach captures normal system variation). Calculate the standard deviation (σ) of the absolute retention time yielded for each analyte for the set of analyses used for the RTW study. Define each analyte's RTW as the mean retention time ±3σ, such that the Upper Limit = +3σ and the Lower Limit = -3σ.

RTWs should be centered on the midpoint standard if applied to samples run immediately after an initial calibration, or centered on the beginning CCV when samples are not directly preceded by an initial calibration. Sample matrices may cause drift that requires further Analyst interpretation. In the chromatography data system, RTWs and integration parameters should be set to err on the side of false positives, so target compounds are not missed by the data system. See Appendix I for chromatographic interpretation examples.

8.7 SAMPLE ANALYSIS (IDENTIFICATION, CALCULATIONS, REPORTING)
A constant volume, generally 1µL of each standard, blank, QC or field sample extract is directly injected into the GC via the automated injector. Sample extracts are diluted to maintain response within the linear range when necessary. All prepared extracts contain the surrogate (see Section 9). Extract concentration and sample concentration are determined as discussed below.

8.7.1 Note that ALS employs a sample splitter that facilitates simultaneous dual column injections; therefore, both columns are calibrated in the same manner and either column may serve as the column for quantitation.

8.7.2 Tentative identification occurs when a peak from a sample extract falls within the RTW of one column. If the peak also falls within the RTW of the second column, then the analyte's presence has been confirmed. Quantitation is calculated from both column responses and the best result is reported. The analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the quantitation column for each analyte detected and reported. If there are interferences present on one column, these need to be documented by printouts of that region of the chromatogram; the lower (i.e., other column's) results may be reported in this case. If results from both columns are of comparable quality, the higher concentration is reported (per SW8000).

8.7.3 Sample concentration is calculated using the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)(V_t)(DF)}{m(V_s \text{ or } W_s)}$$

where:

x = concentration of the analyte

y = analyte instrument response (area units)

b = calculated intercept

m = calculated slope of the line

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

V_s or W_s = volume or weight of sample extracted (mL or g)

If the average CF is used for quantitation, the sample concentration is calculated using the following equation:

$$\text{Concentration}_{(ug/L,\ ug/kg)} = \frac{(A_x)(V_t)(DF)}{(\text{Average CF})(V_s \text{ or } W_s)}$$

where:

A_x = analyte response (area units)

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

Average CF = average calibration factor

V_s or W_s = volume or weight of sample extracted (mL or g)

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specifications for additional or alternative requirements.

9.2 METHOD BLANKS

MBs are aliquots of matrix (i.e., organic-free water for liquids, boiling chips for solids) that have been prepared and analyzed in the same manner as the associated field samples. MBs are analyzed to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Concentrations of target analytes, if any, must be less than the reporting limit (RL), or as otherwise prescribed in the LIMS program specification. If this criterion is not achieved, then analyses must be halted and the source of the contamination found and corrected.

As the analyst deems necessary, aliquots of solvent may be injected to clean the analytical system and demonstrate that it is free from contamination.

9.3 LABORATORY CONTROL SAMPLE

The LCS is analyzed to measure the accuracy of the analytical system. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample and/or matrix spike sample is performed in duplicate (LCSD, MSD). The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$RPD = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. See QC Table for evaluation criteria. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%R = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right)(100)$$

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation is made in the data package narrative.

9.6 SURROGATE RECOVERY

The %R of the surrogate is calculated (see equation shown for LCS recovery above) for all field and QC samples. If the recovery is outside QC limits, the sample is reanalyzed to verify that analytical error was not the problem. If after re-injection the recovery is still out-of-control, initiate an NCR (SOP 928) to consult with the Project Manger (and client, as needed) and apply the decided upon action.

9.7 METHOD DETECTION LIMIT STUDY

A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven (7) replicates for each target analyte at a concentration level near to the capabilities of the method. The MDL study is performed as needed, at minimum, annually, following the guidance of SOP 329.

10. DEVIATIONS FROM THE METHOD

10.1 This SOP meets the requirements of Methods SW8141A,B. There are no known deviations from the methods.

10.2 As of this writing, ALSLF-FC does not analyze wastewater samples for compliance monitoring purposes. ALS extracts aqueous samples according to SW-846 protocol. Following are deviations from EPA Method 614:

10.2.1 EPA Method 614 prescribes preparation via a separatory funnel shakeout. ALS extracts samples using continuous liquid-liquid extractors (CLLEs).

10.2.2 EPA Method 614 prescribes an extraction mixture of 15% methylene chloride and 85% hexane. ALS extracts samples with methylene chloride only, per SW3520C protocol.

10.2.3 EPA Method 614 requires a relative standard deviation (RSD) of less than 10% in order to quantify samples by the average response factor. ALS requires an RSD of less than 20% to quantify samples by the average response factor, per SW8000C protocol.

10.2.4 EPA Method 614 requires a percent difference of ±10% for continuing calibration standards. ALS requires a percent difference of ±20%, per SW8000C protocol.

10.2.5 ALS does not analyze the full list of compounds from EPA Method 614.

10.2.6 For Health & Safety and waste stream management reasons, ALS does not support use of mercuric chloride as a preservative.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 USEPA, SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, Volume 1B, Method 8141A, Revision 1, September 1994.

12.2 USEPA, SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, Volume 1B, Method 8141B, Revision 2, February 2007.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12.4 USEPA, EPA 821 RR-92-002, April 1992. Method 614. "The Determination of Organophosphorous Pesticides in Municipal and Industrial Wastewater".

Analytical Method: SW8141A,B or EPA614	**Parameter:** Organophosphorous Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL); minimum 5-points, all analytes (SW 8141B); Method EPA 614 does not require 5-points	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, use mean RFs and CFs to quantitate. If RSD >20% , calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r^2) is ≥ 0.990 or calculate quadratic regression (minimum of six points required); use for quantitation if COD (r^2) ≥0.990	Evaluate/correct instrument malfunction and reanalyze ICAL to obtain acceptable curve
Initial Calibration Verification (ICV); conc. not equal to midpoint of calibration curve; second source	After each ICAL	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.
Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Brackets each set of 20 field sample analyses (standard practice is every 10 samples injections)	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re-analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.
Retention Time Window (RTW); based on minimum of 3 non-consecutive injections throughout at least a 72-hour period to be	Whenever a new column is installed	Column and compound specific Window is ±3x the standard deviation of the 3-injection average for the respective	If SD=zero, then either do additional injections or use a default SD of 0.01 minutes. Experience of analyst weighs heavily in interpretation of chromatograms

Analytical Method: SW8141A,B or EPA614	**Parameter:** Organophosphorous Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
representative of variation		column Note that the ICV and CCV analyses are also used to monitor RT drift	(refer also to RT Shift).
Retention Time Shift; RT of analytes in CCV are evaluated against the midpoint of the ICAL	Each CCV; RT of analytes evaluated against the ICAL	Column and compound specific	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate. Evaluate data based on a comparison with other standards run during the analytical sequence; consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question.
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition
Blank Spike (BS); Laboratory Control Sample (LCS)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds must be within laboratory limits or other limits as specified in the LIMS program specification	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed) - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be

Analytical Method: SW8141A,B or EPA614	**Parameter:** Organophosphorous Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			extracted outside of the holding time and the data will be submitted with appropriate narration
Matrix Spike (MS)	1 per batch, not to exceed 20 samples of like matrix	See laboratory limits; recoveries for spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per batch not to exceed 20 samples of like matrix	See laboratory limits; see Matrix Spike information above for MSD recoveries RPDs should be within advisory limits	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.
Surrogate Spike	All field and laboratory QC samples	See laboratory limits; recoveries should be within current limits, alternative criteria as defined in the LIMS program specifications may apply	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery. - if surrogate recovery in the associated MB is not within limits and the samples are within the holding time, then re-extract and reanalyze all associated samples - if samples are beyond the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.

Analytical Method: SW8141A,B or EPA614	**Parameter:** Organophosphorous Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Method Detection Limit (MDL) Study	As needed, at minimum, annually	Concentrations for the MDL study shall be at a level lower than that of the reporting limit	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

Uncontrolled Document

ALS Standard Operating Procedure

DOCUMENT TITLE:	ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBS) BY GAS CHROMATOGRAPHY
REFERENCED METHOD:	SW 8082 AND EPA 608
SOP ID:	409
REV. NUMBER:	7
EFFECTIVE DATE:	OCTOBER 15, 2013

STANDARD OPERATING PROCEDURE 409 REVISION 7

TITLE: ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBs) BY GAS CHROMATOGRAPHY -- METHODS SW8082 and EPA 608

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references - SW846 Method 8082 and EPA 608, are used to determine the concentration of Aroclors 1016 through 1268 in various matrices. The following Aroclors may be analyzed:

Aroclor 1016	Aroclor 1242	Aroclor 1260
Aroclor 1221	Aroclor 1248	Aroclor 1262*
Aroclor 1232	Aroclor 1254	Aroclor 1268*

* Aroclors 1262 and 1268 are not routinely analyzed by the laboratory, but may be determined upon request. Decachlorobiphenyl (DCB) is normally added as a surrogate standard. However it can be reported as an analyte.

Aroclors are multi-component mixtures. Qualitative and quantitative determination may be more difficult if a sample contains more than one Aroclor, has been subjected to environmental degradation (weathering), or has been degraded by treatment technologies. Weathered and degraded samples exhibit patterns that differ from Aroclor standards.

The body of this SOP specifies the procedures to be used for Method SW8082 analysis. Any additional or contradictory requirements for Method EPA 608 are addressed in Section 10.

2. SUMMARY

Samples are extracted and the extracts concentrated and solvent exchanged using appropriate ALS SOPs (i.e., 617 [CLLE]; 620 [Microwave]; 626 [Separatory Funnel]; 625 [Soxhlet]; 622 [Waste Extraction]; 607 [Kuderna-Danish Reduction]; and 637 [Concentration and Solvent Exchange]).

NOTE: With prior arrangement with the laboratory, solid samples may also be extracted using pulse sonication techniques.

The extracts are injected into a gas chromatograph (GC) containing a sample splitter and two columns of varying selectivity (i.e., dissimilar elution and retention time properties.

The target analytes are separated in the columns and detected by two electron capture detectors (ECDs). This chromatography system allows tentative identification by one column and confirmation by the other column to be performed simultaneously.

Quantitation is performed using the best column response yielded for each analyte. The Analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the primary quantitation column for each analyte detected and reported. The particular value that is selected for reporting is often marked (designated) in the raw data (e.g., quantitation report, run log). If results from both columns are comparable, the highest result is reported. Second column confirmation is not required if the primary column does not detect an Aroclor pattern, and may not be necessary if the sample matrix is well characterized by previous analyses.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Interference from phthalate esters can be minimized by using plastic-free solvent containers and scrupulously cleaned glassware (SOP 334) that has been kiln-baked or solvent-rinsed prior to use. Use of low phthalate gloves, such as nitrile, may also

important. These practices are important both in the field and in the laboratory.

4.2 Sulfuric acid clean up techniques may be used to remove interferences caused by the presence of organochlorine and/or organophosphorous pesticides. See SOP 651 for instructions regarding Method SW3665A sulfuric acid clean up.

4.3 Elemental sulfur (particularly in sediment samples) may interfere with PCB pattern identification and can be removed by using appropriate clean up techniques prior to sample analysis. See SOP 634 for instructions regarding Method SW3660B sulfur cleanup.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH (GC), AUTOSAMPLER AND DETECTORS:

5.1.1 - Hewlett Packard (HP) 5890 Series II GC equipped with HP 7673A autosampler, dual on-column injection, and electron capture detectors (ECDs) or equivalents

5.1.2 Agilent Technologies 7890A GC equipped with Agilent 7693 autosampler and electron capture detectors (U-EDS or ECD)

5.2 CHROMATOGRAPHIC DATA ACQUISITION AND PROCESSING SYSTEM - Agilent Technologies (EZChrom Elite) or equivalent

5.3 COLUMNS –
The following specified columns or equivalent columns are used with this analytical method.

Rtx-5 or equivalent (30m, 0.25 or 0.32mm ID, 0.5µm film),
Rtx-CLPesticides II or equivalent (30m, 0.25 or 0.32mm ID, 0.25µm film), guard column

Restek Capillary Column:	RTX-CLPesticides	#11139 (0.32mm)
Restek Capillary Column:	RTX-CLPesticides2	#11324 (0.32mm)

5.4 GASES - ultra high purity (99.999%)
Helium - carrier gas
Nitrogen - make-up gas

5.5 MEASURING DEVICES
Syringes - 1.0µL-1000µL precision Hamilton™, or equivalent
Volumetric flasks, Class A with ground glass stoppers, 10mL and 25mL

5.6 GC CONSUMABLES

- Vials - Resolution Systems 67-VT011LM-1232, or equivalent
- Caps - Resolution Systems 67-C141-11, or equivalent
- Inlet Seals, dual vespel ring - Restek 0.8mm #21243, or equivalent
- Septa, 11mm - Restek #20365 or equivalent

- O-ring, graphite, 6.5mm - Restek #20299, or equivalent
- O-ring, Viton - Restek #20377, or equivalent
- Liner, splitless, 4mm ID - Restek #20799-214.5, or equivalent
- Glass Wool, deactivated - Restek #20789, or equivalent
- Gold Seal - Restek #21306, or equivalent
- Universal Presstight Y - Restek #20469, or equivalent
- Vespel/Graphite Ferrule (detector) - Restek #20221, or equivalent
- Compact Vespel/Graphite Ferrule - Restek #20249, or equivalent
- Graphite Ferrules - various sizes
- Split Vent Trap (SW8081) - Agilent RDT-1023, or equivalent

6. REAGENTS AND STANDARDS

6.1 SOLVENTS - **Only pesticide residue grade or equivalent may be used.**
Hexane - Burdick and Jackson 216-4, or equivalent
Methanol - Burdick and Jackson 230-4, or equivalent

6.2 STANDARDS
All standards are stored following ALS SOP 300 guidance, which is superseded by any guidance in this SOP. Generally after opening vials, the standards for this procedure are stored in the freezer (-10°C and -20°C), in PTFE-capped, or equivalent vials. Unopened stock standards in flame-sealed ampules are valid until the manufacturer's expiration date and may be stored at room temperature, if recommended by the manufacturer. Opened stock standards and intermediate standards expire six months from opening (preparation) or the manufacturer's expiration date, whichever is sooner. Standards may be replaced sooner if laboratory QC analyses or other factors indicate deterioration.

All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

6.2.1 Stock Standards: An approximately 1000mg/L (per component) stock solution is purchased from a suitable vendor or prepared in-house gravimetrically by accurately weighing 0.0100g of pure material into a 10mL Class A volumetric flask and diluting to volume with n-hexane. If purity of the compound is 96% or greater, no weight correction is necessary; if compound purity is less than 96%, the concentration must be corrected mathematically based on weight used.

A combination standard containing Aroclor 1016 and 1260 will generate peaks covering the range of all Aroclors of interest. Individual standards for all Aroclors must be created to assist in pattern recognition. The primary stock standards are subsequently diluted to create the intermediate stock standards. Undiluted, opened primary stock standards may be retained for up to six months.

6.2.2 Intermediate Standards: Generally prepared by diluting 1mL of stock standard to 25mL using a Class A volumetric flask and n-hexane. The intermediate stock standard is further diluted to create the calibration standards. Intermediate stock standards may be retained for up to six months.

6.2.3 Calibration Standards: Calibration standards are made daily from intermediate stock standards. Typically, ALS prepares a mixture of Aroclor 1016/1260 at a minimum of 5 different concentrations that define the working linear range of the detector. The initial calibration must have a standard at or below the analyte reporting limit. A single point standard for the remaining requested Aroclors should be prepared at the analyte reporting limit. The single point standard will be used for pattern recognition, and to verify the sensitivity of the instrument for each Aroclor.

If the presence of an Aroclor other that 1016/1260 is suspected, a calibration curve containing a minimum of five concentration levels should be prepared for that Aroclor. Create calibration standards by preparing serial dilutions of the intermediate stock standard in n-hexane. A calibration standard at a concentration near the midpoint of the calibration curve will be used as a continuing calibration verification (CCV) standard. See SOP 300 for additional information about standard expiration dates.

6.2.4 Independent Calibration Verification Standard (ICV): Certified and purchased from a vendor or made gravimetrically in-house. Uses a source different from that of the calibration standard so that the accuracy of the calibration standard may be independently verified. Created and analyzed at a concentration level that is near the midpoint of the calibration range. The ICV should be prepared at a level different from the concentration used for the CCV, in order to verify a wider range of the calibration.

6.2.5 Surrogate Spike Solution - Certified and purchased from a vendor or made in-house. Typically, this standard contains 500ng/mL each tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in methanol. During preparation, 1.0mL of this standard is spiked into each sample, standard, and quality control (QC) sample. Other concentrations or solvents may be used as needed (i.e., as defined in the applicable LIMS Program Specification).

6.2.6 Spike Solution - A commercial (i.e., purchased from a vendor) primary standard containing 1,000ppm Aroclor 1016/1260 is used to prepare a 5ppm in methanol solution to be used by the Organics Extractions Group in preparing laboratory control samples (LCS/LCSD) or matrix spiked samples (MS/MSD); typically 1mL of spike solution is added. Other concentrations and/or solvents may be used as appropriate (i.e., as indicated in the applicable LIMS program specification).

NOTE: An internal standard is not currently used for Aroclor analysis. Qualitative identification is determined by pattern recognition and quantitation is accomplished using the external standard method. Dual column confirmation is also routinely performed.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Samples should be collected according to an approved sampling plan.

7.2 Liquid samples are not chemically preserved and must be collected in amber glass containers (generally 1L) with Teflon-lined lids. Samples should be maintained at 4±2°C .

7.3 Solid samples are collected in 250mL widemouth glass containers with Teflon-lined lids. Solid samples are not chemically preserved and should be maintained at 4±2°C. .

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 TYPICAL GAS CHROMATOGRAPHIC CONDITIONS

Carrier Gas (He):	1-6mL/min
Make-up Gas (N_2):	20-40mL/min
Purge:	on, 0.75min
Injector Temperature:	205°C
Detector Temperature:	325°C

Oven Temperature Program:

Initial Temperature:	110°C
Oven Ramp:	15°C/min. to 250°C
Oven Ramp A:	20°C/min. to 300°C
Oven Ramp B:	5°C/min. to 270°C
Hold:	5 min

8.2 CHROMATOGRAPHIC MAINTENANCE

8.2.1 Aliquots of solvent may be injected and analyzed to show that the

analytical system is free from contamination. They may be injected following samples of unusually high concentration to check the status of the analytical system and to facilitate re-equilibration of the system. Solvent injections may be used to prime the system if it has sat idle for awhile.

8.2.2 Peak tailing for all components will be exacerbated by a dirty injector. Clean per manufacturer's instructions as needed.

8.2.3 ECD detector leak checks are performed semi-annually per the procedures outlined in SOP 016.

8.3 INITIAL CALIBRATION

Prepare calibration standards as discussed above (including addition of surrogate). Typically, a 5-point curve of Aroclor 1016/1260 is prepared and a single-point reporting-limit standard of the remaining Aroclors is prepared. Inject 1-2µL of each standard directly into the GC and analyze. Quantitation is accomplished using 3 to 8 peaks for each Aroclor via the external standard method of quantitation. Where possible, peaks should be chosen that are at least 25% of the height of the largest Aroclor peak. Analyte calibration factors (CFs) are calculated for each peak as follows:

$$CF = \frac{\text{Selected Peak Areas}}{\text{Mass of Aroclor Injected On-Column (ng)}}$$

NOTE: SW-846 8000C also allows for use of concentration instead of mass injected.

If the CFs over the working range of the detector are constant (i.e., ≤20% RSD), then response can be assumed to be invariant and the average (mean) CF may be used to quantitate sample content. Relative Standard Deviation (RSD) is calculated as:

$$RSD\ (\%) = \frac{\text{Standard Deviation (SD)}}{\text{Average (mean) CF}} \times 100$$

When RSD over the calibration range is ≥20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The type of curve fit applied should be chosen to best represent the data. The regression calculation will yield a coefficient of determination (r^2 value) that must be >0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit" with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of 6 points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The type of curve fit applied should be chosen to best represent the data.

If the comparison of a sample to a one-point standard suggests that Aroclor 1221, 1232, 1242, 1248, 1262, or 1268 may be present, then a 5-point curve of the appropriate Aroclor standard is prepared. The sample is re-analyzed and quantified using the appropriate 5-point curve.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The Analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration.

If regression criteria cannot be met, a new initial calibration must be performed.

8.4 INITIAL CALIBRATION VERIFICATION (ICV)

Though not required to be a second source by SW-846 Method 8082, a second source standard (ICV) is run after calibration. The concentration of the ICV should be varied over time and should not be equal to that of the continuing calibration verification (CCV). The acceptance criteria for the ICV are identical to those of the CCV described below. If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.5 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end, of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum, and some clients' LIMS program specifications may require more frequent CCV analysis. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

Calculate the percent difference or drift (%D) between the initial and continuing calibration using the equations below:

$$\% \text{ Difference} = \frac{\overline{CF}_I - CF_c}{\overline{CF}_I} x100$$

where:

CF_I = The average calibration factor in the initial calibration

CF_c = Calibration factor for the continuing calibration

$$\%Drift = \frac{CC - TC}{TC} \times 100$$

where:

CC = Calculated concentration

TC = Theoretical concentration

Note that if a least squares regression is used, the CCV must be evaluated using a percent drift calculation.

If the %D is ≤20%, the CCV is acceptable, and sample analysis may begin. If a compound shows *elevated* response (> 20%D) and is not present in any samples associated with the CCV, re-analyses of those samples are not necessary. If a compound shows *low* response (> 20%D) and was not detected in the samples associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If any CCV does not meet acceptance criteria, analyses must be halted and corrective action taken. Reanalyze the CCV. If the CCV still fails, the instrument must be recalibrated and all samples injected since the last compliant CCV must be reanalyzed.

8.6 RETENTION TIME WINDOWS

Retention Time Windows (RTWs) are established according to the criteria prescribed by Method SW8000C, Section 11.6. Analyze a mid-level standard in triplicate for each Aroclor, non-consecutively, during a 72-hour period. Calculate the mean and standard deviation (σ) of the three absolute retention times for three to five major peaks in each Aroclor. Each Aroclor's RTW is defined as three times the calculated standard deviation (±3σ) of each major peak, such that the Upper Limit = +3σ and the Lower Limit = -3σ.

8.7 SAMPLE ANALYSIS, CALCULATION AND REPORTING

A constant volume, generally 1µL, of each standard, blank, QC or field sample extract is directly injected into the GC via the automated injector. Sample extracts are diluted to maintain response within the linear range when necessary. All prepared extracts contain the surrogate (see Section 9).

8.7.1 Note that ALS employs a sample splitter that facilitates simultaneous dual column injections; therefore, both columns are calibrated in the same manner and either column may serve as the column for quantitation.

8.7.2 Aroclors are identified through pattern recognition. Tentative identification occurs when selected peaks fall within the RTW of one

column. If selected peaks also fall within their RTW on the second column, the analyte's presence has been confirmed. Quantitation is calculated from both column responses and the result with the least interference is reported. If both results are of equal quality, the higher result is reported. For the multi-response Aroclors, three to eight peaks are used for identification/quantitation. The same selected peaks must be consistently used for quantitation between the standard and sample set.

NOTE: Unlike ALS's normal protocol with regard to single component target compounds, the visual presence of a particular aroclor pattern will be used as confirmation of a positive detection even if the quantitation on the confirming column is less than the method detection limit.

8.7.3 Analyst expertise is crucial in identifying and quantitating samples containing multiple Aroclors or Aroclors that are heavily weathered. Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to such an extent that a specific Aroclor pattern is no longer recognizable. Consult the Department Manager for assistance in interpreting complex chromatograms.

8.7.4 Sample analyte concentration is calculated using the equation of the linear curve (i.e., y = mx + b) or average calibration factor generated, which can be represented as:

linear: $A = mC + b$, or $C = (A - b) / m$

average calibration factor: $C = A/R_f$

where:

A = analyte response (area counts)
m = slope of the linear equation
C = concentration present at the instrument
b = the y-intercept of the linear equation
R_f = the average calibration factor from the initial calibration

A concentration is determined for each peak, and the total Aroclor concentration is then determined by averaging the concentrations of the 3 to 8 individual peaks.

8.7.5 Identification of Aroclors may be simplified by comparing standard peak patterns to sample patterns (i.e., RTW of the group of peaks, comparison of peak heights and ratios; software overlays may also be used as desired/possible). Assessment observations may be confirmed using the calculated RTWs. If a minor shift of the entire pattern is observed, the Aroclor may still be positively identified provided that the confirming column result also supports the decision.

8.7.6 Integration between the reference standard and the sample must be consistent. For example, the baseline may be drawn from the start of the first peak in the group to the baseline following the last peak, and lines dropped from peak valleys to baseline for calibration standards and the samples alike.

9. QUALITY CONTROL

9.1 DEFINITION OF ANALYSIS BATCH

For this method, an analysis batch is defined as a group of samples that are associated with one unique set of batch QC samples and analyzed together. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS) and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specification for additional or alternative requirements.

9.2 METHOD BLANKS

Method blanks are aliquots of matrix (i.e., organic-free water for liquids analyses; Ottawa Sand for solids analyses), which have been prepared and analyzed in the same manner as the associated field samples. To be acceptable, concentrations of analytes of interest detected (if any) in the MB must be below the analyte reporting limit, or as otherwise specified in the LIMS program specification. If this criterion is not achieved, analyses should be halted and the source of the contamination found and corrected.

A reagent or instrument blank is an injection of solvent analyzed to demonstrate that the analytical system is free from contamination. These blanks are typically analyzed following extremely contaminated samples.

9.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the method and analytical system. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this analysis, either a field sample containing target compound contamination may be analyzed in duplicate, or the laboratory control sample (LCSD) or matrix spike (MSD) analysis can be performed in duplicate. The results of the duplicate analyses are evaluated in terms of Relative

Percent Difference (RPD) as shown below. See QC Table for evaluation criteria.

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. See QC Table for evaluation criteria. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%\text{R} = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right)(100)$$

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

Also note that for projects in which the client is to designate MS/MSD samples, an analysis batch may not contain an MS/MSD pair. Where this occurs, a notation will be made in the data package narrative.

9.6 SURROGATE RECOVERY AND ACCEPTANCE CRITERIA

The two surrogates (SUR) used for this procedure are those suggested in SW8081A. Both surrogates are added to all field and quality control samples prior to extraction; recovery is calculated per the recovery formula shown previously for the LCS. The two surrogates used were selected because they respond in a similar manner as the target compounds respond at the detector. Additionally these surrogates are not similar enough chemically to the target compounds to co-elute with the single component targets, nor do they suffer interferences from the multiple component targets. Tetrachloro-m-xylene (TCMX) elutes before any of the target compounds, and decachloro-biphenyl (DCB) elutes after all of the target compounds. However, because the surrogates are not deuterated analogs of targets as in GC/MS methods, they are not extracted with exactly the same efficiencies as the target compounds. Therefore, surrogate recovery problems are not representative of target analyte recoveries.

Heavy co-extractive non-target compounds generally do not interfere with TCMX; light co-extractive non-target compounds generally do not interfere with DCB. However, some samples may produce matrix effects that cause surrogate recovery to be high or low; because of high concentrations of target and/or non-target compounds, quantification of the surrogates may even be precluded in some samples. Muddy aqueous samples, for example, generally adsorb DCB after spiking and limit recovery to a few percent. High concentrations of heavy hydrocarbons in soils oftentimes have a similar matrix effect on DCB recovery.

The extraction process itself can have an effect on surrogate recovery. An example of this process-caused effect is use of Method SW3520 for extraction of aqueous samples and associated "low" recoveries of DCB. DCB is a heavy molecule and very hydrophobic. When spiked into water, this compound tends to rapidly adsorb to particulates at the liquid-liquid interface and exhibits a low recovery.

For the reasons listed above, ALS does not view the evaluation of surrogate recovery in this procedure to be a straightforward process. Therefore, ALS observes the following guidance for evaluating surrogate recovery:

- ALS's practice is to evaluate and report the recovery of both surrogates. When one or both surrogates are within laboratory control limits, the process is considered to be in control and no further action is taken (unless additional measures are stipulated in the LIMS program specification.

- When, due to elevated target concentrations, an extract requires a dilution of greater than 5X, ALS does not consider the surrogate recoveries to have meaning; no further action is required.

- When both surrogates are outside of laboratory control limits (or other limits specified in the LIMS program specification), the extract is re-injected to assure that instrument error was not the cause. If after re-injection the recoveries of both surrogates remain out of control, then re-extraction and re-analysis may be performed as directed by the client. A non-conformance report (NCR; SOP 928) to document the problems is required.

This process of evaluating surrogate recovery is based on several methods and guidance documents and has evolved in particular from Method SW8080 guidance as well as from the National Functional Guidelines for Data Review.

9.7 METHOD DETECTION LIMITS

The MDL study should be performed as needed and at a minimum, annually. See SOP 329 for further guidance.

10 DEVIATIONS FROM METHOD

10.1 This SOP meets the requirements of Method SW8082. The following is the only known deviation from the method: The method allows for single point calibration of Aroclors 1221, 1232, 1242, 1254, 1262 and 1268 using a point near the midpoint concentration of the 1016/1260 curve. ALS analyzes a single point for these Aroclors at the reporting limit to assist in pattern recognition as well as to show instrument sensitivity for all the targets. If Aroclor 1221, 1232, 1242, 1254, 1262 or 1268 is detected in the sample, then a calibration curve with at least 5 points is prepared and the sample re-analyzed for that Aroclor following the procedures discussed in this SOP. This more stringent approach is intended to provide more accurate quantitaton for these Aroclors. A single point may be done for Aroclor 1268. Client requirements dictate if a single point can be used.

Note that ALS defines analytical shift (8000C) as per 24hrs, not 12hrs.

10.2 EPA 608: The items discussed in this Section list differences between Methods EPA 608 and SW8082:

10.2.1 EPA 608 states specific extraction methods, chromatographic conditions, etc. to be used in the execution of the method. Some of these materials, apparatus, and conditions have been eclipsed by the more modern technology listed in this SOP. Section 8.1.2. of EPA 608 also states that technological advances are recognized and allowed for use provided that the precision and accuracy requirements put forth by the method can be achieved.

10.2.2 EPA 608 specifies extraction of samples by separatory funnel. ALS uses continuous liquid-liquid extractors (CLLEs) or separatory funnels to extract aqueous samples.

10.2.3 EPA 608 specifies that calibration standards be kept in isooctane. ALS uses hexane as a solvent for calibration standards.

10.2.4 EPA 608 states that if the RF value over the range demonstrated by the initial calibration is $\leq$10%, the average response factor can be used for calculations. otherwise construct a linear regression curve. ALS typically quantifies Aroclors from a linear regression curve.

10.2.5 EPA 608 specifies that a continuing calibration standard be analyzed every 24 hours. ALS follows Method SW8082 that specifies a continuing calibration verification be analyzed every 12 hours and every 20 samples

thereafter (although 10 samples between CCV's is recommended to prevent reruns)..

10.2.6 The use of surrogate standards is not addressed in EPA 608. ALS uses the two surrogates discussed in this SOP.

10.2.7 Because samples from several sites are usually batched together, only one spiking level is used for each compound. Per EPA 608, it is impractical to match each compound's spike amount with the amount of the compound in the samples chosen for spiking, and to match the spike amount to the appropriate regulatory level for each compound. This difference must be stated in the data package narrative for EPA 608 sample analyses.

10.2.8 Section 7.5 of EPA 608 prescribes the use of Florisil columns to remove co-extractives; a sulfuric acid clean up procedure is not discussed in EPA 608. ALS routinely performs a Method SW3665A sulfuric acid clean up on all samples analyzed for PCBs only (see SOP 651).

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 3rd edition, Final Update III, Method 8082, Revision 0, December 1996.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12.3 40 CFR, Part 136, Appendix A, 7-1-99 edition; Method 608.

Analytical Method: SW8082, EPA 608	**Parameter:** Polychlorinated Biphenyls (PCBs)		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point; all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20% (Method SW8082) or ≤10% (EPA 608), use mean CFs to quantitate. If RSD >20% , calculate linear regression (not forced through origin); use for quantitation if coefficient of determination (r^2) is ≥ 0.99.	Evaluate/correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Initial Calibration Verification (ICV); run near midpoint of calibration, but at a different concentration than CCV	With each initial calibration	If ≤20% D analyses may proceed.	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV); run at or near midpoint of calibration	Daily prior to sample analyses; brackets each set of 20 field sample analyses (10 sample analyses recommended)	If ≤20% D analyses may proceed.	Evaluate/correct instrument malfunction as needed (e.g., remove 1 meter from the guard column of the GC, prepare a new standard); reanalyze. - If CCV still non-compliant, recalibrate using a new curve. Samples analyzed after a failed CCV will be reanalyzed. - If target(s) in CCV fails high (>20%) and target is not present in samples, re-analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. -
Retention Time Window (RTW)	Whenever a new column is installed, based on 3 injections throughout a 72-hour period to be more representative of daily operations	Column and compound specific. Window is ±3x the standard deviation of the 3-injection average for the respective column. Note that the ICV and CCV analyses are also used to monitor RTW shift.	If SD=zero, then either do additional injections or use a default SD of 0.01 minutes. Experience of analyst weighs heavily in interpretation of chromatograms (refer also to RT Shift).
Retention Time (RT)	Each CCV; RT of	Column and compound	Inspect chromatographic system for malfunction;

Analytical Method: SW8082, EPA 608	**Parameter:** Polychlorinated Biphenyls (PCBs)		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Shift	analytes evaluated against the ICAL	specific	correct identified malfunctions, if appropriate. Evaluate data based on a comparison with other standards run during the analytical sequence; consider the RTs for the surrogates and spiked compounds analyzed before and after the sample in question:
Method Blank (MB)	1 per each preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise -
Laboratory Control Sample (LCS)	1 per batch of ≤20 samples of like matrix	See laboratory limits; recoveries for the spiked compounds must be within the laboratory limits or other limits as specified in the LIMS program specification.	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions or analytical preparation was the cause. - if still non-compliant, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that does not meet criteria. -
Matrix Spike (MS)	1 per batch of samples, not to exceed 20 samples of a given matrix	See laboratory limits; recoveries for the spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Duplicate	1 per batch of samples, not to exceed 20 samples of a given matrix.	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within advisory limits.	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). If no errors are found and, if analyzed, LCSD RPD is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Surrogate Spike	All extractions including field and laboratory QC samples.	See laboratory limits; recoveries should be within current limits for one or both surrogates; alternative criteria as defined in the LIMS program specifications may apply.	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with both surrogate recoveries outside the recovery limits, with no visible chromatographic cause, should be re-injected to determine if an injection error was the

Analytical Method: SW8082, EPA 608	**Parameter:** Polychlorinated Biphenyls (PCBs)		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
			cause for the low recovery. - if both surrogate recoveries in the associated MB are not within limits, , then re-extract and reanalyze all associated samples. -
Method Detection Limit (MDL) Study	As needed and at minimum, annually	Concentrations for the MDL study shall be at a level lower than that of the reporting limit	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If `criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

ALS Standard Operating Procedure

DOCUMENT TITLE:	ANALYSIS OF TOTAL VOLATILE PETROLEUM_HYDROCARBON (TVPH) GASOLOINE RANGE ORGANICS (GRO) BY GAS CHROMATOGRAPHY
REFERENCED METHOD:	EPA SE 8015B OR D, AND CAL-LUFT
SOP ID:	425
REV. NUMBER:	17
EFFECTIVE DATE:	APRIL 17, 2014

STANDARD OPERATING PROCEDURE 425 REVISION 17

TITLE: ANALYSIS OF TOTAL VOLATILE PETROLEUM HYDROCARBON (TVPH) GASOLINE RANGE ORGANICS (GRO) BY GAS CHROMATOGRAPHY -- METHODS SW 8015B or D, and CAL-LUFT

FORMS: NONE

APPROVED BY:

PRIMARY AUTHOR _____ DATE 5-6-14

QUALITY ASSURANCE MANAGER _____ DATE 4-17-14

LABORATORY MANAGER _____ DATE 04/17/2014

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- SW8015B and D, and CAL-LUFT -- are used to determine the concentration of Gasoline Range Organic (GRO) compounds in aqueous and solid/sludge samples.

Method SW8015 defines the alkane range corresponding to GRO as C_6 through C_{10}, with the approximate boiling points ranging from 60°C to 170°C. State-specific or client-specific fuels analysis protocols may require analysis of a modified carbon range.

Gasoline is identified by pattern recognition; however, certain volatile aromatic compounds ~~(e.g., benzene, xylenes, MtBE) are detectable as target analyte indicators. Analyst expertise~~ is crucial to this method, as multiple patterns may be present in a sample. Also, pattern responses in environmental samples may differ from textbook characterizations because of weathering. Any peak(s) present in the current GRO retention time window will be integrated and reported as GRO.

2. SUMMARY

Sample aliquots are introduced onto a purge and trap device and are subsequently desorbed onto a gas chromatograph (GC). The GC is temperature programmed to facilitate separation of surrogate standards, to produce a good GRO pattern, and to resolve the early diesel elution pattern. Analytes of interest are detected using a Flame Ionization Detector (FID). Detector responses are recorded and stored by an electronic data system. The sample response is compared to the GRO response of reference standards using the external standard method of quantitation.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the benchsheet indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work, and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving this procedure to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 High levels of heavier petroleum products (e.g., diesel fuels) may contain some volatile compounds that elute within the retention time range of GRO. Other organic compounds including halogenated solvents, ketones, and ethers are also measurable. As defined in the methods, GRO quantitation includes these compounds.

4.2 Samples may be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling, handling and storage protocols can serve as a check on such contamination. Weekly checks for possible sample storage refrigerator contamination are performed per SOP 512.Trip blanks are analyzed by VOA lab, unless specifically requested for GRO analysis.

4.3 If carryover contamination is suspected (as when a sample containing higher concentrations of volatile compounds, is followed by a sample containing lower levels of the same volatile compounds), all samples that may have been affected should be re-analyzed. Sample analysis may continue if a blank or sample following the higher concentration sample, is demonstrated as free (below the

reporting limit) of compounds that were present over the calibration range in the higher level sample. Analyst experience and judgment should be used to determine which compounds tend to carryover and at what levels. Annotations made to instrument run logs should indicate if a sample contains possible carryover contamination. If the subsequent rerun of the sample confirms the presence and level of the volatile compounds, either analysis may be used. If, however, the rerun shows that the presence of the compounds was carryover contamination, only the rerun data should be used.

5. APPARATUS AND MATERIALS

5.1 PURGE AND TRAP AUTOSAMPLER DEVICE

OI Model 4552 Archon automated sampler with concentrator (i.e., Tekmar 3000) device equipped with Supelco Trap K #21066-U, or equivalent

5.2 GAS CHROMATOGRAPH (GC) AND DETECTORS
Hewlett Packard 5890 Series II GC or equivalent equipped with a flame ionization detector (FID) .

NOTE: An FID must be used for GRO quantitation because its response is similar for all hydrocarbons; other detectors will not produce accurately quantitated total GRO results.

5.3 GC COLUMNS - Equivalent columns/guard columns may also be used

J&W Capillary Column:	DB-624 # 21512-304 *	(30m x 0.53mm ID, 3.0µm film thickness)
J&W Capillary Column:	DB-VRX # 21512-270 *	(30m x 0.45mm ID, 2.55µm film thickness)

* VWR part numbers

NOTE: The minimum acceptable column resolution should provide separation of 2-methyl pentane from the methanol solvent front and ethylbenzene from m/p-xylenes in standards.

5.4 CHROMATOGRAPHIC DATA SYSTEM
Agilent EzChrome Elite or equivalent

5.5 GASES - use only ultra high purity (99.999%)

Helium (purge and carrier gas)

Hydrogen (FID gas)

Compressed Air (FID gas)

5.6 MEASURING DEVICES

5.6.1 microsyringes, gas-tight, Precision Hamilton or equivalent, 5µL – 1.0mL sizes

5.6.2 volumetric flasks, Class A, with ground glass stoppers, various sizes

5.6.3 balance, capable of weighing ±0.01g

5.6.4 Luer-lock syringes, 5mL or 25mL, Becton & Dickinson or equivalent

5.7 CONSUMEABLES

5.7.1 Compact Vespel/Graphite Ferrules, Restek #20264 or equivalent

5.7.2 Graphite Ferrules, various sizes

5.7.3 VOA vials, unpreserved, 40mL

5.7.5 PTFE-Coated Magnetic Stir Bars

6. REAGENTS AND STANDARDS

6.1 organic-free reagent water (SOP 511)

6.2 Methanol (CH_3OH, MeOH), purge and trap grade or higher, Burdick and Jackson #230-4 or JT Baker #9077-02 or equivalent

6.3 Ottawa sand, EMD #SX0075-3 or equivalent. Pre-condition by drying in an oven set at 105°C or greater overnight

6.4 STANDARDS

6.4.1 All standards are maintained per SOP 300. In the event of a conflict, the specific guidance in this SOP will supersede that of SOP 300. Two independent sources of commercial stock standards, in methanol, are required for GRO. The stock standards are purchased as certified solutions from suitable vendors. Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampules, if recommended by the manufacturer. Standards for this procedure must be equilibrated to -10-20°C (stored in freezer) before opening. After opening/initial use, transfer remaining stock standard to a suitable vial (preferably Certan™) with minimal headspace, and store in a freezer (-10-20°C). All opened stock standards must be replaced after 6 months from date opened, or sooner, if comparisons with laboratory control samples indicate a problem.

6.4.2 First source materials are used to create calibration and continuing calibration verification (CCV) standards. Second source materials are used to create the initial calibration verification (ICV) solution. Spike standards may be from either source.

ALS typically uses a commercial gasoline *composite* standard as the GRO target stock standard. This mix is 50,000µg/mL total concentration.

Non-target analyte surrogate (SS) stock standards are also purchased. The surrogate (2,3,4-trifluorotoluene) is used to monitor system performance and method effectiveness in dealing with each sample matrix. Section 9 of this SOP gives definitions and uses of surrogates and QC (i.e., LCS/LCSD, MS/MSD) samples.

NOTE: The surrogate may be spiked in the initial calibration standards at the same concentration as it is spiked in the samples themselves. With this option, the surrogate standards in the initial calibration are averaged to produce a response factor and (effectively), a one-point calibration *with the sole purpose of measuring the surrogate recovery using the same concentration for each sample analysis*. Alternately, the surrogate can be calibrated in the same manner as the target analytes (i.e., multipoint initial calibration). If this latter option is used, an equipment validation study must be performed (if using the OI Archon autosampler) to determine the actual volume delivered by the autosampler loop. The concentration of standard may be adjusted accordingly for the actual volume delivered by the autosampler at the 1µL setting. For example: (1.135µL actual delivery) (441µg/mL IS/SS spiking solution)/5mL = 100µg/L.

6.4.3 An appropriate volume of stock standard is diluted, with methanol, to a specific volume to create intermediate standards. All dilutions should be performed using microsyringes, Class A volumetric flasks, and purge & trap grade (or higher) MeOH.

A 100µL aliquot of the GRO 50,000µg/mL stock standard is spiked into a 10mL Class A volumetric flask and diluted to the mark using methanol, to create a 500µg/mL intermediate calibration standard. The second source GRO stock standard is likewise diluted to create a 500µg/mL ICV intermediate standard.

Several intermediate SS standards may be needed:

- Automated addition is used when the OI Model 4552 Archon purge & trap autosampler is employed.

- For manual addition, a larger volume (e.g. 5mL) of a 100µg/mL intermediate standard is spiked; for automated addition, a smaller volume (e.g. 1µL) of a 500µg/mL intermediate standard is spiked.

All intermediate standards must be prepared every 3 months, or sooner if laboratory quality control analyses or other factors indicate a problem.

6.4.4 The intermediate GRO calibration standard is injected into syringes containing reagent water to create calibration working standards. Working standards (ICAL, ICV, CCV) are prepared on the day of use and documented on the sequence log. A detailed description of the concentration of the calibration standards and how they are used can be found in Section 8 of this SOP.

6.4.5 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Aqueous samples are collected in 40mL glass VOA vials with screw tops and Teflon™-lined septa. Aqueous samples should be headspace-free. Aqueous samples should be acidified to pH<2 with hydrochloric acid (HCl). Typically the addition of 3-4 drops of concentrated HCl to each 40mL VOA vial is sufficient to bring the pH of the sample to <2. It is recommended that a minimum of three vials should be collected for each field sample. For a designated MS/MSD, the client may need to provide as many as six vials.

The pH of an aqueous aliquot for each sample is measured and recorded on the sequence log at the time of analysis. If the pH of the sample is greater than 2, this is noted on the log.

7.3 Where applicable, aqueous samples should be dechlorinated with sodium thiosulfate at time of collection.

The ALS Project Manager may direct that each aqueous sample is tested for residual chlorine upon receipt at the laboratory. Notify the Project Manager immediately if residual chlorine is detected.

7.4 All samples are to be kept chilled (4±2°C).

7.5 The maximum holding time to analysis for acid-preserved aqueous samples is 14 days from date of collection. If the water sample is unpreserved, the maximum holding time to analysis is 7 days from date of collection.

7.6 If Method SW5035A direct purge is requested, solid samples must be collected in Encore™ tubes or VOA vials with preservative (as specified in SW5035A). For SW5035A methanolic extractions, or SW5035A (mod) direct purge preparation, solid samples may be collected in 125mL wide-mouth glass containers with

Teflon™-lined lids. Solid samples are not chemically preserved and must be analyzed within 14 days of collection.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 TYPICAL PURGE & TRAP DEVICE SETTINGS (OI Model 4560 concentrator)

Purge Gas Flow (He) Rate:..approximately 40mL/min

Preheat to 40°C…….2min

Purge……..……8min

Dry Purge.……1min

Desorb Preheat.…….none

Desorb.……..2min at 255°C

Bake.…….13min at 255°C

Valve.……120°C

Transfer Line.……120°C

Sample Heaters…..……40°C

NOTE: It is recommended to use the trap manufacturer's temperature parameters. Also note that the dry purge time can remove water vapor and methanol from the injection; however, if the dry purge is overextended, it may cause breakthrough and limited recovery of lighter molecular weight target compounds.

Though not required by any of the referenced methods, ALSLG-FC typically applies a heated purge to both aqueous and solid samples.

8.2 TYPICAL GC OPERATING CONDITIONS - Conditions may be altered to improve resolution of GRO compounds.

Purge and Carrier gas (He) Flow Rate……30-50mL/min

FID H_2 Flow Rate……30mL/min

FID Air Flow Rate……350mL/min

FID Temperature……..……260°C

PID Temperature……..300°C

Injector Temperature…….180°C

Initial Oven Temperature……..3min at 35°C

Initial Oven Ramp…….10°C/min to 120°C

Oven Ramp A……..…….25°C/min to 220°C

Hold……..……220°C for 2min

8.3 AUTOSAMPLER CLEANING

After use, each purge tube is removed from the autosampler and washed per SOP 334. Additionally, each purge needle is flushed with organic-free DI water, then the exterior of the needle is wiped with a KimWipe™ and MeOH.

8.4 CHROMATOGRAPHIC MAINTENANCE

8.4.1 Bake out the trap and column. Extra blanks may be necessary to achieve an adequate baseline if carryover is observed. Replace trap if performance problems implicate it and cannot be alleviated by routine maintenance.

8.4.2 If trap and front end are clean and functioning properly, clip a loop from the column or replace as necessary. In P&T applications this is rarely required, as only vapor (and thus no high boilers) is transferred to the column.

8.4.3 Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Oxidized columns will exhibit baselines that rise rapidly during temperature programming. If a column is oxidized, replacement may be necessary.

8.5 INITIAL CALIBRATION

8.5.1 Aqueous initial calibration standards are prepared at a minimum of five concentration levels by spiking varying amounts of the prepared analyte (intermediate) standard into syringes containing organic-free reagent water. The range of concentrations of the initial calibration is intended to define the working range of the analytical system (typically 50-6000ug/L). One of the concentrations must be at or below the analyte reporting limit. (See the Note in section 6.4.2 regarding initial calibration options for the surrogate standard). Typical calibration preparation is depicted below in Table 1.

8.5.2 The calibration standards are prepared on the day of use by spiking amounts of intermediate calibration standard into a syringe containing 5mL of organic-free reagent water.

TABLE 1
GRO INITIAL CALIBRATION STANDARDS

Level	µL GRO Standard (500 µg/mL)	µL Surrogate (500µg/mL)	Final Volume (mL)	Final GRO Conc. (ug/mL)	Final Surrogate Conc. (ug/mL)
ICAL 1	0.5	1uL (added by Archon)	5	0.05	0.1
ICAL 2	1	1uL (added by Archon)	5	0.1	0.1

ICAL 3	5	1uL (added by Archon)	5	0.5	0.1
CCV)	10	1uL (added by Archon)	5	1	0.1
ICAL 4	20	1uL (added by Archon)	5	2	0.1
ICAL 5	50	1uL (added by Archon)	5	5	0.1
ICAL 6	100	1uL (added by Archon)	5	10	0.1
ICV (varied)	50	1uL (added by Archon)	5	5.0	0.1

8.5.3 Electronically integrated peak area responses are tabulated and quantitated using external standard quantitation. Calibration Factors (CFs) for individual compounds are calculated as shown below. For GRO, a 'fingerprint' of peaks within an established retention time range is used for quantitation of analyte concentration (Cs), calculated as shown below:

$CF = A_s/C_s$ when using average response factor

$C_s = (A_s - b)/m$ (when using linear regression fit)

where:

A_s = response (in area counts) for the analyte to be measured

C_s = concentration of the analyte to be measured (µg/L, µg/Kg)

b = the intercept of the linear equation

m = the slope of the linear equation

8.5.4 Since each CF represents the slope of the line between the response for that standard and the origin, then if the observed deviation between the CF's is constant (i.e., ≤ 20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample content. Relative Standard Deviation (RSD) is calculated as:

$$RSD\ (\%) = \frac{\text{Standard Deviation (SD)}}{\text{Average (mean) CF}} \times 100$$

When RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be ≥0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of

6 points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration.

The type of curve fit applied should be chosen to best represent the data.

If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are discarded due to easily demonstratable causes.

If regression criteria cannot be met, a new initial calibration must be performed.

8.6 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is analyzed after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time. The acceptance criteria for the ICV are identical to those of the CCV (described below). If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated.

8.7 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end, of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum, and some clients' LIMS program specifications may require more frequent analyses of CCV's. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

The percent difference (%D, drift) must be calculated for each CCV (see equation below):

$$\%D = \left[\frac{(\text{calculate d concentrat ion}) - (\text{expected concentrat ion})}{\text{expected concentrat ion}} \right] (100)$$

Calibration is verified when GRO is ≤20%D of the expected value.

Calibration is verified when GRO is ≤20%D of the expected value. If GRO shows *elevated* response (> 20%D) and is not detected in any samples associated with the CCV, re-analyses of those samples are not necessary. If GRO shows *low* response (> 20%D) and was not detected in the samples associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If a CCV does not meet acceptance criteria, corrective action should be taken and the CCV may be reanalyzed. If the re-analyzed CCV still fails, the instrument must be recalibrated.

8.8 RETENTION TIME WINDOWS

Retention Time Windows (RTWs) for the surrogate peak are established by analyzing replicates (typically three injections) of a mid-level standard containing all single and multi-component analytes, non-consecutively, over a 72-hour period each time a new column is installed. The standard deviation of these analyses is calculated based on the absolute retention time yielded for each component. Each component's RTW is defined as the mean retention time ±3σ such that the Upper Limit = +3σ and the Lower Limit = -3σ.

RTWs should be centered on the midpoint standard if applied to samples run immediately after an initial calibration, or centered on the beginning CCV when samples are not directly preceded by an initial calibration. Sample matrices may cause drift that requires further analyst interpretation.

8.9 SAMPLE PREPARATION AND ANALYSIS (IDENTIFICATION, CALCULATIONS, REPORTING)

Samples are introduced to the GC via purge and trap. Sample aliquot size (or dilution) is chosen to maintain response within the linear quantitation range. QC sample preparation (LCS, MS, etc.) is the same as for samples.

8.9.1 PURGE TEMPERATURE

8.9.1.1 For soil analysis, the ICAL, all CCVs, and all field and QC samples shall be heated to 40°C during the purge.

8.9.1.2 For aqueous analysis, a heated purge is not required. The same purge conditions used for soil analysis may be used for aqueous analysis, however, if the ICAL, all CCVs, and all field and QC samples are heated to 40°C during the purge.

8.9.2 AQUEOUS SAMPLE ANALYSIS by Method 5030C

8.9.2.1 The process of taking an aliquot destroys the validity of aqueous samples for future analysis; therefore, if there is only one VOA vial, the analyst should prepare a second aliquot for analysis concurrently to protect against possible loss of sample integrity, or transfer the remaining sample to a 20mL VOA vial (without headspace) and refrigerate. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly.

8.9.2.2 Remove the plunger from a 5mL Luer-lock syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger, compress the sample and vent any residual air, while adjusting the sample volume to 5.0mL.

8.9.2.3 Manually add 5µL (or appropriate volume) of the surrogate mixture to each sample (if not using an autosampler, such as Archon, that adds the surrogate standard as part of the process). The addition of 5µL of the surrogate standard spiking solution to 5mL of sample is equivalent to a concentration of 100µg/L of each standard.

8.9.2.4 For matrix spike analysis, add 10µL (or appropriate volume) of the intermediate matrix spike solution to the 5mL of sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 1.0mg/L of GRO.

8.9.2.5 The sample is placed in a purge tube on the autosampler. Proceed with purge & trap analysis by Method SW5030C. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the calibrated range of the instrument, the sample must be reanalyzed at a higher dilution.

8.9.2.6 When a sample is analyzed that has GRO over ICAL range, this analysis must be followed by an organic-free reagent water blank analysis. If the blank analysis is not free of GRO, the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences. In instances where a sample response is out of range, investigation must be performed to ensure that ensuing sample results are not biased by carryover.

When high levels of analytes are observed on a multi-position autosampler, a dry tube is installed on that position and purged on

the next pass to demonstrate lack of contamination for that position.

8.9.2.7 The following procedure is appropriate for diluting aqueous purgeable samples. Sample dilution is based on analyte concentration or the presence of surfactants (foaming samples). All steps must be performed without delay until the diluted sample is in a gas-tight syringe. Since GRO is the only target being analyzed for, it is not necessary to keep diluted quantitations in the upper half of the ICAL range.

- Dilutions may be made in volumetric flasks or 5mL Luer-lock syringes. Select that which will allow for the necessary dilution. Serial dilutions may be necessary for extremely large dilution factors.

- Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

- Inject the proper aliquot of sample from a syringe into the flask. Dilute to the mark with organic-free reagent water. Cap the flask and invert three times.

- If using a volumetric flask, fill a 5mL syringe with the diluted sample.

8.9.3 SOIL SAMPLE ANALYSIS BY METHOD SW5035Amod

8.9.3.1 Homogenize the sample well, taking care to minimize the loss of volatile constituents.

8.9.3.2 Weigh 1g of soil into an appropriate purge vessel and add a PTFE-coated stir bar; place the sample on the autosampler. For method blanks and LCSs, 1g of clean Ottawa sand should be added to the purge vessel.

8.9.3.3 Add 5mL of organic-free water to the sample. In the case of LCS or MS samples, the associated spike is added with this aliquot.

8.9.3.4 The following procedure is appropriate for diluting soil purgeable samples. Soil sample dilution is based on analyte concentration. Since GRO is the only target being analyzed for, it is not necessary to keep diluted quantitations in the upper half of the ICAL range.

Soil dilutions can be made by weighing an aliquot of less than 1g of sample into the purge tube. To ensure a representative sample aliquot, no less than 0.5g of soil should be purged. For reporting purposes, a nominal amount of 1g will be considered the purge amount, and amounts less than this will be treated as dilutions. If a dilution greater than can be obtained by 0.5g of soil is required, a medium level extraction must be performed (see Section 8.10.4 below)

8.9.4 MEDIUM LEVEL SOIL SAMPLES (METHANOL-EXTRACTION)
Methanolic extraction/analysis is used for high concentration solid samples requiring dilutions greater than that which can be soundly achieved using smaller sample volume, or for samples that are difficult to homogenize.

8.9.4.1 Homogenize the sample as well as possible, taking care to minimize the loss of volatile constituents.

8.9.4.2 Weigh an aliquot of sample (from 1g to 5g, depending on sample concentration or density) into a labeled, tared 20mL VOA vial. Clean the outer lip of the vial with a Kimwipe™. Record weight to nearest 0.01g.

8.9.4.3 Add 5mL of methanol, cap and shake vigorously for 2 minutes. Allow solid and methanol to separate for at least 10 minutes (for samples with high silt or clay content, centrifugation may be necessary). Enough methanol must be added to the vial to completely cover the sample aliquot.

8.9.4.4 Calculate the volume of the methanol extract that, when brought to a final volume of 5mL in water, will bring the dilution concentration into calibration range. To protect the system from trap or column overload, a maximum of 100µL of methanol may be used. The dilution is prepared in the glass Luer-lock syringe used to transfer the sample to the purge vessel. Proceed with the analysis as discussed for aqueous samples above.

8.9.4.5 A methanol blank should be prepared in the same manner using clean Ottawa sand and 5mL of methanol. It should be analyzed with the sample extract, to ensure no contamination in the methanol.

8.9.5 GRO IDENTIFICATION
Concentration of GRO in the sample is calculated using the sum of all peak responses (excluding surrogates) from the beginning of the 2-methuyl pentane peak to the end of the 1,2,4-trimethyl benzene peak..

8.9.6 LOW/MEDIUM-LEVEL QUANTITATION

The following external standard quantitation formula is employed where CFs are used for calibration:

$$\text{Sample GRO}_{\mu g/L \text{ or } \mu g/ Kg} = \frac{(A_x)(DF)(V_f)}{(\text{avg CF})(V_s \text{ or } W_s)}$$

where:

A_x = Summed GRO peak response in sample, in area units

DF = Dilution factor (if applicable); if no dilution was made, D=1 (dimensionless)

V_f = Final volume of sample/reagent water used, L

CF = Average (mean) FF from latest initial calibration

V_s or W_s = Volume or Weight of sample purged, L or Kg

8.9.7 METHANOL-EXTRACTED SAMPLE QUANTITATION

The following external standard quantitation formula is employed where CFs were used for calibration:

$$\text{Sample GRO}_{\mu g/L \text{ or } \mu g/Kg} = \frac{(A_x)(V_t)(DF)(V_f)}{(\text{mean CF})(V_s \text{ or } W_s)(V_i)}$$

where:

A_x = Summed GRO peak response in sample, in area units

DF = Dilution factor (if applicable); if no dilution was made, D=1 (dimensionless)

V_f = Final volume of sample/reagent water used, L

mean CF = Average (mean) RF from latest initial calibration, mg/area

V_s or W_s = Volume or Weight of sample extracted, L or KG

V_t = Total volume of methanol extract, mL

V_i = Volume of extract used for purging, mL

8.9.8 QUANTITATION WHERE CALIBRATED BY LINEAR REGRESSION

Where linear regression is employed, quantitation of sample concentration is based on the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)(V_t)(DF)}{m(V_s \text{ or } W_s)}$$

where:

x = concentration of the analyte

y = analyte instrument response (area units)

b = calculated intercept

m = calculated slope of the line

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

V_s or W_s = volume or weight of sample extracted (mL or g)

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specifications for additional or alternative requirements.

9.2 BLANKS

Method Blanks (MBs) are aliquots of matrix (i.e., organic-free water for liquids, Ottawa sand for solids) that have been prepared and analyzed in the same manner as the associated field samples. MBs are analyzed to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Concentrations of target analytes, if any, must be less than the reporting limit (RL), or as otherwise prescribed in the LIMS program specification. If this criterion is not achieved, then analyses must be halted and the source of the contamination found and corrected.

9.3 LABORATORY CONTROL SAMPLE

The LCS is analyzed to measure the accuracy of the analytical system. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

NOTE: For this procedure, the preparation and analysis steps are the same for all standards and samples, therefore, the analysis of a single spiked blank matrix (reagent water or Ottowa sand), provides results for both the LCS and CCV simultaneously.

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample and/or matrix spike sample is performed in duplicate (LCSD, MSD). The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. Percent Recovery (%R) for spiked analytes is calculated as follows (see QC Table for evaluation criteria):

$$\%R = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right) (100)$$

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation is made in the data package narrative.

9.6 SURROGATE RECOVERY

The %R of the surrogate is calculated (see equation shown for LCS recovery above) for all field and QC samples. If the recovery is outside QC limits, the sample is reanalyzed to verify that analytical error was not the problem. If after re-injection the recovery is still out-of-control, initiate an NCR (SOP 928) and apply the decided upon action. Typically, matrix effect is cited as the cause and the occurrence is narrated. However, the client may direct that the sample be re-extracted and reanalyzed (holding time is a consideration).

9.7 METHOD DETECTION LIMIT STUDY

The Detection Limit (DL/LOD) is performed as needed, at a minimum, annually, following the guidance of SOP 329.

10. DEVIATIONS FROM THE METHOD

10.1 Method 8015D (Section 11.4.2) specifies the calculation of RTW's for the first and last peaks used to define the GRO, and the subsequent setting of the GRO window to include those calculated windows and everything in between. ALS will use the beginning CCV to set the GRO window (to include 2-methyl pentane through 1,2,4-trimethylbenzene) and then use each CCV thereafter to verify that the window is valid for the peaks of interest (but will not actually *calculate* RTW's for the beginning and end peaks).

10.2 Heated purge of aqueous samples is not required by SW5030C. ALS may perform heated purging of aqueous samples.

10.3 CAL-LUFT specifies a %D criterion of ±10% for daily calibration verification. ALS defaults to the criteria listed in Method 8015D of ±20%D, unless otherwise required by client or project specific needs (as indicated in the LIMS Project Specification).

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 USEPA, SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, Volume 1B, Method 8015B, Revision 2, December 1996.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8015D", Revision 4, June 2003.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12.4 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 5035A", DRAFT Revision 1, July 2002.

12.5 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 5030C", Revision 3, May 2003.

12.6 California LUFT Field Manual, October 1989 update.

TABLE 2

Analytical Method: SW8015B or D	**Parameter:** Total Volatile Petroleum Hydrocarbons		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL); minimum 5-points, all analytes	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, may use mean RF to quantitate, If RSD ≥20%, calculate linear regression (not forced through origin); use for quantitation if correlation coefficient (r) ≥0.995 or calculate quadratic regression (minimum of six points required); use for quantitation if COD (r^2) ≥0.99	Evaluate/correct instrument malfunction and reanalyze ICAL to obtain acceptable curve
Initial Calibration Verification (ICV); conc. not equal to midpoint of calibration curve; second source	After each ICAL	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.
Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Run at start of sequence if ICAL not performed; brackets each set of 20 field sample analyses; more frequent analysis recommended.	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g., rinse or change liner, remove some of the guard column); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. Samples analyzed before and after a failed CCV must be reanalyzed. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV (bracketed by acceptable CCVs) will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.

Analytical Method: SW8015B or D	**Parameter:** Total Volatile Petroleum Hydrocarbons		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time Window (RTW); The retention time range for GRO includes 2-methly pentane through 1,2,4 trimethlybenzene, window is checked against CCV for each batch.	Whenever a new column is installed; and checked with each batch	Note that the ICV and CCV analyses are also used to monitor RT drift	Width of GRO window should include 2-methyl pentane and 1,2,4-trimethylbenzene of bracketing CCVs.
Retention Time Shift; RT of analytes in CCV are evaluated against the midpoint of the ICAL	Each CCV	Column and compound specific	Inspect chromatographic system for malfunction; correct identified malfunctions, if appropriate Evaluate data based on comparison with other standards run during sequence, consider RTs for the surrogates and spiked compounds analyzed before and after the sample in question:
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix NOTE: Methanol extracts additionally require a methanol MB.	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition
Blank Spike (BS); Laboratory Control Sample (LCS)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds must be within laboratory limits or other limits as specified in the LIMS program specification	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if the samples are beyond the extraction holding time, then contact PM via NCR for sample

Analytical Method: SW8015B or D	**Parameter:** Total Volatile Petroleum Hydrocarbons		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration
Matrix Spike (MS)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per preparation batch of ≤20 samples of like matrix	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within advisory limits.	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. Note in narrative. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers. Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental conditions were the cause. - if still non-compliant and the samples are within the extraction holding time, initiate an NCR (associated samples may be reanalyzed)
Surrogate Spike	All extractions including field and laboratory QC samples	See laboratory limits; recoveries should be within current limits alternative criteria as defined in the LIMS program specifications may apply	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for

Analytical Method: SW8015B or D	**Parameter:** Total Volatile Petroleum Hydrocarbons		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
			the low recovery. - if surrogate recovery in the associated MB is not within limits and the samples are within the holding time, then re-extract and reanalyze all associated samples - if samples are beyond the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.

ALS Standard Operating Procedure

DOCUMENT TITLE:	ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY
REFERENCED METHOD:	SW8151A, EPA 615 AND EPA 515.1
SOP ID:	434
REV. NUMBER:	11
EFFECTIVE DATE:	AUGUST 12, 2013

ALS Environmental

STANDARD OPERATING PROCEDURE 434 REVISION 11

TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GAS CHROMATOGRAPHY -- METHODS SW8151A, EPA 615 AND EPA 515.1

FORMS: NONE (instrument printout used as run log)

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operation Procedure (SOP) and the methods it references, SW8151A, EPA 615 and EPA 515.1, are used to determine the concentration of chlorinated herbicides in liquid (all) and solid matrices (SW8151A). The following compounds typically comprise ALS Laboratory Group – Fort Collins (ALS)'s target analyte list:

Dalapon	Silvex
Dicamba	2,4,5-T
MCPP	2,4-DB
MCPA	Dinoseb
Dichloroprop	2,4-Dichlorophеylacetic acid
2, 4-D	

Other compounds may be analyzed if successful method detection limit (MDL) and demonstration of capability (DOC) studies are performed.

2. SUMMARY

The target herbicides are commonly applied as either an amine salt (usually trimethyl amine) or one of many esters of the base compound. These are easier to handle than the free acids. Hydrolysis, to covert any esters to free acids, is included in the sample preparation process. Samples are extracted, esterified and the extracts concentrated and solvent exchanged using appropriate ALS SOPs (i.e., 664, 607, 637). The extracts are injected into a gas chromatograph (GC) containing a sample splitter and two columns of varying selectivity (i.e., dissimilar RT elution properties). The target analytes are separated in the columns and detected by two electron capture detectors (ECDs). This chromatography system allows tentative identification by one column and confirmation by the other column to be performed simultaneously. Quantitation is performed using the best column for each analyte. The analyst considers performance data such as separation of analytes and interferences, calibration performance and matrix spike results in selecting the quantitation

column for each analyte detected and reported. If results from both columns are comparable, the highest result is reported.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the benchsheet indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.4 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, or other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. Only high purity solvents and reagents are used; prescriptive measures for cleaning glassware are detailed in SOP 334. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis as evidenced by the analysis of interference-free reagent blanks.

4.2 The target herbicides are strong organic acids that react readily with alkaline substances and may be lost during sample preparation steps.

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being analyzed.

4.4 Organic acids, especially chlorinated acids, cause the most direct interference (due to reaction products formed in the methylation sample preparation step). Phenols, including chlorophenols, may also interfere.

4.5 Sample extracts must be free of water prior to methylation or poor recoveries will be obtained. The sodium sulfate drying agent must be acidified before use.

4.6 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might interfere with the electron capture analysis. However, hydrolysis might result in limited recovery of dinoseb (cleavage of alkyl group) and dalapon (hydrolysis to pyruvic acid).

4.7 Historically, interferences are present for some herbicides, and are known to co-allude with target compounds, especially near DCAA.

4.8

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH, AUTOSAMPLER, DETECTORS
Hewlett Packard (HP) 5890 Series II GC equipped with HP7673A autosampler and dual electron capture detectors (ECDs), or equivalents

5.2 CHROMATOGRAPHIC DATA ACQUISITION/PROCESSING SYSTEM
Hewlett Packard ChemStation (Enviroquant™) or equivalent

5.3 COLUMNS - Equivalent columns may also be used

Restek Pesticide Column:	RTX-CLPesticides	#11123	(30m, 0.25mm ID, 0.5µm film)
Restek Pesticide (Guard) Column:	RTX-CLPesticides2	#11323	(30m, 0.25mm ID, 0.5µm film)
Restek Pesticide Column:	RTX-CLPesticides	#11139	(30m, 0.32mm ID, 0.25µm film)
Restek Pesticide (Guard) Column:	RTX-CLPesticides2	#11324	(30m, 0.32mm ID, 0.25µm film)

5.4 GASES - ultra high purity (99.999%)
Helium - carrier gas
Nitrogen - make-up gas

5.5 MEASURING DEVICES
Syringes - 10-1000µL
Volumetric flasks, Class A with stoppers, 10-100mL

5.6 GC CONSUMABLES
- Vials - National Scientific C4011-1, or equivalent
- Caps - National Scientific C4000-51B, or equivalent
- Inlet Seals - dual Vespel ring, 0.8mm, Restek #21243, or equivalent

- Septa - 11mm, Restek #20365, or equivalent
- O-ring - graphite, 6.5mm, Restek #20299, or equivalent
- O-ring - Viton™, Restek #20377, or equivalent
- Liner - splitless, 4mm ID, Restek #20799-214.5, or equivalent
- Glass Wool - deactivated, Restek #20789, or equivalent
- Gold Seal - Restek #21306, or equivalent
- Universal Presstight Y - Restek #20469, or equivalent
- Vespel/Graphite Ferrule (detector) - Restek #20221, or equivalent
- Compact Vespel/Graphite Ferrule - Restek #20249, or equivalent
- Graphite ferrules - various sizes
- Split Vent Trap (SW8081) - Agilent RDT-1023, or equivalent

6. REAGENTS AND STANDARDS

6.1 SOLVENTS - Only pesticide grade solvents may be used!

Methanol (CH_3OH, MeOH) - Burdick and Jackson #230-4, or equivalent

n-Hexane (C_6H_{14}) - Burdick and Jackson #216-4, or equivalent

Diethyl ether ($C_2H_5OC_2H_5$) - Burdick & Jackson #106-4, or equivalent. Must be peroxide-free. If preserved, stabilized with BHT (not ethanol), or unpreserved.

Methyl tert butyl ether $(CH_3)_3COCH_3$, MTBE - JT Baker #9043-02, or equivalent -- Method EPA 515.1 only

6.2 STANDARDS

6.2.1 Storage and Documentation - All standards are maintained per SOP 300, which is superceded by any guidance in this SOP. Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampules, if recommended by the manufacturer. Generally after opening ampules, the standards for this procedure are stored in the freezer (-10--20°C), in PTFE-capped, or equivalent, vials. Opened stock standards and intermediate standards expire six months from opening (preparation) or per the manufacturer's expiration date (whichever is sooner). Standards may be replaced sooner if laboratory quality control (QC) analyses or other factors indicate deterioration.

6.2.2 Calibration and Spike Standards - At minimum, two independent sources (first, second) of commercial stock standards are needed for target analytes. These certified stock standards are purchased from suitable vendors as free acid mixes (first source) and methyl esters (second source). First source materials are used to create calibration, continuing calibration verification (CCV) and QC sample spike standards. Second source materials are used to create the initial calibration verification (ICV) solution (used to independently verify the accuracy of the initial calibration, ICAL). Where commercially-derivatized methyl esters are

used to prepare standards, the concentrations must be corrected back to the free acid form. A (non-target analyte) surrogate stock standard is also purchased. The surrogate used for this method is 2,4-dichlorophenylacetic acid (DCAA). Section 9 of this SOP gives definitions and uses of surrogates and QC (i.e., LCS/LCSD, MS/MSD) samples.

An appropriate volume of stock standard is diluted (in hexane) to a specific volume to create intermediate standards (the QC sample and surrogate spike standards, used by the Organics Extraction Group, are intermediate standards). The intermediate calibration standards are further diluted to volume using an appropriate solvent to create working standards. Working standards are prepared on the day of use and documented in the analytical run log (Form 530). A detailed description of the concentration of the calibration standards and how they are used can be found in Section 8 of this SOP.

6.2.3 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING, HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Liquid samples are generally not chemically preserved for SW8151A analysis and must be collected in amber glass containers (generally 1000mL) with Teflon™-lined lids. Samples must be maintained at 4±2°C and extracted within 7 days of collection (14 days for EPA 515.1). Sodium thiosulfate ($Na_2S_2O_3$) may be used to dechlorinate liquid samples such as drinking water or chlorinated wastewater. This should be accomplished by the client in the field. The Project Manager may designate the need for residual chlorine check of the sample upon receipt. Additionally, samples for EPA Method 608 may need to have pH adjusted upon receipt.

7.3 Solid samples are collected in wide-mouth glass containers with Teflon™-lined lids. Solid samples are not chemically preserved and must be maintained at 4±2°C. Solid samples must be extracted within 14 days of collection.

7.4 Extracts, from liquid or solid samples, must be refrigerated and analyzed within 40 days of preparation.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 TYPICAL GAS CHROMATOGRAPHIC CONDITIONS

Carrier gas (He):	2.2 mL/min
Make-up gas (N_2):	20-40 mL/min
Injection port temperature:	205°C
Injection volume:	1-2µL, splitless
Detector temperature:	325°C
Initial oven temperature:	50°C, hold 4min
Oven ramp A:	15°C/min to 130°C
Oven ramp B:	7°C/min to 190°C, hold 3.0min
Oven ramp C:	20°C/min to 320°C, hold 7.0min

The conditions used for sample analysis must be the same as the conditions used during initial calibration**.**

8.2 SYSTEM MAINTENENANCE AND PREPARATION

Because of the low concentrations injected on a GC/ECD, column adsorption (active sites) may be a problem when the GC has not been used for a day or more. Therefore, the GC column may need to be conditioned by injecting a suitable prime, such as mid to high concentrations of expired standards, prior to calibration. Solvent blanks are typically injected following the priming to demonstrate that the system is free from carryover.

8.3 INITIAL CALIBRATION

8.3.1 Prepare a minimum of 5 concentrations of calibration standards, defining the linear range of the detector. The lowest concentration standard shall be at a level at or below the reporting limit (RL) of each analyte. Create calibration standards by diluting aliquots of the intermediate calibration standard using hexane. The mid-range calibration standard is used for continuing calibration verification (CCV). For herbicides analysis, the calibration range varies contingent upon the target analyte. Calibration standards are prepared with surrogate standards at similar levels to target analytes. A typical calibration sequence and preparation steps are shown below in Table 1.

TABLE 1
CALIBRATION STANDARDS

Working Standard	Hexane (µL)	Intermediate Standard (µL)	Standard Concentration (ng/mL)
ICAL Level 6 (30%)	700	300	varies per analyte
ICAL Level 5 (25%)	750	250	varies per analyte
ICAL Level 4 (20%)	1000	250	varies per analyte
ICAL Level 3 (10%)	900	100	varies per analyte
ICAL Level 2 (5%)	950	50	varies per analyte
ICAL Level 1 (2.5%)	975	25	varies per analyte

2nd source ICV (10%)	900 varied over time	100 varied over time	varies per analyte
CCV (20%)	1000	250	varies per analyte

8.3.2 Inject and analyze 1µL of each calibration standard under the GC conditions listed previously. Each data file quantitation is accomplished via the external standard method of quantitation. Analyte Calibration Factors (CFs) are calculated as follows:

$$CF = \frac{\text{integrated peak area of analyte}}{\text{analyte concentration}}$$

Since each CF represents the slope of the line between the response for that standard and the origin, then if the CFs over the working range of the detector are constant (i.e., ≤20% RSD), then if the observed deviation between the CF's is constant (i.e., ≤20% RSD), then the response is assumed to be invariant and the average (mean) CF may be used to quantitate sample content.

Relative Standard Deviation (RSD) is calculated as:

$$RSD\ (\%) = \left(\frac{\text{standard deviation of the analyte's response factors}}{\text{mean response factor}} \right)(100)$$

When RSD over the calibration range is greater than 20%, linearity through the origin cannot be assumed. A first or second order regression fit of five or more calibration points that does not pass through zero (e.g., least squares method) may be constructed. The regression calculation will yield a coefficient of determination (r^2 value) that must be ≥0.99 to be used for sample quantitation. Note that the coefficient of determination (COD) is an expression of "goodness of fit", with perfect fit being a value of 1.0. Non-linear (quadratic) regression curve fitting is also allowed, with a minimum of 6 points, following the guidelines in SW-846 Method 8000C. A quadratic regression should not be used to compensate for detector saturation.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weight the curve to either the inverse of the concentration or to the inverse of the square of the concentration

The type of curve fit applied should be chosen to best represent the data.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

If regression criteria cannot be met, a new initial calibration must be performed.

8.4 INITIAL CALIBRATION VERIFICATION (ICV)

A second source ICV standard is injected after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time. The acceptance criteria for the ICV are identical to those of the CCV (described below). If the below criteria are not met, the ICV shall be remade and analyzed to verify true concentration. If the ICV still fails, a new initial calibration must be generated. Exception: ALS has noted a consistent comparative elevated response for dinoseb and subdued response for dalapon between the first and second source vendor standards used. ALS narrates this occurrence in the data package narrative.

8.5 CONTINUING CALIBRATION VERIFICATION (CCV)

The concentration of the CCV is at or near the midpoint of the initial calibration. A CCV check is performed at the beginning (when initial calibration is not performed) and end, of each 12-hour analytical sequence (or batch of 20 samples). While QC samples are counted as part of the number of samples, solvent blanks are not.

NOTE: The CCV frequency given above should be considered a bare minimum, and some clients' LIMS program specifications may require more frequent CCV analysis. Even if this is not the case, a higher CCV frequency is strongly recommended. ALS typically analyzes a CCV every 10 samples in order to reduce the amount of repeat injections necessary in the event of CCV failure.

Calibration is verified when all compounds are ≤20%D, when calculated as shown below:

$$\%D = \left[\frac{(\text{calculated concentration}) - (\text{expected concentration})}{\text{expected concentration}} \right] (100)$$

NOTE: Method 515.1 provides guidance that the concentration of calibration checks should be varied with one below the midpoint and one above the midpoint of the calibration range.

If a compound shows *elevated* response (> 20%D) and is not detected in any samples associated with the CCV, re-analysis of those samples are not necessary. If a compound shows *low* response (> 20%D) and was not detected in the samples

associated with the CCV, reinjection is necessary to ensure that a false negative result is not reported.

If any CCV does not meet acceptance criteria, analyses should be halted and corrective action taken. Reanalyze the CCV. If the CCV still fails, the instrument must be recalibrated and all samples injected since the last compliant CCV must be reanalyzed

8.6 RETENTION TIME WINDOWS

For GC methods, retention times are used for analyte identification. Retention Time Windows (RTWs) are established by analyzing replicates (typically three injections) of a mid-level standard containing all single and multi-component analytes, non-consecutively, over a 72-hour period each time a new column is installed. The standard deviation of these analyses is calculated based on the absolute retention time yielded for each component. Each component's RTW is defined as the mean retention time $\pm 3\sigma$ such that the Upper Limit = $+3\sigma$ and the Lower Limit = -3σ.

RTWs should be centered on the midpoint standard if applied to samples run immediately after an initial calibration, or centered on the beginning CCV when samples are not directly preceded by an initial calibration. Sample matrices may cause drift that requires further Analyst interpretation. In the chromatography data system, RTWs and integration parameters should be set to err on the side of false positives, so target compounds are not missed by the data system. See Appendix I for chromatographic interpretation examples.

8.7 SAMPLE ANALYSIS (IDENTIFICATION, CALCULATIONS, REPORTING)

A constant volume, generally 1µL of each standard, blank, QC or field sample extract is directly injected into the GC via the automated injector. Sample extracts are diluted to maintain response within the linear range when necessary. All prepared extracts contain the surrogate (see Section 9). Extract and sample concentration are determined as discussed below.

8.7.1 Note that ALS employs a sample splitter that facilitates simultaneous dual column injections; therefore, both columns are calibrated in the same manner and either column may serve as the column for quantitation.

8.7.2 Tentative identification occurs when a peak from a sample extract falls within the RTW of one column. If the peak also falls within the RTW of the second column, then the analyte's presence has been confirmed. Quantitation is calculated from both column responses and the best result is reported. The analyst considers performance data such as separation of interferences, calibration performance and matrix spike results in selecting the quantitation column for each analyte detected and reported. If there are interferences present on one column, these need to be documented by printouts of that region of the chromatogram; the lower (i.e., other column's) results may be reported in this case. If results from both columns are of comparable quality, the analyst has discretion regarding

which result to report if an interference is historically known to be present on a specific column. If no interferences are known to be present, the higher concentration is reported.

8.7.3 the higher concentration is reported (per SW8000C).

8.7.4 Sample concentration is calculated using the equation of the linear curve generated during initial calibration (i.e., y = mx + b), as follows:

$$x = \frac{(y - b)\,(V_t)(DF)}{m(V_s \text{ or } W_s)}$$

where:

x = concentration of the analyte

y = analyte instrument response (area units)

b = calculated intercept

m = calculated slope of the line

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

V_s or W_s = volume or weight of sample extracted (mL or g)

If the average CF is used for quantitation, the sample concentration is calculated using the following equation:

$$\text{Concentration}_{(ug/L,\ ug/kg)} = \frac{(A_x)(V_t)(DF)}{(\text{Average CF})(V_s \text{ or } W_s)}$$

where:

A_x = analyte response (area units)

V_t = total volume of concentrated extract (mL)

DF = Dilution Factor (if applicable); if no dilution, then DF = 1

Average CF = average calibration factor

V_s or W_s = volume or weight of sample extracted (mL or g)

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

A batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples

should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specifications for additional or alternative requirements.

9.2 BLANKS

MBs are aliquots of matrix (i.e., organic-free water for liquids and acidified boiling chips for solids) that have been prepared and analyzed in the same manner as the associated field samples. MBs are analyzed to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Concentrations of target analytes, if any, must be less than the reporting limit (RL), or as otherwise prescribed in the LIMS program specification. If this criterion is not achieved, then analyses must be halted and the source of the contamination found and corrected. As the analyst deems necessary, aliquots of solvent may be injected to clean the analytical system and demonstrate that it is free from contamination.

9.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the analytical system. The LCS is similar to the matrix spike analysis in that known concentrations of target analytes are spiked into reagent matrix (as opposed to sample matrix, as with the MS) and the percent recoveries for the analytes are calculated as shown below. See QC Table for evaluation criteria.

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this measurement, the laboratory control sample and/or matrix spike sample is performed in duplicate (LCSD, MSD). The results of the duplicate analyses are evaluated in terms of Relative Percent Difference (RPD), which is calculated as shown below. See QC Table for evaluation criteria.

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

9.5 MATRIX SPIKE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. See QC Table for evaluation criteria. Percent Recovery (%R) for spiked analytes is calculated as follows:

$$\%R = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right)(100)$$

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation is made in the data package narrative.

9.6 SURROGATE RECOVERY

The %R of the surrogate is calculated (see equation shown for LCS recovery above) for all field and QC samples. If the recovery is outside QC limits, the sample is reanalyzed to verify that analytical error was not the problem. If after re-injection the recovery is still out-of-control, initiate an NCR (SOP 928) and apply the decided upon action.

9.7 METHOD DETECTION LIMIT STUDY

A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven (7) replicates for each target analyte at a concentration level near to the capabilities of the method. The MDL study is performed as needed, at a minimum, annually, following the guidance of SOP 329.

10. DEVIATIONS FROM METHOD

This SOP meets the requirements of Methods SW8151A, EPA 615 and EPA 515.1. Performance data substantiating the use of Soxhlet extraction as an appropriate preparative technique for solid samples is on file with ALS's Quality Assurance Department.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods For Evaluating Solid Waste -- Physical/Chemical Methods, 3rd edition, Final Update III, "Method 8151A", Revision 2, December 1996.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12.3 US EPA Method 515.1, "Determination of Chlorinated Acids in Water by Gas Chromatography with Electron Capture Detector".

12.4 US EPA Method 615, "The Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater".

12.5 ALS Technical Report: "Soxhlet Extraction of Herbicides in Soil", Steven Ignelzi, July 2006.

Analytical Method: SW8151A, EPA 615 & 515.1	**Parameter:** Analysis of Chlorinated Herbicides by Gas Chromatography		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL); minimum 5-points, all analytes (SW 8151A); Methods EPA 615 & 515.1 do not require 5-points	As needed (i.e., when daily calibration verification does not meet criteria)	When RSD ≤20%, may use mean RF to quantitate Calculate linear regression (not forced through origin); use for quantitation if correlation coefficient (r) ≥0.995 or Calculate quadratic regression (minimum of six points required); use for quantitation if COD (r^2) ≥0.99	Evaluate/correct instrument malfunction and reanalyze ICAL to obtain acceptable curve
Initial Calibration Verification (ICV); conc. not equal to midpoint of calibration curve; second source	After each ICAL	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Prepare another ICV and analyze. If second ICV fails, system must be recalibrated with freshly prepared standards.
Continuing Calibration Verification (CCV); analyzed at midpoint of calibration curve	Brackets each set of 20 field sample analyses (standard practice is every 10 samples injections)	≤20%D of each compound or as otherwise specified in applicable LIMS program specification	Evaluate/correct instrument malfunction as needed (e.g., rinse or change liner, remove some of the guard column); prepare a new standard and reanalyze. - If CCV still non-compliant, recalibrate. Samples analyzed after a failed CCV must be reanalyzed. - if target(s) in CCV fails high (>20%) and target is not present in samples, re-analyses of samples are not necessary. - If a failed CCV for an autosampler analysis returns to acceptable calibration later in the sequence, samples following the acceptable CCV will be reported, and samples between the failed CCV and subsequent compliant CCV will be reanalyzed. - If holding times are an issue, complete a Non Conformance Report (NCR) and notify the PM for sample disposition.

Retention Time Window (RTW); based on minimum of 3 non-consecutive injections throughout at least a 72-hour period to be representative of variation	Whenever a new column is installed; based on at least 3 injections throughout a 72-hour period.	Column and compound specific. Window is ±3x the standard deviation of the 3-injection average for the respective column Note that the ICV and CCV analyses are also used to monitor RT drift	If SD=zero, then either do additional injections or use a default SD of 0.01 minutes. Experience of analyst weighs heavily in interpretation of chromatograms (refer also to RT Shift).
Retention Time Shift; RT of analytes in CCV are evaluated against the midpoint of the ICAL		Column and compound specific	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are outside the extraction holding time, then complete an NCR and contact PM for sample disposition.
Method Blank (MB)	1 per preparation batch of ≤20 samples of like matrix	<RL: MB should not contain any target compounds at or above the reporting limit (RL) or per other criteria as specified in the applicable LIMS program specification	Reanalyze to determine if instrument contamination was the cause. If MB still non-compliant, initiate corrective action: - if a sample contains target compounds at ≥10X amount found in MB or if target compounds are not detected in the sample, then results may be reported; otherwise - if the samples are within the extraction holding time, then re-extract and reanalyze all associated samples containing target compounds at <10X amount found in MB - if the samples are beyond the extraction holding time, then complete an NCR and contact PM for sample disposition
Blank Spike (BS); Laboratory Control Sample (LCS)	1 per each preparation batch of ≤20 samples of like matrix	See laboratory limits; recoveries for spiked compounds must be within laboratory limits or other	Check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze to determine if instrumental

		limits as specified in the LIMS program specification	conditions was the cause. - if still non-compliant and the samples are within the extraction holding time, then request re-extraction using an NCR, and reanalyze all associated samples for the analyte that do not meet criteria - if the samples are beyond the extraction holding time, then contact PM via NCR for sample disposition. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration
Matrix Spike (MS)	1 per batch, not to exceed 20 samples of like matrix	See laboratory limits; recoveries for spiked compounds should be within advisory limits	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated BS (LCS) is within limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD) or Laboratory Control Sample Duplicate (LCSD)	1 per batch, not to exceed 20 samples of like matrix	See laboratory limits; see Matrix Spike information above for MSD recoveries. RPDs should be within advisory limits.	See Matrix Spike actions above for recoveries outside of advisory limits. If RPDs for the spiked compounds are not within advisory limits, check for documentable errors (e.g., calculations and spike preparation). Check unspiked sample results and surrogate recoveries for indications of matrix effects. If significant differences between the MS and MSD exist, reanalysis of the sample and spikes may be necessary. Discuss with Department/ Project/QA Managers.
Surrogate Spike	All extractions including field and laboratory QC samples	See laboratory limits; recoveries should be within current limits; alternative criteria as defined in the LIMS program specifications may apply	Check calculations and spike preparation for documentable errors. - if no errors are found and the surrogate recoveries in the MB and LCS are within limits, then sample matrix effects are the most likely cause. However, any samples with surrogate recovery outside the QC limits with no visible chromatographic cause, should be re-injected to determine if an injection error was the cause for the low recovery. - if surrogate recovery in the associated MB and LCS is not within limits and the samples are

			within the holding time, then re-extract and reanalyze all associated samples - if samples are beyond the holding time, then contact the PM via an NCR. Unless otherwise directed, samples will not be extracted outside of the holding time and the data will be submitted with appropriate narration.
Method Detection Limit (MDL) Study	As needed, at minimum, annually	Positive result < the analyte reporting limit (RL)	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

ALS Standard Operating Procedure

DOCUMENT TITLE:	SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY
REFERENCED METHOD:	SW8270 D
SOP ID:	506
REV. NUMBER:	20
EFFECTIVE DATE:	MARCH 6, 2014

ALS Laboratory Group (ALS)
STANDARD OPERATING PROCEDURE 506 REVISION 20

TITLE: SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY – METHODS SW8270 D

APPROVED BY:

PRIMARY AUTHOR _____ DATE 04-14-14

QUALITY ASSURANCE MANAGER _____ DATE 4-16-14

LABORATORY DIRECTOR _____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the method it references – Methods SW8270 D - describe a procedure to determine the concentration of semivolatile organic compounds (SVOCs) in extracts prepared from all types of solid waste matrices, soils, TCLP leachates and ground water. Direct injection of a sample may be used in limited applications.

The body of this SOP specifies the procedures to be used for SW-846 Methods 8270 D.

The following analytes have been successfully determined utilizing this analytical procedure after appropriate preparation methods are utilized. Other compounds may be determined after successful demonstration of capability (i.e., method detection limit studies and other demonstration of capability, as applicable). Analytes that are part of ALS's typical reporting list are presented in bold text.

TABLE 1

APPLICABLE COMPOUNDS for SEMIVOLATILE ANALYSIS by SW8270 D

COMPOUND	CAS Number
pyridine	110-86-1
N-nitrosodimethylamine	62-75-9
aniline	62-53-3
phenol	108-95-2
bis(2-chloroethyl)ether	111-44-4
2-chlorophenol	95-57-8
1,3-dichlorobenzene	541-73-1
1,4-dichlorobenzene	106-46-7
1,2-dichlorobenzene	95-50-1
benzyl alcohol	100-51-6
bis(2-chloroisopropyl)ether	108-60-1
2-methylphenol	95-48-7

TABLE 1

APPLICABLE COMPOUNDS for SEMIVOLATILE ANALYSIS by SW8270 D

COMPOUND	CAS Number
4-methylphenol	106-44-5
hexachloroethane	67-72-1
nitrobenzene	98-95-3
isophorone	78-59-1
2-nitrophenol	88-75-5
2,4-dimethylphenol	105-67-9
bis(2-choloroethoxy)methane	111-91-1
2,4-dichlorophenol	120-83-2
benzoic acid	65-85-0
1,2,4-trichlorobenzene	120-82-1
naphthalene	91-20-3
4-chloroaniline	106-47-8
hexachlorobutadiene	87-68-3
4-chloro-3-methylphenol	59-50-7
2-methylnaphthalene	91-57-6
hexachlorocyclopentadiene	77-47-4
2,4,6-trichlorophenol	88-06-2
2,4,5-trichlorophenol	95-95-4
2-chloronaphthalene	91-58-7
2-nitroaniline	88-74-4
dimethyl phthalate	131-11-3
2,6-dinitrotoluene	606-20-2
acenaphthylene	208-96-8
3-nitroaniline	99-09-2
acenaphthene	83-32-9
2,4-dinitrophenol	51-28-5
4-nitrophenol	100-02-7
dibenzofuran	132-64-9
2,4-dinitrotoluene	121-14-2
diethyl phthalate	84-66-2
fluorene	86-73-7
4-chlorophenyl phenyl ether	7005-72-3
4-nitroaniline	100-01-6
azobenzene	103-33-3
4,6-dinitro-2-methylphenol	534-52-1
N-nitrosodiphenylamine	86-30-6
4-bromophenyl phenyl ether	101-55-3
hexachlorobenzene	118-74-1
2,3,4,6-tetrachlorophenol	58-90-2
pentachlorophenol	87-86-5
phenanthrene	85-01-8
anthracene	120-12-7
carbazole	86-74-8
di-n-butyl phthalate	84-74-2
fluoranthene	206-44-0

TABLE 1

APPLICABLE COMPOUNDS for SEMIVOLATILE ANALYSIS by SW8270 D

COMPOUND	CAS Number
benzidine	92-87-5
pyrene	129-00-0
butyl benzyl phthalate	85-68-7
benzo(a)anthracene	56-55-3
3,3'-dichlorobenzidine	91-94-1
chrysene	218-01-9
bis(2-ethylhexyl)phthalate	117-81-7
di-n-octyl phthalate	117-84-0
benzo(b)fluoranthene	205-99-2
benzo(k)fluoranthene	207-08-9
benzo(a)pyrene	50-32-8
indeno(1,2,3-CD)pyrene	193-39-5
dibenzo(a,h)anthracene	53-70-3
benzo(g,h,i)perylene	191-24-2
2-acetylaminofluorene	53-96-3
acetophenone	98-86-2
4-aminobiphenyl	92-67-1
aramite	140-57-8
atrazine	1912-24-9
benzaldehyde	100-52-7
1,1'-biphenyl	92-52-4
caprolactam	105-60-2
chlorobenzilate	510-15-6
1-chloronaphthalene	90-13-1
diallate	2303-16-4
dibenz(a,j)acridine	224-42-0
2,6-dichlorophenol	87-65-0
dimethoate	60-51-5
4-dimethylaminoazobenzene	60-11-7
N,N-dimethylaniline	121-69-7
7,12-dimethylbenz(a)anthracene	57-97-6
3,3'-dimethylbenzidine	119-93-7
A,A-dimethylphenethylamine	122-09-8
1,2-dinitrobenzene	528-29-0
1,3-dinitrobenzene	99-65-0
1,4-dinitrobenzene	100-25-4
disulfoton	298-04-4
N-ethylaniline	103-69-5
ethyl methanesulfonate	62-50-0
ethyl parathion	56-38-2
famphur	52-85-7
hexachloropropene	1888-71-7
isodrin	465-73-6
isosafrole	120-58-1
kepone	143-50-0
N-methylaniline	100-61-8

TABLE 1

APPLICABLE COMPOUNDS for SEMIVOLATILE ANALYSIS by SW8270 D

COMPOUND	CAS Number
tetramethylurea	632-22-4
4-nitroquinoline-n-oxide	56-57-5
N-nitrosodi-n-butylamine	924-16-3
N-nitrosodiethylamine	55-18-5
phorate	298-02-2
pronamide	23950-58-5
safrole	94-59-7
methapyrilene	91-80-5
3-methylcholanthrene	56-49-5
methyl methanesulfonate	66-27-3
1,4-naphthoquinone	130-15-4
1-naphthylamine	134-32-7
2-naphthylamine	91-59-8
N-nitrosomethylethylamine	10595-95-6
N-nitrosomorpholine	59-89-2
N-nitrosopiperidine	100-75-4
N-nitrosopyrrolidine	930-55-2
5-nitro-o-toluidine	99-55-8
pentachlorobenzene	608-93-5
pentachloroethane	76-01-7
pentachloronitrobenzene	82-68-8
phenacetin	62-44-2
2-picoline	109-06-8
1,2,4,5-tetrachlorobenzene	95-94-3
sulfotepp	3689-24-5
o,o,o-triethylphosphorothioate	126-68-1
1,3,5-trinitrobenzene	99-35-4
4-phenylenediamine	106-50-3
2-toluidine	95-53-4
thionazin	297-97-2
bis(2-ethylhexyl)adipate	103-23-1
1-methylnaphthalene	90-12-0
2,3,5,6-tetrachlorophenol	935-95-5
methyl parathion	298-00-0
1,4-dioxane	123-91-1
ethyl methacrylate	97-63-2
diphenyl ether	101-84-8
hydroquinone	123-31-9
quinone	106-51-4
SURROGATES	
1,2-dichlorobenzene-d_4	2199-69-1
2,4,6-tribromophenol	118-79-6
2-chlorophenol-d_4	93951-73-6
2-fluorobiphenyl	321-60-8
2-fluorophenol	367-12-4

TABLE 1

APPLICABLE COMPOUNDS for SEMIVOLATILE ANALYSIS by SW8270 D

COMPOUND	CAS Number
SURROGATES	
phenol-d_6 (d_5)	13127-88-3
terphenyl-d_{14}	1718-51-0
nitrobenzene-d_5	4165-60-0
hydroquinone-d_6 (surr); optional	
INTERNAL STANDARDS	
acenapthene-d_{10}	15067-26-2
1,4-dichlorobenzene-d_4	3855-82-1
chrysene-d_{12}	1719-03-5
naphthalene-d_8	1146-65-2
perylene-d_{12}	1520-96-3
phenanthrene-d_{10}	1517-22-2

2. SUMMARY

Prior to using this instrumental analytical method, the samples are extracted and appropriate concentration and cleanups are performed to prepare the extract for analysis. The semivolatile compounds are introduced into the GC/MS by injecting the extract into a gas chromatograph (GC), equipped with a narrow-bore, fused-silica capillary column. The GC oven housing the column is temperature-programmed to facilitate analyte separation. As analytes elute from the column, they are introduced into the mass spectrometer (MS) detector via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major quantitation ion relative to an internal standard, using a calibration curve.

Methods 8270 D can be used to quantify 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 fused-silica capillary GC column, coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons (PAHs), chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, and aromatic nitro compounds and phenols, including nitrophenols.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analyses according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 The ALS Project Manager is responsible for directing a chlorine residual check to be performed just prior to analysis, as applicable.

3.4 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.5 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.6 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples, and take corrective action to eliminate the problem.

4.2 Contamination by carryover can occur whenever high-concentration and low-concentration extracts are sequentially analyzed. To reduce carryover, the sample syringe must be thoroughly rinsed with solvent between sample introductions.

Whenever an unusually concentrated sample is encountered, a solvent blank may be injected to check for carryover contamination, thus ensuring that the autosampler, injector, and also column bleed, are not contributing carryover contamination.

4.3 Phthalate esters are used in the production of plasticizers and are ubiquitous in many commercial products used in laboratories. In particular, bis(2-ethylhexyl) phthalate is the only one of this class of compounds that is typically present in all extracts analyzed using this procedure.

4.4 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration. Also, chromatography is poor.

- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-nitrosodimethylamine, *pyridine* and *1,4-dioxane* are difficult to separate from the solvent front under the chromatographic conditions described in the method.
- N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

5. APPARATUS AND MATERIALS

5.1 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

Hewlett Packard (HP) Model 6890 GC or equivalent (temperature-programmable oven, capable of splitless or split/splitless injection, constant differential flow controllers).

HP Model 5973 or equivalent MS detector (capable of scanning from 35 to 500amu every 1sec or less; using 70 volts, nominal, electron energy in the electron impact ionization mode; capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all of the tune criteria in Table 3 for a 0.5µL, 100ng, injection of tuning standard; and to ensure sufficient precision of mass spectral data, the MS scan rate shall allow acquisition of at least five spectra, while a sample component elutes from the GC).

5.2 DATA ACQUISITION AND PROCESSING SYSTEM

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

The computer must have software that can search any GC/MS data file for ions of a specific mass, and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

The most recent version of the EPA/NIST Mass Spectral Library, or similar spectral search library, should also be available. This library is used to help to identify non-target compounds generally referred to as tentatively identified compounds (TICs). ALS's Windows NT software uses a NIST 98K library.

5.3 COLUMNS*

Capillary Column - 30m x 0.25mm ID (or 0. 32mm ID), 0.5µm film thickness; J&W Scientific DB-5.625 or equivalent.

* equivalent columns/guard columns may also be used, providing that all method QC criteria can be met

5.4 GASES- use only ultra high purity (99.999%)
Helium: carrier gas

5.5 MEASURING DEVICES

- Microsyringes, gas-tight, Precision Hamilton™ or equivalent, 1µL - 1.0mL sizes (used for spiking)
- Luer-lock syringes, Becton & Dickinson or equivalent, disposable, 5mL or 25mL (used for sample introduction)
- Balance, 0.01g sensitivity (used for weighing solid sample aliquots)
- Volumetric flasks, Class A, with ground glass stoppers, various sizes

5.6 CONSUMEABLE SUPPLIES

- Septa, 11mm, Restek #20365 or equivalent
- Fluorocarbon O-ring, 6.5mm, Restek #20372 or equivalent
- ID splitless liner, 2mm, Restek #20796 or equivalent
- Gold seal, Agilent #18740-20885 or equivalent
- Bottles, glass, with Teflon™-lined screw caps or crimp tops

6. REAGENTS AND STANDARDS

6.1 Methylene chloride, Burdick and Jackson #299-4 or equivalent

6.2 Methanol, Burdick and Jackson #230-4 or equivalent

6.3 STANDARDS

6.3.1 All standards are maintained per PAR SOP 300, refer to this SOP for expiration information. Note that any standard or reagent must be Replaced sooner that its expiration, if laboratory control samples indicate a problem, or deterioration is evident.

All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer.

Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

6.3.2 Care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards (typically in methylene chloride) be stored in a freezer (-10°C to -20°C) in amber vials with firmly sealed Teflon™-lined screw-caps. If permitted by the manufacturer, unopened stock standards may be stored at room temperature in flame-sealed ampules. Standards for this procedure are sonicated, and allowed to equilibrate to room temperature before opening.

6.3.3 Two independent sources of commercial target analyte stock standards are required. The stock standards are purchased as certified solutions from suitable suppliers. Typically, concentrations of stock solutions vary from 1,000-5000µg/mL. First source materials are used to create calibration and continuing calibration verification (CCV) standards. Second source materials are used to create the initial calibration verification (ICV) solution. Laboratory control and matrix spike standards, consisting of compounds that will be representative of the compounds being investigated, may be from either source (a third source is often used for spiking).

6.3.4 Non-target analyte internal standard (IS) and surrogate (SS) stock standards are also purchased. The IS is used to quantitate analytes detected in samples. The SS is used to monitor system performance and method effectiveness for each sample matrix.

The internal standards (ISs) used for this method are: 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12} (see also Table 5). These internal standards are purchased in a mix with all compounds at a concentration of 2,000µg/mL and diluted to an intermediate concentration of 1,000µg/mL. Alternative internal standard stock concentrations may be used.
An appropriate aliquot of IS solution is added to an aliquot of calibration standard or extract prior to analysis so that the resulting concentration is 40ng/µL of each internal standard. In the case of sample extracts that require dilution, the extract is first diluted and then IS is added.

If selected ion monitoring (SIM) is being used to reach lower reporting limits, a less concentrated IS solution is recommended. As a general rule, use an IS concentration that will produce a response factor of approximately 1. For most SIM applications, an IS concentration of 4ng/µL will be sufficient.

The surrogate standards currently utilized are: phenol-d_5, 2- fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d_5, 2-fluorobiphenyl, and p-terphenyl-

d_{l4}, 2-chlorophenol-d_4, and 1,2-dichlorobenzene-d_4. A surrogate intermediate standard is prepared in methanol at a concentration of 50µg/mL for base/neutral compounds and 75µg/mL for acid compounds. Other compounds may be used as surrogates if required by the client. During preparation, 1.0mL of this standard is typically spiked into all client and QC samples

6.3.5 Intermediate standards are created by diluting the stock standards. All dilutions should be performed using syringes, and pesticide grade solvent. The intermediate standard may contain the compounds of interest singly or mixed together. After opening/initial use, transfer the remaining stock standard to a suitable vial, such as an amber vial with a Teflon™-lined screw-cap; store with minimal headspace in a freezer (-10 to -20°C). Dilute target analyte stock standards with dichloromethane. Dilute QC standards with methanol. Intermediate standards should be checked frequently for signs of degradation, especially just prior to preparing working calibration standards from them.

6.3.6 For tuning purposes, a methylene chloride solution containing 100ng/µL of decafluorotriphenylphosphine (DFTPP) should be purchased or prepared. Since .5 µL is the standard injection volume, the standard should also contain 100ng/µL each of 4,4'-DDT, pentachlorophenol and benzidine to verify injection port inertness and GC column performance. Though it is not ALS standard practice, it is also possible to check the degradation of these compounds in the ICV or CCV as long as co-elution with other compounds is not present.

6.3.7 Target analyte intermediate standards are further diluted in dichloromethane to create calibration (working) standards. Prepare, at minimum, five concentrations, a minimum of six concentrations is required if higher order fits (e.g., quadratic) are to be used. One of the calibration standards should be created at a level that yields concentrations less than or equal to the reporting limit (RL). The remaining concentrations should correspond to the expected range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. The laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standards.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Samples should be collected according to an approved sampling plan.

7.2 All samples must be kept chilled (4±2°C).

7.3 Aqueous samples must be collected in one-liter amber glass bottles with Teflon™-lined caps. Soil samples must be collected in glass containers with Teflon™-lined caps.

7.4 Samples are not chemically preserved, however, sodium thiosulfate may be used to dechlorinate liquid samples that contain residual chlorine. When applicable, the Project Manager will designate the need for residual chlorine check.

7.5 Aqueous samples must be extracted within 7 days of collection, soil samples must be extracted within 14 days of collection. Extracts must be analyzed within 40 days of extraction.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 Several techniques are available for sample preparation (i.e., extraction and concentration):

MATRIX	SW-846 METHODS
Water	3520 (SOP 617), 3510 (SOP 626)
Soil/Sediment	3540 (SOP 625), 3550 (SOP 647)
Waste	3540 (SOP 625), 3580 (SOP 622)

All surrogates, and matrix spikes (as applicable) must be added to samples prior to performing the extraction step. Internal standards must be added to the resultant extracts prior to performing the GC/MS instrumental analysis.

Extracts may be cleaned up using Gel Permeation Chromatography (GPC) by Method SW3640. See SOP 641 for GPC procedures. On a limited basis, contingent upon the list of compounds to be analyzed, silica gel cleanup may be performed for semivolatile compounds. See SOP 604 for further details. All compound recoveries must be verified prior to using silica gel as a clean up technique.

8.2 Direct injection

In very limited applications, direct injection of the non-aqueous liquid sample into the GC/MS system with a μL syringe may be appropriate. The detection limit is very high (approximately 10,000μg/L), therefore, it is only permitted where concentrations in excess of 10,000μg/L are expected.

8.3 Modes of Data Acquisition

Mass spectra may be collected in one of two ways: scan mode or selected ion monitoring (SIM) mode. Each mode of type of acquisition is discussed below:

8.3.1 SCAN MODE

A selected mass range is scanned repeatedly over the course of analysis. The typical mass range for Method 8270D is 35-500amu. A scan rate of ~1scan/sec or manufacturer's specifications should be consistent throughout the analysis.

8.3.2 SIM MODE

Specified masses are monitored at specified retention times over the course of analysis. The dwell time (length of time each ion response is measured) can also be adjusted from ion to ion. Instrument conditions, however, must be consistent for all standards, samples and quality control samples.

An example of a SIM method for polyaromatic hydrocarbons (PAHs) is listed below. In this example at the retention time of 2.80 minutes, each ion in the associated group of six ions is searched for 50msec. This monitoring will continue until 9.60 minutes when the next group of fourteen ions is searched for 50msec each. These groups include only the primary and major secondary ions of the compounds of interest. Because the mass spectrometer is looking for fewer ions (less than 15 per scan in the example below vs 465 per scan during a standard scan mode), lower reporting limits can be achieved.

Retention Time (min)	Selected Ions	Dwell Time (msec)
2.80 (start scan time)	82, 108, 127, 128, 129, 136	50
9.60	115, 141, 142, 151, 152, 153, 154, 162, 164, 165, 166, 167, 171, 172	50
12.40	94, 122, 176, 178, 179, 188, 200, 201, 202, 203	50
15.00	125, 226, 228, 229, 236, 240, 252, 253, 260, 264	50
17.60	138, 139, 276, 277, 278, 279	50

If selected ion monitoring is being used, concentration ranges that are 10 to 100 times lower than standard scan mode may be achieved. However, a large amount of mass spectral confirmation and tentatively identified compound information is lost. Because much of the mass spectral confirmation data is not present in SIM mode, retention time confirmation becomes more important. It may be necessary to use tighter RT windows.

8.4 Instrument operating conditions

Whether using scan or SIM mode, instrument operating condition may be the same. Typical instrument operation conditions are shown below:

Initial temperature:	60°C, hold for 3 minutes; 2.80 (start scan time)
Temperature program:	65-100°C at 20°C/min, hold for 1 minute; then
	100-335°C at 30°C/min
Final Temperature:	335°C, hold until benzo[g,h,i]perylene has eluted
Injector temperature:	240°C
Transfer line temperature:	280°C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, splitless
Injection volume:	0.5-2µL
Carrier Gas:	Helium at 40cm/sec

Split injection is allowed if the sensitivity of the mass spectrometer is sufficient.

8.5 TUNING

8.5.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 3 for a 50ng injection of DFTPP. System calibration and analyses may not begin until all criteria are met. Acquisition of the mass spectrum of DFTPP must be performed as follows:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak, or any other discrete peak that does not co-elute with DFTPP.
- The GC/MS tuning standard should also be used to assess GC column performance and injection port inertness. All subsequent standards, samples, MS/MSDs and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

8.5.2 DFTPP evaluation will always be done using scan mode so that the full mass range may be evaluated. Dwell times for ions during SIM mode will differ from those during the scan mode, but all other settings should be kept the same.

8.5.3 Benzidine and pentachlorophenol should be present at their normal responses. The tailing factor of benzidine must be less than 2 and of pentachlorophenol must be less than 2. The HP software performs the tailing factor calculation. Tailing factors are typically evaluated during the analysis of DFTPP. However, though not ALS standard practice, tailing may also be evaluated using benzidine and pentachlorophenol from the calibration verification standards.

8.5.4 Degradation of DDT to DDE and DDD may not exceed 20%. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to remove the first 6-12in. of the capillary column. The use of a guard column between the injection port and the analytical column may help prolong analytical column performance.

8.5.5 The internal standards selected should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 2). If interferences are noted, use the next most intense ion as the quantitation ion (i.e., for 1,4-dichlorobenzene-d4, use 152m/z for quantitation).

8.6 INITIAL CALIBRATION

8.6.1 Analyze a 0.5 to 2.0µL injection of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound. All standard and sample injections must use a consistent injection volume. The following Table is an example of the typical calibration levels associated with initial and continuing calibration standards:

Internal Standard (ng/µL on column)	**Final Concentration (ng/µL on column)**
40	120
40	100
40	80
Internal Standard (ng/µL on column)	**Final Concentration (ng/µL on column)**
40	60
40	40
40	20
40	10
40	5
40	1
40	50 ICV level

Each calibration standard should be carefully evaluated before inclusion in the calibration curve.

8.6.2 To confirm that a complete injection took place, the internal standard recovery results must be within the required acceptance range. The analyst must view the standard in the data analysis section of the software. The chromatography of the standard should be examined to ensure that peak shape and separation appear to be acceptable. Extra care should be taken with the low and the high standards as these are useful to evaluate performance problems. Compounds that commonly have poor chromatographic characteristics should be checked in each standard. These compounds are:

pyridine	n-nitrosodimethylamine
aniline	phenol
bis(2-chloroethyl)ether	benzyl alcohol
bis(2-chloroisopropyl)ether	n-nitroso-di-n-propylamine
2,4-dimethylphenol	benzoic acid
2,4,5-trichlorophenol	4-nitroaniline
benzo(b) fluoranthene	benzo(k)fluoranthene

8.6.3 Isomeric compounds and compounds which exhibit similar spectra must be checked in the data analysis section of the software and on the quantitation report to ensure that the correct compound names have been assigned to each peak. The compounds that should be checked are:

Aniline & bis(2-chloroethyl)ether
The dichlorobenzenes
Benzyl alcohol, 2-methylphenol, 3-methylphenol & 4-methylphenol
2,4-dimethylphenol & benzoic acid
Naphthalene & 4-chloroaniline
2,4,6- and 2,4,5-trichlorophenol
2-, 3- and 4-nitroaniline
2,6- and 2,4-dinitrotoluene
Fluoranthene & pyrene
Phenanthrene & anthracene
Benzo(a)anthracene & chrysene
The phthalates
3,3'-dichlorobenzidine, benzo(b) and (k) fluoranthene, benzo(a)pyrene
Indeno(1,2,3-c,d)pyrene & benzo(g,h,i)perylene

8.6.4 Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF=(A_xC_{IS})/(A_{IS}C_x)$$

where:

Ax = 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 (ng/µL)

C_x = Concentration of the compound being measured (ng/µL)

Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte.

It is also recommended that a minimum response factor for the most common target analytes, as noted in Table 4, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet this criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also strive to place more emphasis on meeting the calibration criteria for those compounds that are critical project compounds, rather than meeting the criteria for those less important compounds.

$$\%RSD = \frac{SD}{\overline{RF}} x100$$

where:

RSD = relative standard deviation

$\overline{RF}$ = Mean of all initial RFs for a compound. (Minimum, 5-point curve).

SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N-1}}$$

where:

RFi = RF for each of the calibration levels (minimum, 5-point cal)

N = Number of RF values (i.e., 5)

If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternate curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Section 8.4.

8.6.5 The relative retention times (RRT) of each compound are evaluated as defined in Section 8.8.

8.7 Linearity

If the %RSD of any compound is 20% 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 the %RSD of any compound is greater than 20%, a calibration curve of area ratio (A/A_{is}) versus concentration ratio (C/C_{is}), using first or second order regression fit of the five or more calibration points may be constructed. The type of curve fit applied should be chosen to best represent the data. 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. The coefficient of determination (COD, r^2 value) of the linear or higher order regression used to define the calibration curve, is an expression of "goodness of fit", and must be $\geq$0.99. Quadratic regressions may be used with a minimum of 6 calibration points, and must yield a COD (r^2 value) of $\geq$0.99.

Consult SW-846 Method 8000C for specific requirements pertaining to each calibration technique. Quadratic or higher order calibrations are not to be employed solely to extend the calibration range for compounds that show saturated or nearly saturated response at higher concentrations.

8.8 Initial calibration verification (ICV)

An ICV must be analyzed each time a new initial calibration is performed. The ICV consists of a standard at or near the mid-point concentration of the initial calibration. The standard is prepared from a source independent from that used for the initial calibration standards.

The measured concentration of analytes in the ICV must be within 30% of the expected value for each analyte. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding these results could be used for screening purposes and would be considered estimated values. The ICV should be analyzed at a concentration level different from that of the CCV to ensure that the curve is valid over much of the range of the initial calibration.

8.9 Routine Maintennace

8.9.1 Bake out column. Extra blanks may be necessary to achieve an adequate baseline if carryover is observed. Replace the injection port liner, pre-column, cut contaminated section from column, or replace column as necessary to alleviate sample effects limiting performance of the front of the system.

Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Oxidized columns will exhibit baselines that rise rapidly during temperature programming. If a column is oxidized, replacement may be necessary.

8.9.2 INJECTION PORT MAINTENANCE

- Cool injection port to room temperature.
- Disconnect column and plug with old septa.
- Open top of injection port and remove liner for cleaning.
- Remove inlet seal and clean with Q-tip and $MeCl_2$.
- Rinse inlet with $MeCl_2$, scrub with micro grit, rinse again with $MeCl_2$ to remove all traces of micro grit.
- Install cleaned or new injection port seal.
- Install cleaned or new injection port liner.
- Replace septa and insert O-ring as needed.
- Seal injection port and turn injection temperature on.
- When injection temperature is reached tighten seal and turn carrier gas on for about 30 seconds. Clip about 2 or 3 inches of column and re-install.
- Turn oven to the maximum temperature of the run and bake for 5 to 10 minutes.

8.10 Daily GC/MS calibration

8.10.1 Prior to analysis of samples, the GC/MS tuning standard must be analyzed. A 50ng injection of DFTPP must result in a mass spectrum for DFTPP that meets the criteria given in Table 3. Also, benzidine and pentachlorophenol should be present at their normal responses. The tailing factor of benzidine must be less than 2 and the tailing factor of pentachlorophenol must be less than 2. The degradation of 4,4'-DDT must be ≤20%. These criteria must be demonstrated during each 12-hour shift.

8.10.2 CONTINUING CALIBRATION VERIFICATION (CCV)
A calibration standard(s) at mid-concentration containing all semivolatile analytes, including all required surrogates, must be analyzed every 12 hours prior to sample analysis. Compare the instrument response factor from this calibration check with the %D criteria discussed below.

If the percent difference or percent drift for a compound is less

than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\overline{RFC}_I - RFC_c}{\overline{RFC}_I} x100$$

where:

$\overline{RFC}_I$ = The average response of check compound in the initial calibration.

RFC_c = RF for CCC in continuing calibration.

If a least squares regression is used, the CCC must be evaluated using a percent drift calculation. Calculate the percent drift using:

$$\%\text{Drift} = \frac{CC - TC}{TC} \times 100$$

where:

CC = Calculated concentration

TC = Theoretical concentration

Problems similar to those listed under initial calibration could affect the ability to pass the calibration verification standard analysis. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The calibration verification criteria must be met before sample analysis begins. See introductory comments of this SOP for a listing of typical problematic compounds. Examples of non-standard target list compounds that may be problematic include benzidine and 3,3'-dichlorobenzidine. CCV responses for non-standard target list compounds >20% may be narrated or controlled as directed in the applicable LIMS program specification.

The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most

recent initial calibration sequence, 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 that in the midpoint standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

8.11 Extract ANALYSIS

8.11.1 Prior to analysis, the sample extract must be brought to room temperature and spiked with internal standards. The entire extract may be spiked, or an aliquot of the extract may be removed and spiked with an appropriate aliquot of the internal standard mixture. The resulting concentration in the extract to be analyzed must be the same as the concentration of the internal standards in the calibration standards (usually 40µg/mL).

8.11.2 Inject an aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for calibration. The injection volume must be the same volume used for the calibration standards.

8.11.3 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must be performed. A new aliquot of the extract is diluted and IS is added to achieve a concentration of 40µg/mL of each IS. The analyst should take care to ensure that extracts are analyzed at the greatest concentration possible while avoiding damage to the instrument.

8.11.4 Extracts may be diluted for matrix interferences to a point at which the baseline rise is equal to one half the height of the nearest internal standard, or non-target compound is the same height or greater of the nearest internal standard. Further, extracts may be diluted if, in the opinion of the analyst, the color or viscosity of the extract indicates that the matrix will interfere with the instrument's future performance.

8.11.5 Perform all qualitative and quantitative measurements as described in Section 8.12 below. Return the extracts to refrigerated storage.

8.12 Data interpretation

8.12.1 COMPOUND IDENTIFICATION

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method or be obtained from the NIST mass spectral library. Because close co-elution of compounds

commonly masks standard spectra and cannot be corrected with background subtraction, the use of NIST mass spectral library spectra, when available, is preferred. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion. Analyst judgment is critical in interpreting spectral data and target compound assignment by the software.
- The RT of the sample component is within ±0.06 RT units of the most recent standard component. If a retention time shift is suspected due to the sample matrix, the relative retention (RRT) time should be evaluated vs. the relative retention time of the most recent standard.

  RRT = compound RT / internal standard RT
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.
- Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds.

- When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of tentatively identified compound (TIC) determination will be determined by the purpose of the analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

8.12.2 QUANTITATIVE ANALYSIS

When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.

If the %RSD of a compound's average response factor is 20% or less, then the concentration in the extract may be determined using the average

response factor ($\overline{RF}$) from initial calibration data and the following equation:

$$C_{ex}(mg/L) = \frac{(A_x x C_{IS})}{(A_{IS} x \overline{RF})}$$

where:

Cex = The concentration of the compound in the extract
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 (ng/µL)

Alternatively, the regression line fitted to the initial calibration may be used for determination of the extract concentration. See Method 8000C, Section 7.0 for the equations describing internal standard calibration and either linear or non-linear calibrations.

Compute the concentration of the analyte in the sample using the equations below:

8.12.2.1 The concentration of the analyte in the liquid phase of the sample, is calculated using the concentration of the analyte in the extract and the volume of liquid extracted, as follows:

$$\text{Concentration in liquid}(\mu g/L) = \frac{(C_{ex} x V_{ex})}{V_o}$$

where:

Vex = extract volume, in mL
Vo = volume of liquid extracted, in L

8.12.2.2 The concentration of the analyte in the solid phase of the sample, is calculated using the concentration of the analyte in the extract and the weight of the solids, as follows:

$$\text{Concentration in solid}(\mu g/kg) = \frac{(C_{ex} x V_{ex})}{W_s}$$

where:

Vex = extract volume, in mL
Ws = sample weight, in kg (typically reported on a dry weight basis)

8.12.2.3 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formula given above should be used with the following modifications: The areas Ax and Ais should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The concentration obtained should be

reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

9. QUALITY CONTROL

9.1 Definition of Batch

For this method, an analysis batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). For soil and waste samples where detectable amounts of organics are present, replicate samples may be appropriate in place of matrix spike samples.

All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch.

Consult LIMS program specification for additional or alternative requirements. See QC Table for additional details.

9.2 BLANK ANALYSIS

Before processing any samples, the analyst should demonstrate, through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a batch of 20 or fewer field samples is extracted or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. An MB must be analyzed for each 12-hour DFTPP tune.

Target compounds may not be detected above one-half the reporting limit (RL), or as otherwise directed in the applicable LIMS program specification. Common laboratory contaminants, such as bis(2-ethylhexyl) phthalate, are allowed at levels as high as the RL. Occurrence of common laboratory contaminants should be considered a warning and must be reported in the data package case narrative. See QC Table for further information.

9.3 SURROGATES

Surrogate recovery is monitored to assess method performance of the particular matrix. Surrogates are added to all standards, blanks, samples and QC samples prior to analysis. Recoveries should be compared to laboratory-established surrogate control limits or to client specified limits as listed in the LIMS program specification. See QC Table for corrective actions.

NOTE: Because of the number of surrogates used by this method, the laboratory will allow for samples to have one acid and one base/neutral surrogate outside limits if the remaining surrogates suggest the problem is matrix

related and that there were no problems with the laboratory's performance during the extraction and analysis.

9.4 INTERNAL STANDARDS
Internal standards are added to all standards, field and quality control samples analyzed. Retention times and responses are evaluated for internal standards. See QC Table for acceptance limits and corrective actions.

9.5 LABORATORY CONTROL SAMPLES
A matrix-specific laboratory control sample (LCS) is analyzed per batch of 20 field samples. It is ALS's practice to also analyze a laboratory control sample duplicate (LCSD) per batch of 20 field samples, if no MS/MSD samples are extracted with this batch. LCS (LCSD) samples are analyzed to evaluate the efficiency of the method performed. See QC Table for acceptance limits and corrective actions.

9.6 MATRIX SPIKE(S)
A matrix spike (MS) and matrix spike duplicate (MSD) sample are analyzed to evaluate the effect of the matrix. Additional sample volume of client samples is needed to perform these analyses. The frequency of the MS/MSD shall be one pair per batch of 20 field samples, assuming adequate volume has been provided. See QC Table for acceptance limits and corrective actions.

9.7 DETECTION LIMIT (MDL) STUDY
The Detection Limit (DL/LOD) is performed as needed, at a minimum, annually, following the guidance of SOP 329.

10. DEVIATIONS FROM METHOD

10.1 Compounds may utilize primary ions other than those listed in Table 2 of this SOP and Table 1 of the Method, if co-elution problems exist. Because the elution pattern will differ from instrument to instrument, changes may be instrument specific.

11. REFERENCES

11.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Method 625," October 26, 1984.

11.2 US EPA SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, "Method 8270C", Update III, December 1996.

11.3 US EPA SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, "Method 8270D", Revision 4, January 1998.

11.4 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 8000C", Revision 3, March 2003.

12 SAFETY, HAZARDS AND WASTE DISPOSAL

12.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:
Chemical Hygiene Plan (CHP)
Radiation Protection Plan (RPP).
Emergency and Contingency Plan (ECP)
Respiratory Protection Plan (RESPP)

12.2 WASTE DISPOSAL

All wastes are disposed of in accordance with the Waste Management Plan (WMP)

TABLE 2
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Suggested)

COMPOUND	PRIMARY ION	SECONDARY ION
pyridine	79	52
N-nitrosodimethylamine	74	42
aniline	93	66, 65
phenol	94	65, 66
bis(2-chloroethyl)ether	93	63, 95
2-chlorophenol	128	64, 130
1,3-dichlorobenzene	146	148, 111
1,4-dichlorobenzene	146	148, 111
1,2-dichlorobenzene	146	148, 111
benzyl alcohol	108	79, 77
bis(2-chloroisopropyl)ether	45	77, 121
2-methylphenol	107	108, 77, 79
N-nitroso-di-n-propylamine	70	42, 101, 130
3-methylphenol	108	107, 77, 79
4-methylphenol	108	107, 77, 79
hexachloroethane	117	201, 199
nitrobenzene	123	77, 51
isophorone	82	95, 138
2-nitrophenol	139	109, 65
2,4-dimethylphenol	107	122, 121
bis(2-choloroethoxy)methane	93	95, 123
2,4-dichlorophenol	162	164, 98
benzoic acid	105	122, 77
1,2,4-trichlorobenzene	180	182, 145
naphthalene	128	129, 127
4-chloroaniline	127	129, 65, 92
hexachlorobutadiene	225	223, 227
4-chloro-3-methylphenol	107	144, 142
2-methylnaphthalene	142	141, 115
hexachlorocyclopentadiene	237	235, 272
2,4,6-trichlorophenol	196	198, 97, 132
2,4,5-trichlorophenol	196	198, 97, 132
2-chloronaphthalene	162	127, 164
2-nitroaniline	65	92, 138
dimethyl phthalate	163	194, 164
2,6-dinitrotoluene	165	63, 89
acenaphthylene	152	151, 153, 76
3-nitroaniline	138	108, 92
acenaphthene	154	153, 152
2,4-dinitrophenol	184	63, 154
4-nitrophenol	109	139, 65
dibenzofuran	168	139
2,4-dinitrotoluene	165	63.89
2,3,4,6-tetrachlorophenol	232	230, 131, 61
diethyl phthalate	149	177, 150
fluorene	166	165, 167
4-chlorophenyl phenyl ether	204	206, 141
4-nitroaniline	138	65, 108, 92
azobenzene	77	51, 182, 105

TABLE 2
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Suggested)

COMPOUND	PRIMARY ION	SECONDARY ION
4,6-dinitro-2-methylphenol	198	51, 105
N-nitrosodiphenylamine	169	168, 167
4-bromophenyl phenyl ether	248	250, 141
hexachlorobenzene	284	142, 249
pentachlorophenol	266	264, 268
phenanthrene	178	179, 176
anthracene	178	176, 179
carbazole	167	139, 140
di-n-butyl phthalate	149	150, 104
fluoranthene	202	101, 203, 100
benzidine	184	92, 185
pyrene	202	200, 203, 101
butyl benzyl phthalate	149	91, 206
benzo(a)anthracene	228	229, 226
3,3'-dichlorobenzidine	252	254, 126
chrysene	228	226, 229
bis(2-ethylhexyl)phthalate	149	167, 279
di-n-octyl phthalate	149	167, 43
benzo(b)fluoranthene	252	253, 125
benzo(k)fluoranthene	252	253, 125
benzo(a)pyrene	252	253, 125
indeno(1,2,3-CD)pyrene	276	138, 277
dibenzo(a,h)anthracene	278	139, 279
benzo(g,h,i)perylene	276	138, 277
benzaldehyde	77	105, 106, 51
acetophenone	105	77, 120, 51
caprolactam	113	55, 85, 56
1,1'-biphenyl	154	153, 76
atrazine	215	200, 202
N,N-dimethylaniline	120	121, 77, 51
N-ethylaniline	106	121, 77
N-methylaniline	106	107, 77
tetramethylurea	72	44, 116
2-acetylaminofluorene	181	180, 223, 152
4-aminobiphenyl	169	115, 141
aramite	191	185, 319, 334
chlorobenzilate	251	253, 139, 111
disulfoton	88	89, 97, 274
diallate	234	236, 86
2,6-dichlorophenol	162	164, 63, 126
dimethoate	87	93, 125
4-dimethylaminoazobenzene	225	120, 77, 42
famphur	218	93, 125
4-nitroquinoline-n-oxide	160	89, 75, 190
N-nitrosodi-n-butylamine	116	158, 57, 99
N-nitrosodiethylamine	102	44, 42, 56
7,12-dimethylbenz(a)anthracene	256	241, 239
3,3'-dimethylbenzidine	212	213, 211
ά,ά-dimethylphenethylamine	58	91, 134, 65
1,3-dinitrobenzene	168	76, 75, 122

TABLE 2
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Suggested)

COMPOUND	PRIMARY ION	SECONDARY ION
ethyl methanesulfonate	79	109, 97
phorate	260	231, 97
pronamide	173	175, 145
safrole	162	104, 131, 77
isosafrole	104	162, 131, 77
methapyrilene	97	58, 261
3-methylcholanthrene	268	252, 253, 126
methyl methanesulfonate	80	65, 95
1,4-naphthoquinone	158	104, 130, 76
1-naphthylamine	143	115
2-naphthylamine	143	115
N-nitrosomethylethylamine	88	42, 56
N-nitrosomorpholine	56	116, 86
N-nitrosopiperidine	114	42, 55
N-nitrosopyrrolidine	100	41, 42
5-nitro-o-toluidine	152	106, 77, 79
pentachlorobenzene	250	248, 252, 215
pentachloroethane	117	119, 167, 165
pentachloronitrobenzene	237	295, 214
phenacetin	108	109, 179, 137
2-picoline	93	66, 39
1,2,4,5-tetrachlorobenzene	216	214, 218, 108
sulfotepp	322	202, 266, 238
o,o,o-triethylphosphorothioate	198	121, 93, 65
1,3,5-trinitrobenzene	213	120, 167
hexachloropropene	213	211, 117, 141
4-phenylenediamine	108	80, 107
2-toluidine	107	106, 77
thionazin	107	96, 248
bis(2-ethylhexyl)adipate	129	57, 71, 70
1,2-dinitrobenzene	168	50, 63, 76
1,4-dinitrobenzene	168	50, 75, 76
1-methylnaphthalene	142	141, 115
2,3,5,6-tetrachlorophenol	232	230, 131, 166
1-chloronaphthalene	162	164, 127
isodrin	193	195, 263, 66
kepone	272	274, 237
methyl parathion	263	125, 109
ethyl parathion	291	109, 97, 139
dibenz(a,j)acridine	279	280, 277
ADDITIONAL COMPOUNDS		
1,4-dioxane	88	58, 43
ethyl methacrylate	69	41, 86, 114
diphenyl ether	170	51, 77, 141
hydroquinone	110	81, 55
hydroquinone-d_6 (surr)	114	
quinone	108	54, 82
SURROGATES		

TABLE 2
CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS (Suggested)

COMPOUND	PRIMARY ION	SECONDARY ION
1,2-dichlorobenzene-d_4	152	150, 115
2,4,6-tribromophenol	330	332, 141
2-chlorophenol-d_4	132	68, 134
2-fluorobiphenyl	172	171
2-fluorophenol	112	64
phenol-d_6 (d_5)	99	42, 71
terphenyl-d_{14}	244	122, 212
nitrobenzene-d_5	82	128, 54
INTERNAL STANDARDS		
acenapthene-d_{10}	164	162, 160
1,4-dichlorobenzene-d_4	152	
chrysene-d_{12}	240	120, 236
naphthalene-d_8	136	68, 108
perylene-d_{12}	264	260, 265
phenanthrene-d_{10}	188	94, 80

TABLE 3

DFTPP KEY ION ABUNDANCE CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	10-80% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of mass 198
197	<2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-60% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>50% of mass 198
443	15-24% of mass 442

TABLE 4

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds Minimum Response Factor (RF)

Benzaldehyde 0.010
Phenol 0.800
Bis(2-chloroethyl)ether 0.700
2-Chlorophenol 0.800
2-Methylphenol 0.700
2,2'-Oxybis-(1-chloropropane) 0.010
Acetophenone 0.010
4-Methylphenol 0.600
N-Nitroso-di-n-propylamine 0.500
Hexachloroethane 0.300
Nitrobenzene 0.200
Isophorone 0.400
2-Nitrophenol 0.100
2,4-Dimethylphenol 0.200
Bis(2-chloroethoxy)methane 0.300
2,4-Dichlorophenol 0.200
Naphthalene 0.700
4-Chloroaniline 0.010
Hexachlorobutadiene 0.010
Caprolactam 0.010
4-Chloro-3-methylphenol 0.200
2-Methylnaphthalene 0.400
Hexachlorocyclopentadiene 0.050
2,4,6-Trichlorophenol 0.200
2,4,5-Trichlorophenol 0.200
1,1'-Biphenyl 0.010
2-Chloronaphthalene 0.800

Factor (RF)
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2-Nitroaniline 0.010
Dimethyl phthalate 0.010
2,6-Dinitrotoluene 0.200
Acenaphthylene 0.900
3-Nitroaniline 0.010
Acenaphthene 0.900
2,4-Dinitrophenol 0.010
4-Nitrophenol 0.010

TABLE 4
(continued)
Semivolatile Compounds Minimum Response Factor (RF)

Dibenzofuran 0.800
2,4-Dinitrotoluene 0.200
Diethyl phthalate 0.010
1,2,4,5-Tetrachlorobenzene 0.010
4-Chlorophenyl-phenyl ether 0.400
Fluorene 0.900
4-Nitroaniline 0.010
4,6-Dinitro-2-methylphenol 0.010
4-Bromophenyl-phenyl ether 0.100
N-Nitrosodiphenylamine 0.010
Hexachlorobenzene 0.100
Atrazine 0.010
Pentachlorophenol 0.050
Phenanthrene 0.700
Anthracene 0.700
Carbazole 0.010
Di-n-butyl phthalate 0.010
Fluoranthene 0.600
Pyrene 0.600
Butyl benzyl phthalate 0.010
3,3'-Dichlorobenzidine 0.010
Benzo(a)anthracene 0.800

Factor (RF)

Chrysene 0.700
Bis-(2-ethylhexyl)phthalate 0.010
Di-n-octyl phthalate 0.010
Benzo(b)fluoranthene 0.700
Benzo(k)fluoranthene 0.700

Semivolatile Compounds Minimum Response

Benzo(a)pyrene 0.700
Indeno(1,2,3-cd)pyrene 0.500
Dibenz(a,h)anthracene 0.400
Benzo(g,h,i)perylene 0.500

2,3,4,6-Tetrachlorophenol 0.010

TABLE 5

TYPICAL SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-dichlorobenzene-d_8
aniline
benzyl alcohol
bis(2-chloroethyl) ether
bis(2-chloroisopropyl) ether
2-chlorophenol
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichlorobenzene
2-fluorophenol (surr)
hexachloroethane
2-methylphenol
4-methylphenol
N-nitrosodimethylamine
N-nitrosodi-n-propylamine
phenol
phenol-d_6 (surr)

naphthalene-d_8
benzoic acid
bis(2-chloroethoxy)methane
4-chloroaniline
4-chloro-3-methylphenol
2,4-dichlorophenol
2,4-dimethylphenol
hexachlorobutadiene
isophorone
2-methylnapthalene
naphthalene
nitrobenzene
nitrobenzene-d_8 (surr)
2-nitrophenol
1,2,4-trichlorobenzene

acenapthene-d_{10}
acenapthene
acenapthylene
1-chloronapthalene
2-chloronapthalene
4-chlorophenyl phenyl ether
dibenzofuran
diethyl phthalate
2,4-dinitrophenol
2,4-dinitrotoluene
2,6-dinitrotoluene
fluorene
2-fluorobiphenyl (surr)
hexachlorocyclopentadiene
2-nitroaniline
3-nitroaniline
4-nitroaniline
4-nitrophenol
2,4,6-tribromophenol (surr)
2,4,6-trichlorophenol
2,4,5-trichlorophenol

phenanthrene-d_{10}
anthracene
4-bromophenyl phenyl ether
di-n-butyl phthalate
4,6-dinitro-2-methyl-phenol
diphenylamine
fluoranthene
hexachlorobenzene
N-nitrosodiphenylamine
pentachlorophenol
phenanthrene

chrysene-d_{12}
benzidine
benzo(a)anthracene
bis(2-ethylhexyl)phthalate
butyl benzyl phthalate
chrysene
3-3'-dichlorobenzidine
pyrene
terphenyl-d_{14} (surr)
di-n-octyl phthalate

perylene-d_{12}
benzo(b)fluoranthene
benzo(k)fluoranthene
benzo(g,h,i)perylene
benzo(a)pyrene
dibenz(a,h)anthracene
indeno(1,2,3-cd)pyrene

Analytical Method: SW8270C or D; EPA 625	**Parameter:** Semivolatile Organic Compounds by GC/MS		**Summary of Internal Quality Control (QC) Procedures and Corrective Action**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 12 hour period	DFTPP ion ratio criteria (Table 3) PCP Tailing <2 Benzidine Tailing <2 DDT degradation ≤20%	Retune. Do not proceed with analysis until tune meets criteria. Perform injection port and column maintenance. Do not proceed until tune meets criteria
Initial Calibration (ICAL)	Following major instrument maintenance; when the daily calibration do not meet criteria	all anylytes: ≤20% RSD Linear regression, $r^2 \geq 0.990$ Quadratic fit, COD ≥ 0.990	Up to 10% of compounds may be noncompliant. Any noncompliant compounds may be reported as estimated values
Initial Calibration Verification (ICV); independent source from ICAL	Following every ICAL	Measured concentrations of all compounds should be within ±30% of expected concentrations;	Up to 10% of the compounds may exceed 30% of expected concentration but the samples must be reported as estimated values.
Continuing Calibration Verification (CCV); at or near mid-point	Every 12-hour period following tune, if ICAL not performed	All analytes: <20 %D IS retention times <30 seconds drift from mid-point in most recent ICAL. IS areas -50 to +100% of corresponding internal standard areas in the mid-pint of the most recent ICAL. See Section 8.10.2	Re-analyze the daily standard. If failure repeats, evaluate/correct instrument malfunction; perform a new ICAL. Up to 20% of the analytes may exceed the 20% criteria but the compounds must be reported as estimated values. NOTE: Recoveries that are high and outside of the stated acceptance criteria may be acceptable in some programs if the analyte that is high was not detected in the associated samples.
Instrument Blank	Every 12-hour period After each calibration An extraction method blank may be used. The extraction method blank should be analyzed with the associated samples.	< ½ RL for all target compounds, or as otherwise specified in applicable LIMS program specification.	Reanalyze to determine if instrument contamination was the cause. If the instrument blank is still non-compliant, correct the problem before analysis of samples.
Extraction Method Blank	One per extraction batch of ≤20samples of similar matrix.	< ½ RL for all target compounds, or as otherwise specified in applicable LIMS program specification.	Re-analyze to determine if instrument contamination was the cause. If MB is still non-compliant, correct the problem and obtain a successful MB analysis before resuming analysis of samples. Samples associated with the failed MB may need to be reanalyzed. NOTE: If problem is isolated to the method blank (associated samples meet all IS and SS criteria and no target compounds are detected above limits), report and complete a non-conformance report (SOP 928).
Surrogate Spikes (SS)	Every standard, client and	See laboratory or client-specified limits; recoveries should be	If non-compliant, check calculations and

Analytical Method: SW8270C or D; EPA 625	**Parameter:** Semivolatile Organic Compounds by GC/MS		**Summary of Internal Quality Control (QC) Procedures and Corrective Action**
QC Check	Frequency	Acceptance Criteria	Corrective Action
	QC sample	within acceptance limits. Surrogates will be considered diluted out, if the dilution of the extract is ≥ 10X.	spike preparation for documentable errors. Reanalyze sample once (re-analysis requirements may be fulfilled by existing multiple extractions, e.g., MS, MSD, REP). If still out, report results and note in narrative. Note: Because of the number of surrogates used by this method, the laboratory will allow for samples to have one acid and one base/neutral surrogate outside limits if the remaining surrogates suggest the problem is matrix related and that there were no problems with laboratory performance during the extraction and analysis. If out-of-limit areas are explained by the sample matrix (e.g. high hydrocarbon content contributes to SS areas), reanalysis is not required. Narrate. At the client's discretion, the sample may be Submitted for re-extraction.
Internal Standard (IS)	Every standard, client and QC sample	EICP area within -50% to +100% of daily calibration check standard.	Inspect instrument for malfunction; correct identified malfunctions, then reanalyze samples. If no instrument malfunction is identified, reanalyze. If analysis of sample extract is still out, report results and note in narrative. Re-analysis requirements may be fulfilled by existing multiple analyses (e.g., MS, MSD, REP, sample dilutions)
Matrix Spike & Matrix Spike Duplicate (MS/MSD)	One per extraction batch of ≤20samples of similar matrix.	See laboratory or client-specified limits; recoveries should be within advisory limits. RPDs for the spiked compounds should also be within advisory limits	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause. Narrate. If significant differences (>15%) exist between the MS and MSD (or between duplicates) reanalysis of the sample and spikes may be necessary.
Laboratory Control Spike & Laboratory Control Spike Duplicates (LCS/LCSD)	One per extraction batch of ≤20samples of similar matrix; typically the LCSD is analyzed only when matrix spikes are not performed	See laboratory or client-specified limits; recoveries must be within acceptance limits. When the full list of compounds is spiked, the laboratory will accept	If non-compliant, check calculations and spike preparation for documentable errors. If no errors are found, then reanalyze LCS to determine if instrumental conditions or extraction preparation was the cause. Notify the Supervisor and initiate corrective action

Analytical Method: SW8270C or D; EPA 625	**Parameter:** Semivolatile Organic Compounds by GC/MS		**Summary of Internal Quality Control (QC) Procedures and Corrective Action**
QC Check	Frequency	Acceptance Criteria	Corrective Action
		a small number of sporadic marginal exceedances, based on the probability that a certain number of compounds will exceed their control limits. Systematic or gross failures shall not be accepted.	(NCR). Re-analyze associated samples, if appropriate. Note: recoveries that are high and outside of acceptance criteria may be acceptable, when the same target compound is not detected in any sample in the batch. Narrate.
Retention Time Shift (RT)	Every sample, standard, and blank	RT shift <30 seconds compared to daily standard Relative retention time (RRT) of sample must be ± 0.06 RRT units of daily calibration check.	Inspect chromatographic system for malfunction; correct identified malfunctions, then reanalyze sample.
Detection Limit Study	See ALS SOP 329	Value must be < RL.	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria Consult with Quality Assurance Manager; RL may be adjusted if needed.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRPAHY/MASS SPECTROMETRY
REFERENCED METHOD:	EPA 624 AND SW8260C
SOP ID:	525
REV. NUMBER:	17
EFFECTIVE DATE:	MAY 12, 2014

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 525 REVISION 17

TITLE: **DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY -- METHODS SW8260C, or EPA 624**

FORMS: **NONE** **(instrument printout used as run log)**

APPROVED BY:

PRIMARY AUTHOR_____DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- SW-846 methods 5030C, 5035A and 8260C; also EPA 624 -- are used to determine volatile organic compounds in a variety of matrices. This SOP is applicable to nearly all types of samples, regardless of water content, including: groundwater, aqueous sludges, caustic or acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons or catalysts, soils, and sediments. The following compounds are presently being analyzed using this SOP. Other compounds can be analyzed after successful demonstration of capability (DOC) and method detection limits study (MDL). Analytes in the Table below are listed in typical elution order. Analytes that are part of ALS's standard reporting list are depicted in bold.

Parameter	CAS No[b]	Purge & Trap
dichlorodifluoromethane	75-71-8	A
chloromethane	74-87-3	A
vinyl chloride	75-01-4	A
bromomethane	74-83-9	A
chloroethane	75-00-3	A
trichlorofluoromethane	75-69-4	A
acrolein	107-02-8	A
1,1-dichloroethene	75-35-4	A
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	A
acetone	67-64-1	PP
iodomethane	74-88-4	A
carbon disulfide	75-15-0	PP

Parameter	CAS No[b]	Purge & Trap
methylene chloride	75-09-2	A
trans-1,2-dichloroethene	156-60-5	A
methyl tertiary butyl ether	1634-04-4	A
acrylonitrile	107-13-1	A
1,1-dichloroethane	75-34-3	A
vinyl acetate	108-05-4	A
cis-1,2-dichloroethene	156-59-2	A
2-butanone	78-93-3	PP
bromochloromethane	74-97-5	A
chloroform	67-66-3	A
1,1,1-trichloroethane	71-55-6	A
2,2-dichloropropane	594-20-7	A
carbon tetrachloride	56-23-5	A
1,1-dichloropropene	563-58-6	A
1,2-dichloroethane	107-06-2	A
benzene	71-43-2	A
trichloroethene	79-01-6	A
1,2-dichloropropane	78-87-5	A
dibromomethane	74-95-3	A
bromodichloromethane	75-27-4	A
2-chloroethyl vinyl ether	110-75-8	A
cis-1,3-dichloropropene	10061-01-5	A
4-methyl-2-pentanone	108-10-1	PP
toluene	108-88-3	A
trans-1,3-dichloropropene	10061-02-6	A
1,1,2-trichloroethane	79-00-5	A
2-hexanone	591-78-6	PP
tetrachloroethene	127-18-4	A
1,3-dichloropropane	142-28-9	A
dibromochloromethane	124-48-1	A
1,2-dibromoethane	106-93-4	A
1-chlorohexane	544-10-5	A
chlorobenzene	108-90-7	A
1,1,1,2-tetrachloroethane	630-20-6	A
ethylbenzene	100-41-4	A

Parameter	CAS No[b]	Purge & Trap
m- and p-xylene	108-38-3/106-42-3	A
o-xylene	95-47-6	A
styrene	100-42-5	A
bromoform	75-25-2	A
isopropylbenzene	98-82-8	A
1,2,3-trichloropropane	96-18-4	A
1,1,2,2-tetrachloroethane	79-34-5	A
bromobenzene	108-86-1	A
n-propylbenzene	103-65-1	A
2-chlorotoluene	95-49-8	A
1,3,5-trimethylbenzene	108-67-8	A
4-chlorotoluene	106-43-4	A
tert-butylbenzene	98-06-6	A
1,2,4-trimethylbenzene	95-63-6	A
sec-butylbenzene	135-98-8	A
1,3-dichlorobenzene	541-73-1	A
p-isopropyltoluene	99-87-6	A
1,4-dichlorobenzene	106-46-7	A
n-butylbenzene	104-51-8	A
1,2-dichlorobenzene	95-50-1	A
1,2-dibromo-3-chloropropane	96-12-8	PP
1,2,4-trichlorobenzene	120-82-1	A
hexachlorobutadiene	87-68-3	A
naphthalene	91-20-3	A
1,2,3-trichlorobenzene	87-61-6	A
trans-1,4-dichloro-2-butene	110-57-6	PP
acetonitrile	75-05-8	PP
allyl chloride	107-05-1	A
chloroprene	126-99-8	A
1,4-dioxane	123-91-1	PP
ethanol	64-17-5	PP
ethyl methacrylate	97-63-2	A
ethyl-tert-butyl ether	637-92-3	n/a
hexachloroethane	67-72-1	PP
isobutyl alcohol	78-83-1	PP

Parameter	CAS No[b]	Purge & Trap
isopropyl ether	108-20-3	n/a
methacrylonitrile	126-98-7	PP
methyl methacrylate	80-62-6	A
propionitrile	107-12-0	PP
tert-amyl methyl ether	994-05-8	n/a
tert-butanol	75-65-0	n/a

A Adequate response by this technique.
b Chemical Abstract Services Registry Number.
PP Poor purging efficiency resulting in high EQLs.
n/a Not applicable; not designated in method.

This SOP describes purge & trap GC/MS procedures that can be used to identify and quantify most organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. However, for the more soluble compounds, quantification limits are approximately five to ten times higher because of poor purging efficiency. Ketones, alcohols and aldehydes are typical of classes of compounds that may have elevated reporting limits due to their high degree of water solubility.

Note that the body of this SOP specifies the procedures to be used for Methods SW8260 C. Any additional or contradictory requirements for EPA Method 624 are addressed in Section 10.

When requested, samples may be analyzed for Gasoline Range Organics (GRO). The carbon range integrated for GRO extends from C6 to C10, which is identified by analyzing a gasoline component standard. A gasoline composite standard is used for initial calibration and the quantification of sample results. The concentration of GRO is calculated using the external standard technique, and the sum of all peak responses within the 2-methyl pentane to 1,2,4-trimethyl benzene retention time range.

2. SUMMARY

Volatile compounds are introduced into the gas chromatograph (GC) by purge & trap. Purged sample components are trapped in a tube containing suitable sorbents in accord with Methods SW5030C or SW5035A. When purging is complete, the sorbent tube is heated rapidly and back-flushed with helium to desorb trapped sample components. The analytes are desorbed directly onto a narrow-bore capillary column for analysis. The column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph.

As analytes elute from the capillary column, they are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantification) ion relative to an

internal standard with the response factor or calibration equation generated from a multi-point calibration curve using average response factors or regression equations.

3. RESPONSIBILITIES

3.1 It is the responsibility of the Analyst to perform the analyses according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The ALS Project Manager is responsible for directing a chlorine residual check to be performed upon sample receipt as applicable.

3.5 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.6 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks and the above sources are suspected, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter and/or sorbent trap. Many trace impurities in the purge (carrier) gas are removed by

passing the He through a heated catalyst bed that is capable of removing hydrocarbons and oxygen.

4.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing the purge needle or apparatus and sample syringes with three portions of organic-free reagent water between samples. Sample tubes are only reused if washed and baked before the next use. After analysis of a sample containing high concentrations of volatile organic compounds, one or more reagent blanks should be analyzed to check for cross-contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purge needle or apparatus with methanol and then rinse it thoroughly with organic-free reagent water. In extreme situations, the entire sample pathway of the purge & trap may require dismantling and cleaning or replacement. The relatively low purging efficiency of many analytes from a large volume sample (e.g., 10mL, 25mL) often results in significant concentrations remaining in the sample purge tube after analysis. Archon autosamplers (or equivalent) use the same purge vessel repetitively for water analysis, but rinse the purge vessel with He and water between samples. If carryover contamination is suspected, (this is likely when a sample containing high concentration levels of volatile compounds is followed by a sample containing low levels of the same volatile compounds), all samples that may have been affected must be re-analyzed. Sample analysis may continue if a cleanup blank or sample following the high concentration sample is free (below the reporting limit) from compounds present over the calibration range in the high level sample. Analyst experience should be used to determine which compounds tend to carryover and at what levels.

4.2.1 Annotations made to instrument run logs should indicate if a sample contains possible carryover contamination. If the subsequent rerun of the sample confirms the presence and level of the volatile compounds, either analysis may be used. If, however, the rerun shows that the presence of the compounds was carryover contamination, only the rerun should be used. The original analysis should be considered non-usable data for the analytes that may have carried over.

4.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, or random background levels will result. Because methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean because clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

4.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol serves as a check on such contamination. To check for cross-contamination during sample storage, the laboratory periodically analyzes sample storage refrigerator blanks (SOP 512).

5. EQUIPMENT AND SUPPLIES

5.1 PURGE & TRAP AUTOSAMPLER DEVICE

Autosampler - OI 4552/Archon, Varian Archon, or equivalent

Sample concentrator - OI 4560 Liquid Sample Concentrator equipped with OI #10 adsorbent trap, or equivalent.

- Autosampler/concentrator – Teledyne Tekmar Atomx Purge and Trap System with K trap or equivalent.

5.2 GAS CHROMATOGRAPH (GC), DETECTOR AND MASS SPECTRAL LIBRARY

Hewlett Packard (HP) Model 5890A or 6890 GC (or equivalent) capable of splitless or split/splitless injection or direct interface to a purge & trap apparatus. Equipped with variable constant differential flow controllers (so that the column flow rate will remain constant throughout desorption) and a temperature-programmable oven. Also equipped with a HP5971, 5972 or 5973 mass spectrometer detector (or equivalent), capable of scanning from 35 to 270amu every 1sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for p-bromofluoro-benzene (BFB) which meets all of the criteria in Table 1 (shown subsequently) when 50ng or less of the GC/MS tune standard is introduced through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC. The NBS/EPA/NIST mass spectral library (library may vary with instrument) is also used to identify non-target compounds generally known as tentatively identified compounds (TICs).

GC/MS interface to the mass spectrometer: Direct coupling by inserting the column into the mass spectrometer is generally used for 0.18 to 0.32mm-ID columns. Any enrichment device or transfer line can be used if all of the performance specifications described in this SOP (including tuning) can be achieved.

5.3 DATA ACQUISITION AND PROCESSING SYSTEM

A computer system that facilitates continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan number. This type of plot is defined as an extracted ion current profile

(EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits.

5.4 COLUMNS - Equivalent columns/guard columns may also be used

Column 1 - 60m x 0.25mm ID capillary column with RTX-624 stationary phase (Restek), 1.4µm film thickness
Column 2 - 60m x 0.25mm ID capillary column with RTX-VMS (Restek), 1.4µm film thickness

5.5 GASES- **only high purity or higher grade gases may be used!**
Helium: purge & trap and carrier gas

5.6 MEASURING DEVICES

- Microsyringes - 5, 10, 25, 50, 100, 250, 500, and 1,000µL
- Syringes - 5, 10, or 30mL, glass
- Syringe valve, two-way with Luer ends (three each), if applicable to the purging device
- Laboratory balance, 0.01g sensitivity (used for weighing solid samples); operated per SOP 305 requirements.

5.7 CONSUMABLE SUPPLIES

- Compact Vespel/Graphite Ferrule, Restek #20264 or equivalent
- Graphite Ferrules, various sizes
- Glass scintillation vials, 20mL and 40mL, with Teflon™/-lined/low-level siloxane screw-caps, or, glass culture tubes with Teflon™-lined screw-caps
- Vials, 2mL, with Teflon™-lined screw-caps
- Pasteur pipettes, 5 ¾" and 5mL, disposable
- Volumetric pipettes, 10mL, Class A, disposable
- Volumetric flasks, Class A - 5mL, 50mL, and 100mL, with ground-glass stoppers
- Spatula, stainless steel
- pH paper, acidic narrow range and wide range
- PTFE-coated magnetic stir bars, for use in soils purged with the Archon autosamplers (SW5035, SW5035A)
- Mininert™ or CERTAN™ vials or equivalent

6. REAGENTS AND STANDARDS

6.1 Organic-free reagent water (SOP 511)

6.2 Methanol (CH_3OH), purge & trap quality or equivalent, demonstrated to be free of analytes. Store apart from other solvents. J.T. Baker #907702 or equivalent

6.3 Pre-conditioned Ottawa sand (for use as clean matrix for method blank (MB) and laboratory control sample (LCS) analyses associated with solid matrix sample analyses). Pre-condition by drying in an oven set at 105°C or greater overnight; EMD #SX0075-3 or equivalent

6.4 STANDARDS

NOTE: Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards in methanol be stored at –10°C to –20°C in Mininert™ or CERTAN™ vials with Teflon™-lined screw-caps. Stock standards that are not accessed as part of routine operations may be stored in 2mL glass vials with Teflon™-lined caps (i.e., Mininert™ vials are not required for rarely utilized stock standards).

6.4.1 All standards are maintained per SOP 300. Two independent sources of commercial target analyte stock standards, in methanol, are required. The stock standards are purchased as certified solutions from suitable vendors. Typically, concentrations of stock solutions vary from 1,000-10,000µg/mL.

6.4.2 Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampoules, if recommended by the manufacturer. Standards for this procedure must be equilibrated to —<0°C (stored in freezer) before opening and protected from light. After opening/initial use, transfer remaining stock standard to a suitable vial (CERTAN™ vial with a Teflon™-lined screw-cap) with minimal headspace, and store in a freezer (-—<0°C).

6.4.3 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds 20% drift. Standards for gases may need to be replaced after one week unless the acceptability of the standard can be documented.

Standards for the non-gases should also be monitored closely by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases may need to be replaced after one month for working standards and three months for opened stocks, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether or styrene may need to be prepared more frequently

NOTE: For initial calibrations, either the first or second source should be less than one month old for non-gas working standards (and 3 months for stocks) and one week for permanent gases.

6.4.4 First source materials are used to create calibration and continuing calibration verification (CCV) standards. Second source materials are used to create the initial calibration verification (ICV) solution. Laboratory control and matrix spike standards may be from either source.

Non-target analyte internal standard (IS) and surrogate (SS) stock standards are also purchased. The IS is used to quantitate analytes detected in samples. The SS is used to monitor system performance and

method effectiveness with each sample matrix. The internal standards (IS) currently utilized for this method are: Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. The surrogates currently utilized are: Dibromofluoromethane, 1,2-Dichloroethane-d4, Toluene-d8, 4-Bromofluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. Other compounds may be used as surrogates, depending upon the analysis and client requirements. It is recommended that internal standards and surrogates be combined (intermediate solution) and prepared at a concentration of 50ug/mL (5uL injected) for the Atomx autosampler and 250ug/mL (1uL injected) for the Archon style autosampler. Each standard, sample or QC sample must me spiked with internal standards and surrogates prior to analysis.

NOTE: The surrogates may be spiked in the initial calibration standards at the same concentration as they are spiked in the samples themselves. Response factors for the surrogates are then averaged to produce a one-point calibration with the sole purpose of measuring the surrogate recovery using the same concentration for each sample analysis. Alternatively, the surrogates can be calibrated in the same manner as the targets themselves (i.e. varying concentrations). If this latter option is used, an equipment validation study must be performed to determine the actual volume of standard delivered. The concentration of standard may be adjusted accordingly for the actual volume delivered at the 1µL setting. For example: (1.135µL actual delivery)(441µg/mL IS/SS spiking solution)/5mL = 100µg/L.

Prepare intermediate QC spike standards, in methanol, from volatile organic compounds that will be representative of the compounds being investigated. At a minimum, the matrix spike will include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Consult applicable LIMS program specifications for appropriate compound list. See Section 9 of this SOP for further details regarding QC (i.e., LCS/LCSD, MS/MSD) samples.

6.4.5 4-bromofluorobenzene (BFB) tune standard: A standard solution containing 50ng/µL of BFB in methanol is prepared.

6.4.6 An appropriate volume of target analyte stock standard is diluted, with methanol, to a specific volume to create intermediate standards. The intermediate standard may contain the compounds of interest singly or mixed together. Intermediate standards must be stored with minimal headspace and should be checked frequently for signs of degradation, especially just prior to preparing working calibration standards from them. Store standards in an appropriate vial with minimal headspace.

The standards may be retained as prescribed in SOP 300. All dilutions should be performed using syringes, and purge & trap grade MeOH.

6.4.7 Target analyte calibration (working) standards at a minimum of five concentrations should be prepared from the intermediate standards. Prepare these solutions in organic-free reagent water. One of the concentrations should be at a concentration less than or equal to the reporting limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. The laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s). **Aqueous calibration (working) standards must be prepared on the day of loading on the autosampler.**

To prepare a target analyte calibration standard for purge & trap, add an appropriate volume of an intermediate standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a micro syringe and rapidly inject the standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the capped flask three times only. Transfer the working standard to a 40mL VOC vial without headspace for low-level water analysis or 5mL into a 40mL VOC vial for soil analysis. It is also acceptable to add the appropriate amount of intermediate standard directly to a gas tight syringe containing the desired purge volume of organic-free water for a 5mL working standard. Archon autosamplers (or equivalent) add the internal standards and surrogates to the working calibration solution prior to analysis. Perform purge and trap procedures as outlined in Methods SW5030C or SE5035A.

6.4.8 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials and of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 Samples should be collected according to an approved sampling plan

7.2 Samples from chlorinated water sources should be dechlorinated with sodium thiosulfate ($Na_2S_2O_3$) in the field at the time of collection. These samples should then be acidified with hydrochloric acid (HCl) following dechlorination. Based upon project knowledge provided by the client, where applicable, ALS's Project Manager may instruct the volatiles analysts to test for chlorine residual just prior to preparation for analysis. A chlorine residual test kit, obtainable from the Sample

Receiving Department, is used to check for chlorine residual. Notify the Project Manager immediately if residual chlorine is present.

7.3 Volatile organic analysis of water and soil samples extracted by Methods SW5030C or SW5035A must be performed within 14 days of collection unless otherwise specified by the client. Water samples are usually preserved by adding approximately four (4) drops of concentrated hydrochloric acid (HCl) to each 40mL VOA vial. The purpose of the hydrochloric acid is to prevent microbial degradation of target compounds. If the water sample is unpreserved, the holding time may be shortened to seven (7) days from the date of collection. Volatile organic analysis of soil samples received in EnCore™ (or equivalent) samplers to be extracted by Method SW5035A shall be frozen upon receipt and analyzed within 14 days of collection. Other types of collection and preservation techniques may be required by Method SW5035A and should be evaluated according to the specific needs of the client. Other means of preservation for samples to be prepared for analysis by Method SW5035A include freezing soil in a 40mL vial after addition of water and a stir bar, as well as addition of sodium bisulfate solution ($NaHSO_4$) and a stir bar. Method SW5035A also allows preservation with methanol for solid samples with expected higher concentrations of target analytes. Preservation of samples for subsequent analysis via Methods SW5035A/8260C may be required within 48hrs after time of collection. Consult applicable LIMS program specification.

7.4 Following sample analysis, measure the pH of each sample. Record the result next to the sample's identity on the previously prepared daily sequence log. If the pH of a preserved sample is >2, immediately notify the appropriate Project Manager and discuss the pH excursion in the data package case narrative. Aqueous samples that are intentionally not preserved at the time of collection do not require Project Management notification.

7.5 Samples to be prepared by Method SW5030C must be collected in glass containers with minimal headspace and stored at 4±2°C. Samples to be prepared by Method SW5035A should be collected in EnCore™ (or equivalent) sampling devices and stored at <-7°C, but no less than -20°C. Other types of collection and preservation techniques may be required by Method SW5035A and should be evaluated according to the specific needs of the client. Consult applicable LIMS program specification.

7.6 To prevent loss of volatile organic compounds, samples must not be opened until the time of analysis.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

Three alternate methods are provided for sample introduction. All internal standards, surrogates, and matrix spikes (when applicable) must be added to samples before purging commences:

- Purge & trap per Method SW5030C (aqueous samples)
- Purge & trap per Method SW5030C (for dilution of solid or waste liquid samples via methanol extraction described in SW5035A)
- Purge & trap per Method SW5035A for solid samples collected in a manner consistent with the method or modification thereof (for samples submitted as samples that must be transferred by laboratory personnel to a purge vessel from containers submitted by the client)

8.1 TYPICAL PURGE & TRAP DEVICE SETTINGS

Instrument conditions may be varied as needed, however, the instrument conditions employed during initial calibration (ICAL) must be used for all subsequent sample analyses that are quantitated using the initial calibration. If operating conditions are altered, a new calibration must be prepared.

Purge & trap settings forArchon/ OI 4560A purge & trap device:
Purge time = 7-11 minutes
Desorb temperature = 190°C
Desorb time = at least 0.5 minute
Trap bake = at least 4 minutes at 210°C. (or according to manufacturer's recommendation for all parameters above)

Purge & trap settings for Teledyne Tekmar Atomx purge & trap device:
Purge time = 7-11 minutes
Desorb temperature = 250°C
Desorb time = at least 0.5 minute
Trap bake = at least 4 minutes at 260°C (or according to manufacturer's recommendation for all parameters above)

8.2 TYPICAL GAS CHROMATOGRAPH SETTINGS

Initial temperature = 50°C
Initial time = 0.1 minute
Temperature ramp A = 10°C/minute
Temperature ramp B = 25°C/minute
Final temperature A = 105°C
Final temperature B = 220°C
Final hold time A = 0 minutes
Final hold time B = until all compounds elute
P&T transfer line temperature = 120°C
GC/MS transfer line temperature = 280°C
Injection temperature = 150°C
Electron energy = 70eV (nominal)
Mass range = 35-270amu
Scan time = 0.6-1 second per scan

8.3 AUTOSAMPLER CLEANING

After use, each purge tube is removed from the autosampler, washed and regenerated per glassware cleaning SOP 334. Additionally, each purge needle is flushed with organic-free DI water (note that the purge tube is rinsed in place, as part of the system program, if using the OI Archon or Atomx autosampler).

8.4 CHROMATOGRAPHIC MAINTENANCE

8.4.1 Bake out the trap and column. Extra blanks may be necessary to achieve an adequate baseline if carryover is observed. Replace trap if performance problems are demonstrated and cannot be alleviated by routine maintenance.

8.4.2 If other chromatographic problems are observed (peak tailing, loss of analytes, poor response, etc.) injection port maintenance (replacement of inlet seal, liner, ferrules, clipping column), MSD source cleaning, etc. may be necessary.

8.4.3 Columns will be damaged permanently and irreversibly by contact with oxygen at elevated temperatures. Oxygen may enter the column during a septum change, when oxygen traps are exhausted, through neoprene diaphragms of regulators, and through leaks in the gas manifold. Oxidized columns will exhibit baselines that rise rapidly during temperature programming. If a column is oxidized, replacement may be necessary.

8.5 INITIAL CALIBRATION (ICAL)

Instrument conditions may be varied as needed; however, the instrument conditions employed dUring initial calibration must be used for all subsequent sample analyses that are quantitated using that initial calibration. If operating conditions are altered, a new calibration must be prepared.

8.5.1 Each GC/MS system must be hardware-tuned to meet Method criteria (see Table 1 below) for a 5-to-50ng injection or purging of 4-bromofluorobenzene (BFB). A BFB tune is performed prior to analysis to demonstrate the ability of the system to separate ions and assign proper ratios to fragments. Analyses must not begin until these criteria are met. Typically, 1µL of a 50ng/µL BFB tune solution is analyzed by direct injection.

TABLE 1

BFB MASS INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)

MASS	INTENSITY REQUIRED (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174

174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

8.5.2 Set up the purge & trap system as outlined in Method SW5030C, or Method SW5035A if closed system purge & trap analysis is to be utilized. A set of at least five calibration standards containing all of the target analytes and surrogates is needed. The calibration must contain a standard at or below the reporting limit for each compound, the other calibration standards should contain analytes at concentrations that define the range of the method, but do not exceed the linear range of the instrument. Due to the varying reporting limit requirements of the laboratory's clientele and the varying instrument response of the target compounds, eight levels are typically analyzed. Below is a list of typical calibration levels used during ICAL. Project requirements and instrument performance may require modifications to the levels listed (consult applicable LIMS program specifications).

Internal Standard (mg/L of 5 mL purges	**Final Concentration (µg/L of 5mL and 5g purges)**	**Internal Standard (µg/L of 10mL purges)**	**Final Concentration (µg/L of 10mL purges)**
50	160	25	60
50	120	25	40
50	80	25	20
50	60	25	10
50	40	25	4
50	20	25	2
50	10	25	1
50	4	25	0.5
50	2	25	0.25
50	40 CCV level	25	10 CCV level
50	80ICV level	25	20 ICV level

8.5.3 Calibration must be accomplished using the sample introduction technique that will be used for sample analysis. The purging efficiency for 5mL of water is greater than that for 10mL or 25mL. Therefore, develop the standard curve using the volume of sample to be analyzed. Prepare working calibration standards as described in Section 6.

8.5.4 Tabulate the area response of the characteristic ions (see Table 2 at end of SOP) against concentration for each compound and each internal

standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF 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.

C_x = Concentration of the compound being measured.

The average RF must be calculated and recorded for each compound using at least five RF values calculated for each compound from the initial calibration curve.

8.5.5 Using the RFs from the initial calibration, calculate and record the percent relative standard deviation (%RSD) for all compounds. The percent RSD is calculated as follows:

$$\%RSD = \frac{SD}{\overline{RF_x}} x100\%$$

where:

RSD = Relative standard deviation

RF_x = Mean of the initial RFs for a compound

SD = Standard deviation of the initial RFs for a compound

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

where:

RF_i = RF for each of the calibration levels

n = number of RF values (i.e., 7)

8.5.6 LINEARITY

If the %RSD of any compound is <20%, then the compound's response is assumed to be constant over the calibration range, and the average relative response factor may be used for quantification.

If the %RSD of any compound is >20%, a calibration curve of area ratio (A/A_{is}) versus concentration ratio (C/C_{is}), using first or second order regression fit of the five or more calibration points, may be constructed.

The use of calibration curves is a recommended alternative to average response factor calibration and is a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system. The coefficient of determination (COD, r^2 value) of the linear or higher order regression used to define the calibration curve, is an expression of "goodness of fit", and must be $\geq$0.99.

The mathematics used in least squares regression have a tendency to favor numbers of larger value over numbers of smaller value. The regression curves that are generated will therefore tend to fit points that are at the upper calibration levels better than those points at the lower calibration levels. To compensate for this, a "weighting" factor which reduces this tendency can be used. The analyst may weigh the curve to either the inverse of the concentration (or, more accurately, the concentration *ratio*) or to the inverse of the square of the concentration.

Quadratic regressions may be used with a minimum of 6 calibration points following the guidelines in SW-846 Method 8000C, and must yield a COD (r^2 value) of $\geq$0.99. A quadratic regression should not be used to compensate for detector saturation.

The type of curve fit applied should be chosen to best represent the data.

NOTE: If an initial calibration point is not used for any reason, the analyst must clearly notate why the data point was not used for instrument calibration. "Picking and choosing" among calibration points in order to meet criteria is NOT acceptable. Generally, calibration points are only discarded due to easily demonstratable causes.

8.5.7 Due to the large number of compounds that may be analyzed by this method, some compounds may fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. Client calibration requirements may also be prescribed in the LIMS program specification.

If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99 for r^2 value) for the alternative curve fits, then the chromatographic system is considered too imprecise for analysis (11.3.4.2 – 8260C).

8.5.8 It is recommended that a minimum response factor for the most common target analytes as noted in Table 3, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and

demonstrating the desired sensitivity. ALS demonstrates this sensitivity at the reporting limits in each batch with a reporting limit verification sample (RVS). See section 9.7.

8.6 INITIAL CALIBRATION VERIFICATION (ICV)
A second source ICV standard is analyzed after the ICAL to independently verify the accuracy of the calibration. The concentration of the ICV should be different from that of the CCV and varied over time.

8.6.1 The percent difference for each analyte considered to have an adequate response by preparation technique 5030/5035 (i.e. not a poor purger, high temperature requirement, etc.) for method 8260 (revision 3, August 2006) must be within 30%, allowing for up to two analytes to exceed the 30% criteria. Target analytes which exceed the 30% criteria are considered estimates. Documentation in the associated case narrative, and inclusion of the response factor calibration report (EPA Form 7) shall be considered sufficient client notification.

The second source check can also serve as the laboratory control sample (LCS) for samples analyzed in the same 12 hour shift as the ICAL. The LCS criteria may be different than the ICV criteria described above.

8.7 CONTINUING CALIBRATION VERIFICATION (CCV)
The ICAL curve for each compound of interest must be checked and verified once every 12 hours during analysis with the introduction technique used for samples. This is accomplished by analyzing a calibration standard (CCV) that is at or near the midpoint concentration for the working range of the GC/MS at the beginning of each 12-hour sequence when initial calibration is not performed.

8.7.1 Prior to the analysis of samples, inject or purge 50ng of the 4-bromofluorobenzene standard following Method SW5030C or Method SW5035A. The resultant mass spectra for the BFB must meet all of the criteria given in Table 1 (shown previously) before sample analysis begins. These criteria must be met at the start of each 12-hour shift.

For the CCV analysis, the %D for all target compounds are evaluated against the initial calibration.

If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds may fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples (11.4.5.4 – 8260C). In cases where compounds fail, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to

detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values, or the associated samples re-analyzed.

Data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

- When the acceptance criteria for the continuing calibration verification are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted, or:

- When the acceptance criteria for the continuing calibration verification are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level, if acceptable to client/project. Otherwise the samples affected by the unacceptable verification shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

8.7.2 RETENTION TIME REPRODUCIBILITY

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 that in the midpoint standard level of the most recent initial calibration, 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 that in the midpoint standard level of the most recent initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. Samples should not be analyzed and reported if the criteria described above are not met.

8.8 SAMPLE ANALYSIS

BFB tuning criteria and calibration verification criteria (discussed above) must be met before analyzing samples. All samples and working standard solutions must be allowed to warm to ambient temperature before analysis. Set up the purge & trap system as outlined in Method SW5030C, or Method SW5035A if closed system purge & trap introduction will be used.

8.8.1 PURGE TEMPERATURE

8.8.1.1 For soil analysis, the ICAL, all CCVs, and all field and QC samples shall be heated to 40°C during the purge.

8.8.1.2 For aqueous analysis, a heated purge is not required. The same purge conditions used for soil analysis may be used for aqueous analysis, however, if the ICAL, all CCVs, and all field and QC samples are heated to 40°C during the purge.

It is recommended that purge volumes of 10 to 25mL should not use a heated purge due to the amount of water vapor that may be introduced into the purge & trap system. The ICAL, all CCVs, and all field and QC samples should be left at ambient temperature during the purge.

8.8.2 AQUEOUS ANALYSIS

8.8.2.1 Allow all aqueous samples to come to ambient temperature prior to analysis. All working standards and some sample dilutions are prepared in 50mL volumetric flasks, spiked accordingly, then transferred to a 40mL VOA vial (without headspace). The 40mL sample vials are then placed in the autosampler carrousel. The autosampler is programmed to remove the appropriate sample volume (usually 10mL), add internal standards and surrogates, and proceed with the purge and trap procedure.

8.8.2.2 The process of taking an aliquot destroys the validity of aqueous samples for future analysis; therefore, if there is only one VOA vial, the analyst should prepare a second aliquot for analysis concurrently to protect against possible loss of sample integrity, or transfer the remaining sample to a 20mL VOA vial (without headspace) and refrigerate. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly.

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8.8.2.3 When a sample is analyzed that has saturated ions from a high concentration compound, this analysis must be followed by an organic-free reagent water blank analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences (refer to Section 4 for further details).

8.8.2.4 The following procedure is appropriate for diluting aqueous purgeable samples. Sample dilution is based on analyte concentration, non-target compound concentration, or the presence of surfactants (foaming samples). All steps must be performed without delay until the diluted sample is in a gas-tight syringe. If usable data has not been generated for a less diluted analysis, the dilution should keep the response of the major constituents (previously saturated peaks) in the upper portion of the linear range to generate the lowest reporting limits possible.

Dilutions may be made in volumetric flasks (10 to 100mL) or gas-tight syringes (5mL or 30mL). Select the volumetric flask or syringe that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions

Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

Inject the proper aliquot of sample from the syringe into the flask. Dilute the sample to the mark with organic-free reagent water. Cap the flask and invert three times. The sample is now ready for analysis.

8.8.2.5 The following procedure can be used to composite aqueous samples prior to GC/MS analysis:

The sample must be at 0 to 6°C during this step to minimize volatilization losses. Combine equal portions of the samples to a chilled volumetric flask. Invert the flask 3 times and transfer to an appropriate container for storage or analysis

~~**8.** The following procedure can be used to composite aqueous samples prior to GC/MS analysis:~~

~~The sample must be at 0 to 6°C during this step to minimize volatilization losses.~~
~~Combine equal portions of the samples to a chilled volumetric flask.~~
~~Invert the flask 3 0times and transfer to an appropriate container for storage or analysis.~~

~~The sample must be at 0 to 6°C during this step to minimize volatilization losses. Combine equal portions of the samples to a chilled volumetric flask. Invert the flask 3 0times and transfer to an appropriate container for storage or analysis~~

SOIL SAMPLE ANALYSIS BY METHOD SW5035A

8.8.3.1 Homogenize the sample well, taking care to minimize the loss of volatile constituents.

8.8.3.2 Weigh 5g of soil into an appropriate purge vessel; place the sample on the autosampler. For method blanks and LCSs, 5g of Ottawa sand should be added to the purge vessel.

8.8.3.3 Add 5mL of organic-free water to the sample. In the case of LCS or MS samples, the associated spike is added with this aliquot.

8.8.3.4 The Archon autosampler (or equivalent) adds a total of 5mL of reagent containing internal standards and surrogates to each sample. The sample is now taken through the purge and trap procedure.

8.8.3.5 The following procedure is appropriate for diluting soil purgeable samples. Soil sample dilution is based on analyte concentration or unknown compound concentration. If usable data has not been generated for a less diluted analysis, the dilution should keep the response of the major constituents (previously saturated peaks) in the upper portion of the linear range to generate the lowest reporting limits.

Soil dilutions are made by weighing an aliquot of less than 5g of sample into the purge tube. To ensure a representative sample aliquot, no less than 0.5g of soil should be purged. For reporting purposes, a nominal amount of 5g will be considered the purge amount, and amounts less than this will be treated as dilutions.

8.8.4 MEDIUM LEVEL SOIL SAMPLES (METHANOL-EXTRACTION)
Methanolic extraction /analysis is used for high concentration solid samples requiring dilutions greater than that which can be soundly achieved using smaller sample volume, or for samples that are difficult to homogenize.

8.8.4.1 Homogenize the sample as well as possible, taking care to minimize the loss of volatile constituents.

8.8.4.2 Weigh approximately 5g (record actual weight to 0.01g) of sample into a labeled, tared 20mL VOA vial. Clean the outer lip of the vial with a Kimwipes™ before obtaining the final weight. In some instances, such as low density soils or odd matrices, an aliquot of less than 5g may be necessary.

8.8.4.3 Add 5mL of methanol, cap and shake vigorously for 2 minutes. Allow solid and methanol to separate for at least 10 minutes. Note that alternate soil weights and methanol volumes may be used depending upon the level of sample dilution required. Enough methanol must be added to the vial to completely cover the soil aliquot.

8.8.4.4 Calculate the volume of the methanol extract that when brought to a final volume of 5mL in water, will bring the dilution concentration into the upper portion of the instrument calibration (factor in any dilution that may have been made by the initial extraction of the sample with methanol). To protect the system from trap or column overload, a maximum of 100µL of extract may be used. Proceed with the analysis as discussed for aqueous samples above (Section 8.8.2).

8.8.4.5 A medium level blank should be prepared in the same manner using 5.0g of Ottawa sand and 5mL of methanol. 100µL of this methanol extract injected into 5mL of water is to be analyzed before the sample extract, to ensure no methanol contamination.

8.8.5 SOIL SAMPLE ANALYSIS BY METHOD 5035A

8.8.5.1 Transfer the contents of an EnCore™ (or equivalent) soil sampler to a 40mL VOA vial containing a magnetic stir bar.

8.8.5.2 Use 5g of Ottawa sand in a 40mL VOA vial as the matrix basis for method blanks (MBs)and laboratory control samples (LCSs).

8.8.5.3 Add 5mL of organic-free water to the vial.

8.8.5.4 For matrix spikes, add 2µL (or appropriate amount) of intermediate spiking solution.

8.8.5.5 Samples may be submitted by clients in 40mL vials which already contain water, preservative ($NaHSO_4$) and stir bar or water and stir bar only. Samples submitted in vials are analyzed in the vials without opening the vial.

8.8.5.6 Place vial on the autosampler.

8.8.5.7 The Archon (or equivalent) is used to add internal standards and surrogates solution and 5mL of organic-free water to the purge vessel bringing the final liquid purge volume to 10mL.

8.8.5.8 Place the VOA vial in the Archon autosampler which will automatically inject 1µL of surrogates and internal standards (if appropriate) prior to purging. Note: The 1µL volume is approximated; as instructed by the instrument manufacturer, the internal loop used to deliver the standard is calibrated for each autosampler to determine the absolute volume being delivered. The autosampler will stir and heat the contents of the VOA vial during the purge process.

8.8.5.9 Soil dilutions are made by weighing an aliquot of less than 5g from the Encore™ (or equivalent) into the VOA vial, if acceptable per client or project. To ensure a representative sample aliquot, no less than 0.5g of soil should be purged. For reporting purposes, a nominal amount of 5g will be considered the purge amount, and amounts less than this will be treated as dilutions. If a dilution greater than can be obtained by 0.5g of soil is required, a medium level extraction must be performed by extracting the contents of the EnCore™ as described in Section 8.8.4 above.

8.9 DATA INTERPRETATION

QUALITATIVE ANALYSIS

8.9.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of the method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met:

The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component. (RRT = RT of the analyte/ RT of the internal standard).

The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.

Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of

the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulders or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e., only one total ion current chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound. Analyst experience and judgment is important when evaluating co-eluting compounds.

8.9.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of **tentative identification**. The necessity to perform this type of tentatively identified compound (TIC) determination will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

The relative intensities of the major ions should agree within $\pm$20%. Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.

Molecular ions present in the reference spectrum should be present in the sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

Ions present in the reference spectrum but not in the sample spectrum should 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.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the analyst assign a tentative identification.

8.9.2 QUANTITATIVE ANALYSIS

8.9.2.1 When a compound has been identified, the quantification of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantification will take place using the internal standard technique. The IS used shall be the one nearest the retention time of that of a given analyte.

8.9.2.2 When the detector response is linear and passes through the origin, calculate the concentration of each identified analyte in the sample as follows:

WATER:

$$Concentration(\mu g / L) = \frac{(A_x)(I_s)}{(A_{IS})(\overline{RF})(V_o)}$$

where:

A_x = Area of characteristic ion for compound being measured

I_s = Amount of internal standard injected (ng)

A_{IS} = Area of characteristic ion for the internal standard

$\overline{RF}$ = Mean relative response factor for compound being measured

V_o = Volume of water purged (mL), taking into consideration any dilutions made

SEDIMENT/SOIL SLUDGE (on a dry-weight basis) & WASTE (normally on a wet-weight basis):

$$Concentration(\mu g / kg) = \frac{(A_x)(I_s)V_t)}{(A_{is})(\overline{RF})(V_i)(W_s)(D)}$$

where:

A_x, I_s, A_{is}, $\overline{RF}$ = Same as for water.

V_t = Volume of total extract (µL) (Use 10,000µL or a factor of this when dilutions are made)

Vi = Volume of extract added (µL) for purging

WS = Weight of sample extracted or purged (g)

D = % dry weight of sample/100, or 1 for a wet-weight basis

8.9.2.3 Where requested by the client, an estimate of concentration for non-calibrated components in the sample may be made. The formulae given above should be used with the following modifications: The areas A_x and A_{IS} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. The chromatographic data system calculates the concentration and reports which IS was used in the calculation. Use the nearest IS free of interferences. Upon request, ALS will report the top 10 non-calibrated components (Tentatively Identified Compounds, TICs) with total ion areas > 10% of the total ion area of the nearest internal standard. Identification of TICs with less than 10% relative abundance is difficult at best, and generally should not be attempted. Some clients may request the reporting of more compounds or compounds with lower areas relative to the closest IS. Consult LIMS program specification for further direction.

8.9.2.4 Alternatively, the regression line fitted to the initial calibration may be used for determination of analyte concentration.

9 QUALITY CONTROL

9.1 DEFINITION OF BATCH

For this method, an analysis batch is defined as a group of 20 or fewer field samples that is associated with one unique set of batch QC samples. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike (MS), and duplicate (field sample, LCS or MS). All quality control samples must be carried through all stages of the sample preparation and measurement steps. In addition, batch QC samples should be analyzed on the same instrument as the samples in the batch. Consult LIMS program specification for additional or alternative requirements.

BLANK ANALYSIS

A method (reagent) blank (MB) must be analyzed for each 12-hour BFB tune and per batch of 20 or fewer field samples of similar matrix. Target compounds may not be detected above one-half the reporting limit (RL); or as otherwise stipulated in the applicable LIMS program specification. Common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride) are allowed at levels as high as the RL; or as otherwise stipulated in the applicable LIMS program specification. Occurrence of these common laboratory contaminants should be considered a warning and must be reported in the data package case narrative. See QC Table for further details.

9.2 SURROGATES

Surrogate recovery is monitored to assess method performance of the particular matrix. Surrogates are added to all standards, blanks, samples and QC samples prior to analysis. See QC Table for acceptance limits and corrective actions.

9.3 INTERNAL STANDARDS

Internal standards are added to all standards, field and quality control samples analyzed. Retention times and responses are evaluated for internal standards. See QC Table for acceptance limits and corrective actions.

LABORATORY CONTROL SAMPLES

A matrix-specific laboratory control sample (LCS) is analyzed per batch of 20 field samples. It is ALS's practice to also analyze a laboratory control sample duplicate (LCSD) per batch of 20 field samples. LCS (LCSD) samples are analyzed to evaluate the efficiency of the method performed. See QC Table for acceptance limits and corrective actions.

9.3.1 Internal standards are added to all standards, field and quality control samples analyzed. Retention times and responses are evaluated for internal standards. See QC Table for acceptance limits and corrective actions

MATRIX SPIKE(S)

A matrix spike (MS) and matrix spike duplicate (MSD) sample are analyzed to evaluate the effect of the matrix. Additional sample volume of client samples is needed to perform these analyses. The frequency of the MS/MSD shall be one pair per batch of 20 field samples, assuming adequate volume has been provided. See QC Table for acceptance limits and corrective actions.

DETECTION LIMITS MDL/DL limits determinations are completed annually and as defined by the reference method. A MDL/DL study must also be performed as a component of method validation or whenever the basic chemistry of a procedure changes. See ALS SOP 329 for guidance on detection limits. ALS uses RVS samples run with each batch to assess the method sensitivity on an ongoing basis and to calculate detection limits as needed.

10. DEVIATIONS FROM METHOD

This SOP meets the requirements of Methods SW8260C. Alternate quantitation ions may be used to limit or eliminate common interferences caused by co-elution of standards or matrix contributions.

EPA METHOD 624 REQUIREMENTS

10.1 Suggested surrogates and internal standards are listed in EPA 624, Table 3. ALS uses the same surrogates and internal standards for both Methods SW8260C and EPA 624 as follows: ISs - fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4; SSs - toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4 and dibromofluoromethane. Two of each of the SSs and ISs listed above are included in EPA 624, Table 3.

10.2 Method EPA 624 states that the concentration of the surrogate spike used should be 30µg/L; ALS typically uses a 50µg/L concentration surrogate spike.

10.3 EPA 624 states specific adsorbent trap and purge & trap conditions, and chromatographic columns and conditions, as well as mass spectrometer conditions to

be used in the execution of the method (i.e., specific purge time, use of a packed column, and scanning conditions tailored for packed column use). Some of these materials, apparatus, and conditions have been eclipsed by technology as described in this SOP. Note that Section 8.1.2 of Method EPA 624 provides for the use of technological advances so long as the precision and accuracy requirements put forth by the Method can be achieved.

10.4 Method EPA 624 requires at least three points in the ICAL; ALS quantitates from a 5-to-7-point curve to meet compliance requirements for Methods SW8260C. This approach also meets compliance requirements for EPA 624, as more than three points are used to calibrate.

10.5 Method EPA 624 states that if the %RSD of the average response factor is less than 35%, then an average response factor may be used. Otherwise, construct a linear curve with a correlation coefficient greater than 0.995. ALS follows the calibration criteria discussed previously in the SOP.

10.6 EPA 624 specifies that the BFB tune period is 24 hours. ALS follows the procedure as discussed in SW8260 C, which specifies that BFB criteria must be passed every 12 hours.

10.7 Method 624 states that a continuing calibration verification (CCV) must be performed every working day (i.e., every 24 hours). ALS observes a criterion that a CCV must be performed every 12 hours. Method 624 also requires that the results of the CCV must meet the requirements set forth in Table 5 of the Method, and that any compounds without limits in this Table must have their recovery reported, but corrective actions are not required.

10.8 EPA 624 states that a matrix spike (MS) and laboratory control spike (LCS) must be performed per every 20 samples. The native sample only needs to be spiked once; a matrix spike duplicate (MSD) sample is not required. EPA 624 also states that the matrix spikes and laboratory control (blank) spikes must meet the acceptance criteria listed in Table 5 of the Method (note that not all compounds have acceptance limits in this Table; for these compounds, the recovery must be reported, however, corrective actions based on those results are not required). Furthermore, EPA 624 discusses matching each compound's spike amount with the amount of the compound in the samples chosen for spiking, and also matching the spike amount to the appropriate regulatory level for each compound. Because samples from several sites are usually batched together, it is ALS's practice to use only one spiking level is for each compound.

10.9 Method EPA 624 states that a set of 4 QC Check samples must be analyzed by an analyst before any samples are processed to demonstrate the ability to perform the method. The concentrations of each compound must be 20µg/L, and the results must fall within the acceptance criteria specified in Table 5 of the method. ALS does observe Demonstration of Capability (DOC) requirements, but at the spike levels presented in the SOP and requiring that the results must meet the laboratory's LCS

criteria established for the procedure (based on SW-846 guidance).

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water, Method 524.2, USEPA, Office of Research Development, Environmental Monitoring and Support Laboratory, Cincinnati, OH, 1986.

12.2 40 CFR, Part 136, Appendix A, 7-1-86 Edition, Method 624.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, “Method 8260C”, Revision 3, August 2006.

12.4 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 5030C, “Purge And Trap For Aqueous Samples”, Revision 3, May 2003.

12.5 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 5035A, “Closed System Purge And Trap And Extraction For Volatile Organics in Soil And Waste Samples”, Revision 1, July 2002.

12.6 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition, Method 8000C, “Determinative Chromatographic Separations”, Revision 3, March 2003.

TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
dichlorodifluoromethane	85	87
chloromethane	50	52
vinyl chloride	62	64
bromomethane	96	94
chloroethane	64	66
trichlorofluoromethane	101	151, 153
acrolein	56	55, 58
1,1-dichloroethene	96	53, 61
1,1,2-trichloro-1,2,2-trifluoroethane	101	103, 151, 153
acetone	58	43
iodomethane	142	127, 141
carbon disulfide	76	78
methylene chloride	84	86, 49
trans-1,2-dichloroethene	96	61, 98
methyl tertiary butyl ether	73	57
acrylonitrile	53	52, 51
1,1-dichloroethane	63	65, 83
vinyl acetate	43	86
cis-1,2-dichloroethene	96	61, 98
2-butanone	43	72
bromochloromethane	128	49, 130
chloroform	83	85
1,1,1-trichloroethane	97	99, 61
2,2-dichloropropane	77	97
carbon tetrachloride	117	119
1,1-dichloropropene	75	110, 77
1,2-dichloroethane	62	98
benzene	78	52, 77
trichloroethene	95	97, 130, 132
1,2-dichloropropane	63	112
dibromomethane	93	95, 174
bromodichloromethane	83	85, 127
2-chloroethyl vinyl ether	63	65, 106
cis-1,3-dichloropropene	75	77, 39
4-methyl-2-pentanone	43	58, 85, 100
toluene	91	92
trans-1,3-dichloropropene	75	77, 39

TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
1,1,2-trichloroethane	83	85, 97
2-hexanone	43	58, 57, 100
tetrachloroethene	164	129, 131, 166
1,3-dichloropropane	76	78
dibromochloromethane	129	127
1,2-dibromoethane	107	109, 188
1-chlorohexane	91	55, 93
chlorobenzene	112	77, 114
1,1,1,2-tetrachloroethane	131	133, 119
ethylbenzene	91	106
m- + p-xylene	106	91
o-xylene	106	91
styrene	104	78
bromoform	173	175, 254
isopropylbenzene	105	120
1,2,3-trichloropropane	110	75, 77
1,1,2,2-tetrachloroethane	83	131, 85
bromobenzene	156	77, 158
n-propylbenzene	91	120
2-chlorotoluene	91	126
1,3,5-trimethylbenzene	105	120
4-chlorotoluene	91	126
tert-butylbenzene	119	91, 134
1,2,4-trimethylbenzene	105	120
sec-butylbenzene	105	134
1,3-dichlorobenzene	146	111, 148
p-isopropyltoluene	119	134, 91
1,4-dichlorobenzene	146	111, 148
n-butylbenzene	91	92, 134
1,2-dichlorobenzene	146	111, 148
1,2-dibromo-3-chloropropane	75	155, 157
1,2,4-trichlorobenzene	180	182, 145
hexachlorobutadiene	225	223, 227
naphthalene	128	
1,2,3-trichlorobenzene	180	182, 145
trans-1,4-Dichloro-2-butene	53	88, 75
1,1,1,2-tetrachlorobenzene	131	133, 119
1,4-dioxane	88	58, 43, 57

TABLE 2

CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS

TARGET ANALYTE	PRIMARY CHARACTERISTIC ION(S)	SECONDARY CHARACTERISTIC ION(S)
acetonitrile	41	40, 39
allyl chloride	76	41, 39, 78
chloroprene	53	88, 90, 50
cis-1,4-dichloro-2-butene	75	53, 77, 124
ethanol	45	46, 43
ethyl methacrylate	69	41, 99, 86
ethyl-tert-butyl ether	59	87, 57, 41
hexachloroethane	201	166, 199, 203
isobutyl alcohol	43	41, 42, 74
isopropyl ether	45	43, 87, 59
methacrylonitrile	41	67, 39, 52
methyl methacrylate	69	41, 100, 39
pentachloroethane	167	130, 132, 165
propionitrile	54	52, 55, 40
tert-amyl methyl ether	73	87, 55, 71
tert-butanol	59	41, 57, 43
1,2-dichloroethane-d_4 (SUR)	65	
toluene-d8 (SUR)	98	
4-bromofluorobenzene (SUR)	95	174, 176
dibromofluorobenzene (SUR)	113	
chlorobenzene-d_5 (IS)	82	117
1,4-dichlorobenzene-d_4 (IS)	152	115,150
1,4-difluorobenzene (IS)	114	
fluorobenzene (IS)	96	70

TABLE 3

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichloroflouromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-triflouroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfate	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethane	0.200	0.655
cis-1,2-Dichloroethane	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon Tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.652
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.916
Styrene	0.300	1.916

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

a. The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.

b. Data provided by EPA region III Laboratory.

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 12 hour period	BFB abundance criteria (Table 1) must be met	Re-tune. Do not proceed with analysis until tune meets criteria.
Initial Calibration (ICAL)	Prior to sample analysis.	Ave RF may be used if: analytes are ≤20% RSD r^2 for regression (or quadratic) curve fit must be ≥0.99; a quadratic curve may be used if 6 or more data points are used	When client or method criteria are not met, reanalyze ICAL. Evaluate/correct instrument malfunction if required
Initial Calibration Verification (ICV): different source than that of ICAL standards	Following every ICAL	Measured concentrations of all analytes should be within 30% of expected concentrations. Sporadic failures allowed for up to two analytes IS retention times <30 seconds drift from mid-point in most recent ICAL IS areas –50 to +100% of corresponding internal standard area in the mid-point of the most recent ICAL	Re-analyze ICV. If still out, evaluate/correct instrument malfunction as needed; perform a new ICAL
Continuing Calibration Verification (CCV); at or near mid-point	Every 12-hour period following tune, if ICAL not performed. Required for quantitating all samples analyzed during the 12 hour sequence	Analytes should be within 20% of expected concentrations. • See section 8.7.2 IS retention times <30 seconds drift from mid-point in most recent ICAL IS areas –50 to +100% of corresponding internal standard areas in the mid-point of the most recent ICAL	Re-analyze the daily standard. If failure repeats, evaluate/correct instrument malfunction; perform a new ICAL NOTE: Recoveries that are high and outside of the stated acceptance criteria may be acceptable in some programs if the analyte that is high was not detected in the associated samples.
Method Blank (MB)	Every 12-hour period; after each calibration/check and 1 per batch of 20 samples of like matrix	< ½ RL for all target compounds, except common laboratory contaminants (e.g., acetone, 2-butanone, methylene chloride), which are allowable to the RL; or as otherwise stipulated in the applicable LIMS program specification.	Re-analyze to determine if instrument contamination was the cause. If MB is still non-compliant, correct the problem and obtain a successful MB analysis before resuming analysis of samples. NOTE: Reporting of samples associated with MBs that yield contaminants may be permitted by some program specifications or at the client's discretion. Example: Toluene in MB at RL but not detected in any sample above the MDL. In this case, document occurrence and resolution using a Nonconformance Report (NCR), SOP 928.

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Surrogates (SS)	Every standard, client sample and QC sample	See laboratory or other applicable limits; recoveries should be within these limits	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, sample may be reanalyzed once (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If still non-compliant, report results and narrate. If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon content contributes to SS areas), reanalysis is not required. Narrate NOTE: Per program specifications, surrogate recovery that is high and outside of acceptance criteria, with no associated target compounds detected, may not require reanalysis.
Internal Standard (IS)	Every standard, client sample and QC sample	Average area within -50% to +100% window of corresponding daily calibration verification standard area RT shift <30 seconds compared to daily standard ; relative retention time (RRT) of sample must be ± 0.06 RRT units of standard	Inspect instrument for malfunction, correct. Sample may be reanalyzed (note that reanalysis may be fulfilled by existing multiple analyses - e.g., duplicate, spike duplicate, dilution). If out-of-limit areas are explained by the sample matrix (e.g., high hydrocarbon content contributes to IS areas), reanalysis is not required. Narrate.
Matrix Spike (MS)	1 per batch of 20 samples of like matrix	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits	If non-compliant, check calculations and spike preparation for errors; correct as needed. If no errors are found, and the associated LCS is within control limits, then sample matrix effects are the most likely cause. Narrate.
Matrix Spike Duplicate (MSD) or Duplicate	1 per batch of 20 samples of like matrix	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these advisory limits RPDs for the spiked compounds should also be within advisory limits	If non-compliant, check calculations for errors. If significant differences exist between the duplicate results, consult with Department Manager (reanalysis of the sample and spikes may be necessary, or sample inhomogeniety may be the likely cause).

Analytical Method: SW8260B or C, EPA 624	**Parameter:** Volatile Organic Compounds		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS) or Duplicate	1 per batch of 20 samples of like matrix; typically the LCSD is analyzed when matrix spikes are not performed	See laboratory or other applicable limits; recoveries for the spiked compounds should be within these limits NOTE: When the full list of compounds is spiked, the laboratory will accept a small number of sporadic marginal exceedances, based on the probability that a certain number of compounds will exceed their control limits. Exceedances must be sporadic and marginal, systematic or gross failures shall not be accepted.	If non-compliant, check calculations and spike preparation for documentable errors; correct as needed. If no errors are found, then re-analyze to determine if instrumental conditions was the cause. Notify the Supervisor and initiate corrective action (NCR)if needed. Re-analyze associated samples, if appropriate. Note that recoveries that are high and outside of acceptance criteria may be acceptable, when the same target compound is not detected in any sample in the batch. Narrate.
RVS	Per Batch	Value should be greater than ½ RL	Not used for batch evaluation unless specified by client requirements.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF IGNITABILITY BY THE PENSKY-MARTENS CLOSED-CUP TESTER
REFERENCED METHOD:	METHODS SW1010A AND ASTM93-80
SOP ID:	629
REV. NUMBER:	11
EFFECTIVE DATE:	AUGUST 2, 2010

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ALS
STANDARD OPERATING PROCEDURE 629 REVISION 11

TITLE: DETERMINATION OF IGNITABILITY BY THE PENSKY-MARTENS CLOSED-CUP TESTER -- METHODS SW1010A AND ASTM93-80

FORM: APPENDIX AAPPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the procedure used to determine ignitability (flash point). This SOP is based on EPA SW-846 Method 1010A and ASTM Method 93-80. A Pensky-Martens closed-cup tester is used to determine the flash point of various liquids (e.g., fuel oils, lube oils), including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids may also be tested using this method. A modified test for analyzing soils is also included.

2. SUMMARY OF METHOD

The sample is heated at a slow, constant rate (with liquid samples undergoing continuous stirring). A small flame is directed into the sample cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature, corrected to a barometric pressure of 101.3kPa (760mm Hg), at which application of the test flame ignites the vapor above the sample.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALSLG-FC's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALSLG-FC standard criteria.

It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Interferences that may affect flash point values are: sudden changes in ambient pressure, sample homogeneity, drafts, and operator bias.

4.2 Samples having a pH≤2 or pH≥12 should not be placed in the sample cup as corrosion of the brass may occur.

4.3 Tars and or asphalt-like material may ruin the cup. The analyst should use caution in placing these organic solids in the cup.

5. APPARATUS AND MATERIALS

- Pensky-Martens closed-cup flash tester, as described in Annex A1 of ASTM Method D93-80.
- Verified thermometer with a range of -20 to 150°C.
- Ignition source such as cigarette lighter or matches

6. REAGENTS AND STANDARDS

- p-xylene – 1,4-dimethylbenzene ($C_6H_4(CH_3)_2$), (at least 97% pure)
- Acetone (CH_3COCH_3) and/or dichloromethane (CH_2Cl_2), for cleaning cup between samples

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 All samples should be collected according to an approved sampling plan.

7.2 Samples must be collected in glass containers and stored at 4±2°C. Samples should NOT be collected in plastic bottles, since volatile compounds may diffuse though the walls of the bottle.

7.3 Preservatives shall not be added to samples.

7.4 Method SW1010A does not specify a holding time for this analysis.

8. PROCEDURES

8.1 INSTRUMENT VERIFICATION PROCEDURE

A verification of instrument performance must be done at the beginning of each analytical batch (i.e., immediately before samples are analyzed), after every 10 samples, and immediately after the last field sample is analyzed. See Section 9.3 for acceptance criteria and corrective action if instrument verification fails.

8.1.1 Thoroughly clean and dry all parts of the sample cup and its accessories before starting the verification. Wash the cup with soap and water, rinse thoroughly with water, rinse with acetone three times, and then rinse with dichloromethane three times. Place the cup in the hood to air dry. Be sure that the stirrer is in place before beginning verification.

8.1.2 When the sample cup is clean and dry, the tester must be checked to demonstrate that it is in proper working order.

8.1.3 Add p-xylene to the cup until it reaches the fill line (approximately 75mL). Place the lid on the cup, making sure the locating device is properly engaged. Temporarily turn off the fume hood so that drafts will not affect the verification or analysis. It may be necessary to cool the sample cup before proceeding if the cup begins to retain heat from previous analyses. To cool the cup, gently place the sample cup in the cooling cup on the right side of the apparatus after adding a small amount of ice. Generally, rinsing the cup with methylene chloride will cool the cup enough to start the process.

NOTE: After p-xylene has been added to the cup, do not agitate the cup excessively.

8.1.4 Insert the thermometer into the appropriate slot on top of the sample cup. When the temperature of the p-xylene has dropped below 15°C, remove the sample cup from the cooling cup, wipe off any excess water from the sample cup, and place the sample cup into the heating mantle so that the cup fits securely. Attach the stirring probe to the stirrer on top of the sample cup.

8.1.5 Connect the gas line to the tester. Turn on the gas valve and apply the ignition source to the gas port outlet located on the top front of the sample cup. Adjust the flame to a 3.2-4.8mm diameter (the flame should be under ~13mm in length).

NOTE: Be sure that all flammable materials are removed from the immediate vicinity of the tester before igniting the lighter and that the hood is off.

8.1.6 Turn on the toggle switch labeled "ON." This switch starts the stirring motor.

8.1.7 The black dial located above the toggle switch turns on the heating element. Turn the dial to initiate heating at a rate of approximately 2°C per minute. Usually the "70" mark on the black dial is sufficient.

8.1.8 Apply the test flame at intervals of less than 5°C by operating the mechanism on the cover that controls the shutter and test flame burner. Lower the flame into the vapor space of the cup, for approximately 1 second, and quickly raise it to its high position.

8.1.9 The p-xylene (or any sample) is deemed to have a flash when a large flame appears and instantaneously propagates over the surface of the sample. The temperature at which this occurs is the flash point.

NOTE: Occasionally, when a temperature just below the flashpoint is reached, the application of the test flame will cause a blue halo or an enlarged flame**; this is not a flash point and the appearance of this halo should not be mistaken for a true flash.**

8.1.10 Under normal operating conditions, the corrected flash point for p-xylene should be 27±1°C. If the flash is outside this range then check the instrument and thermometer and determine the flash point of p-xylene again. If corrected flash point is not within ±1°C of 27°C, then analysis of samples can not proceed until the instrument can be verified to be working properly.

8.1.11 Verification of instrument performance must be done after every 10 samples, and after the last field sample is analyzed. See Section 9.3 for acceptance criteria and corrective action if this QC check fails.

8.2 SAMPLE PREPARATION

Reference methods allow for samples that are known to not contain high concentrations of volatiles, or that are very viscous, to be 'warmed' until they are reasonably fluid before being tested.

Note that if the sample *is* suspected to contain a high concentration of volatile components, or is a soil/solid, this treatment is not to be performed.

Per method directives, no sample is to be heated more than is absolutely necessary, and no sample should ever be heated to a temperature that is within 17°C of the expected flash point.

Because ALSLG-FC generally does not have *a priori* knowledge of samples, this warming as described is not performed, because it cannot be conducted without compromising the sample's integrity.

8.3 PROCEDURE FOR LIQUID SAMPLES

8.3.1 Follow the cleaning procedure as outlined in Section 8.1.1.

8.3.2 Fill the sample cup, as outlined in Section 8.1.3, with an aliquot of client sample.

8.3.3 Follow the procedure outlined in Section 8.1.4, with the exception of lowering the temperature of the sample down to a maximum of 4±2°C before placing the sample cup on the apparatus' stove. Therefore, samples should be left in the refrigerator until just prior to analysis.

8.3.4 Follow the procedure outlined in Sections 8.1.5 through 8.1.11 and apply the test flame approximately every 5°C, until a flash point has been observed, or until a temperature of 96.5°C is reached.

NOTE: Occasionally the test flame will ignite the vapor in the cup to such an extent that a flame will shoot out of the cup, or the test flame will grow very large when the shutter is opened, yet the vapors inside the cup do not ignite and propagate across the surface of the liquid. Both of these phenomena are called a vapor flash, and the temperature at which this was observed must be noted on the benchsheet. This occurrence is not a flash point.

8.3.5 Occasionally the test flame will be extinguished as the shutter is opened, but before the test flame can be fully lowered into the cup. The temperature at which this is first observed should be noted on the benchsheet as follows: "Non-flammable vapors began extinguishing the test flame at X°C."

When this occurs, gas will continue to flow into the sample cup, and may ignite the next time the flash point is checked, thus giving a false flash point. Be aware that as soon as the test flame begins to be extinguished, the observation of a true flash point is practically impossible from that point to the completion of the test.

8.3.6 After a flash point has been observed or the temperature has reached 96.5°C, remove the sample cup from the stove and place in the cooling cup so the cup may cool down. Adding ice to the cooling cup will facilitate this step.

8.3.7 If no flash point is observed, report **"The flash point is >96.5 °C."**

8.3.8 If a flash point is observed, it must by confirmed by repeating the determination of the flash point.

The original aliquot shall be discarded, the cup cleaned, and the test repeated with a fresh test aliquot. The 2nd determination should be within ±5°C of the first determined flash point.

When performing the confirmation analysis, apply the test flame approximately every 5°C until a temperature of 15°C below the first observed flash point is reached, and then apply the test flame at intervals of 1-2°C.

Record second observed flash point. If the flash point cannot be confirmed, write in the comments section that the observed flash point could not be confirmed.

8.4 PROCEDURE FOR SUSPENSIONS OF SOLIDS AND HIGHLY VISCOUS LIQUIDS

Bring the material to be tested and the tester to a temperature of 20±5°C. Stirring in a downward direction, raise the temperature throughout the duration of the test at a rate of not less than 1-5°C/min.. With the exception of these requirements for rates of stirring and heating, proceed as prescribed in Section 8.3.

8.5 PROCEDURE FOR SOLIDS AND SOILS

Follow the same procedure as for liquids as outlined in Section 8.3, with the following exceptions:

8.5.1 Remove the stirrer from the sample cup; **Do Not Stir Soil/Solid Samples.** If the stirrer is not removed, either the stirrer will break or the thermometer will break when the toggle switch is turned on.

8.5.2 If non-flammable vapor begins to extinguish the test flame, note in the comments section the temperature at which this was first observed. Water vapor will continue to extinguish the test flame as the temperature is increased, so increase the rate of heating to a rate of 5°C per minute, and apply the test flame at intervals of approximately every 5°C.

8.5.3 Continue testing the sample following the procedure as outlined in section 8.3.5-8.3.7

8.5.4 If a flash point is observed, confirm the flash point by running the confirmation analysis as outlined in Section 8.3.8.

8.6 CALCULATIONS

8.6.1 Call the CSU Atmospheric Sciences weather station or locate their web site (currently http://ccc.atmos.colostate.edu/~autowx/) and record or print the ambient barometric pressure at the Fort Collins Weather Station on the University campus at the time of the test. When the pressure differs from 101.3 kPa (760 mm Hg), correct the flash point as follows:

corrected flash point = C+0.25 (101.3-K) (1)

corrected flash point = C+0.033 (760-P) (2)

where:

C= observed flash point, degrees Celsius

P= ambient barometric pressure, mm Hg

K= ambient barometric pressure, kPa

8.6.2 The barometric pressure used in the correction calculation is the pressure for the laboratory at the time of the test. Many aneroid barometers, like the ones used at weather stations and airports, are pre-corrected to give sea level readings, which would be incorrect for this test. However, the CSU Atmospheric Sciences Fort Collins Weather Station provides an uncorrected barometric pressure, and its readings may be used for this correction.

9. QUALITY CONTROL

9.1 Method blanks, matrix spike samples, matrix spike duplicate samples, and blank spike samples are *not* required for this test.

9.2 One duplicate analysis (on a new aliquot of sample) must be performed after every 10 samples. If one of the samples analyzed in a batch has a flash point that was confirmed, then this sample may be used as the duplicate sample for that batch. If there are no samples in a batch that exhibited a flash point, then any sample in the batch may be selected for duplicate analysis. For this test, a duplicate analysis of the same test sample should have results that agree to within ±5°C of the original. In the event that this criteria is exceeded for a sample that does exhibit a flash point, the operator should examine the results of the p-xylene calibration verifications that preceded and follow the test samples. If these calibration verifications are acceptable, then a non-homogeneous sample matrix is suspected, and this conclusion will be noted in the case narrative.

9.3 Verify the instrument performance at the start of an analytical sequence, after every 10 samples, and again after the last sample, as described in Section 8.1. The acceptance criteria for this verification check standard is that the measured corrected flash point for p-xylene must agree to within ±1°C of the known flashpoint for p-xylene. If the instrument performance cannot be verified, then the test apparatus should be thoroughly cleaned, dried, and the verification performed again. If the

second attempt at verification is successful, then the samples preceding the verification can be reported. If on a second attempt the flashpoint of p-xylene is not within acceptance criteria, then the preceding samples cannot be reported. Maintenance or repairs will be necessary, and successful verification of instrument performance must be completed before samples can be analyzed and reported.

10. DEVIATIONS FROM METHOD

This SOP meets the requirements of Method SW1010A. Method 1010A directs the reader to ASTM Method D93-80 for additional information, and those requirements have been incorporated into this SOP. There are no known deviations from these referenced Methods.

11. HEALTH, SAFETY AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.1.1 .

11.1.2 The analyst must minimize his/her exposure to p-xylene - prolonged overexposure can adversely affect the central nervous system.

11.1.3 The analyst must remove all flammable materials from the immediate vicinity of the testing apparatus, and make other analysts aware of the use of the flame so they do not bring any flammable materials into the vicinity of the apparatus while testing is being performed.

11.1.4 The analyst must exercise and take appropriate safety precautions during the initial application of the test flame, since samples containing low flash material may give an abnormally strong flash when the test flame is applied to the sample.

11.1.5 Perform this test in a fume hood. To prevent disturbance of the flame when samples are tested, the hood should be turned off so as not to be drawing air. Turn the hood back on between analyses.

11.1.6 Any non-original containers being used to hold reagents (e.g., wash bottles or automatic dispenser bottles) shall be labeled at a minimum with compound name, NFPA Health, Flammability, and Reactivity ratings, and date.

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 1010A Revision 1, December 2004.

12.2 American Society for Testing and Materials, Designation D93-94 vol, 05:01, 093, 1995 edition. Refer to D93-79 or D93-80 for more information.

APPENDIX A

IGNITABILITY WORKSHEET (IGNIT) - METHOD SW1010

Workorder # _____ **Batch ID** _____ **Date** _____ **Initials** _____

Start Time _____ **End Time** _____ **p-Xylene Lot ID** _____ **SOP 629 Rev** __ **Reviewed by/date** _____

SAMPLE #	FLASH#1(°C)	FLASH#2 (°C) (Confirmation reported value)	CORR. FLASH (°C) (see formula below)	CALC. BAROMETRIC PRESSURE (mm Hg) (see formula below)	COMMENTS
P-XYLENE **					
DUP (as applicable)					
P-XYLENE **					

Note that 2nd flash confirmation is run (sample volume permitting) when flash is seen on first test. 2nd flash must be within ±5 °C of first flash for confirmation; if not, see Supervisor. **Calculation for Corrected Flash Point:** n °C + 0.033 (760 mm - P), where P equals current barometric pressure (mm). **Calculated Barometric Pressure:** Access website to obtain current barometric pressure (inches). Convert to mm Hg as follows: barometric pressure (inches) X 25.4 = barometric pressure mm Hg. ** **Control Limits for p-xylene ignition temperature:** 27±1°C

ALS Standard Operating Procedure

DOCUMENT TITLE:	ANALYSIS OF TOTAL ORGANIC CARBON
REFERENCED METHOD:	EPA 415.1, SW9060 A, SM5310 C
SOP ID:	670
REV. NUMBER:	14
EFFECTIVE DATE:	AUGUST 5, 2011

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 670 REVISION 14

TITLE: ANALYSIS OF TOTAL ORGANIC CARBON BY METHODS EPA 415.1, SW9060A AND SM5310 C

APPROVED BY:

PRIMARY AUTHOR_____DATE _____

QUALITY ASSURANCE MANAGER _____DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- EPA 415.1, SW9060A and SM5310 C -- describe procedures for the analysis of Total Organic Carbon (TOC) in water. These procedures are applicable to the measurement of organic carbon contained in drinking, surface, ground, and saline waters, as well as domestic and industrial wastes. Exclusions are noted under Interferences (Section 4).

This procedure is applicable only to homogenous samples that can be injected into the instrument reproducibly by the autosampler.

The forms of carbon that can be measured by this procedure include the following:

- Soluble, nonvolatile organic carbon (e.g., natural sugars)
- Soluble, non-purgeable volatile organic carbon (e.g., mercaptans, alkanes, low molecular weight alcohols)
- Insoluble, partially volatile carbon (e.g., low molecular weight oils)
- Insoluble, particulate carbonaceous materials (e.g., cellulose fibers)
- Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter (e.g., oily matter adsorbed on silt particles).

Because of purging, most volatile organic solvents may be lost.

2. SUMMARY

TOC concentration in water is measured by the use of an automated TOC analyzer. The sample is acidified (if not preserved prior to receipt) and sparged with nitrogen (N_2) gas to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide ($CO_{2)}$ by persulfate ($S_2O_8^{-2}$) in the presence of ultraviolet (UV) light. The resultant CO_2 is sparged from the sample and carried in a stream of N_2 gas to a non-dispersive infrared detector (NDIR). TOC concentration in the sample is calculated as a function of CO_2 peak area by use of a linear equation generated from a previously analyzed multipoint initial calibration.

Sample aliquots, reagents and waste are transferred through the system by means of the autosampler apparatus.

Dissolved Organic Carbon (DOC) can also be measured by this procedure. Although ALSLG-FC prefers that samples be filtered prior to receipt at the laboratory, this filtering can be done after receipt.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALSLG-FC's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALSLG-FC standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Any inorganic carbon (e.g., dissolved CO_2, HCO_3^-, and CO_3^{-2}) present in the sample at the oxidation step will contribute to the CO_2 reaching the detector and consequently give a high bias to the measured TOC concentration. Inorganic carbon must either be removed from the sample prior to the oxidation step, or be accounted for in the final calculation. When the Phoenix 8000 instrument is operating in the TOC mode, the sample is routinely acidified and sparged to remove inorganic carbon prior to oxidation of organic carbon. Note that volatile organic compounds may be lost when inorganic carbon is sparged from the sample.

4.2 A study published by the instrument vendor (Tekmar-Dohrmann) indicates that sulfuric acid (H_2SO_4) could form SO_3 gas in the UV reaction cell. Because SO_3 has similar absorption in the infrared region as CO_2, the SO_3 can cause a positive interference in the NDIR detector of the instrument. Therefore, it is recommended that phosphoric acid (H_3PO_4) be used instead of H_2SO_4 where acid preservation is designated for aqueous TOC samples.

Acidification to pH≤2 at time of collection is desirable for unstable samples, however, it should be noted that acid preservation invalidates any inorganic carbon determination on the samples.

4.3 Chloride (Cl^-) ions can react with persulfate in the reaction cell to form Cl_2 (gas). If the Cl^- concentration in a sample is high (≥ 1000mg/L) this reaction can compete with the oxidation of organic C for persulfate. This reaction can lead to excessive peak tailing of the signal from the NDIR detector. At very high Cl^- concentrations (common to brines, seawater, and some chemical wastewaters) the effect can be severe and low TOC recovery can result because some of the organic matter will not be oxidized in the established analysis time. Therefore, hydrochloric acid (HCl) should not be used as a preservative for water samples designated for TOC analysis. As noted previously, the instrument manufacturer recommends the use of phosphoric acid as a preservative for aqueous samples.

4.4 Because solid particles can plug or damage the 8-port valve in the instrument, it may be necessary to filter samples that contain particulates or to allow the solids to settle out prior to analysis.

5. APPARATUS AND MATERIALS

5.1 Phoenix 8000 TOC analyzer (Tekmar-Dohrmann), or equivalent

5.2 pH paper, narrow-range, acidic

5.3 Vials, glass, 40mL VOA-type

5.4 Syringe filters, Life Sciences IC Acrodisc®, 25mm, 0.45um Supor® (PES) membrane, or equivalent, for filtering samples prior to DOC analysis (Section 12)

6. REAGENTS AND STANDARDS

6.1 Nitrogen (N_2), 99.999% purity, used as carrier and purge gas

6.2 Reagent water, (HPLC grade or Milli-Q ASTM Type II)

6.3 Phosphoric acid, H_3PO_4, concentrated, reagent grade

6.4 Acid reagent for IC sparging: Add 100mL conc. H_3PO_4, to 500mL of reagent water.

6.5 Potassium hydrogen phthalate (KHP), used to create the in-house first-source TOC stock solution.

6.6 Copper (Cu) granules

6.7 Tin (Sn) granules

6.8 Sodium persulfate reagent: transfer 100g of sodium persulfate ($Na_2S_2O_8$) to a large beaker. To the beaker add 850mL of reagent water and 36mL of conc. H_3PO_4. Place a magnetic stir bar into the beaker and stir on a magnetic stir plate until all of the solid particles are dissolved. (expiration date = 1 year).

6.9 STANDARDS

6.9.1 All standards are maintained per SOP 300. In the event of a conflict, the specific guidance in this SOP will supersede that of SOP 300.

6.9.2 TOC stock solution, 1000mg/L TOC, first source: Prepared in-house by adding 2.13g of KHP ($C_8H_5KO_4$) to a 1L Class A volumetric flask half-filled with reagent water. Place a magnetic stir bar into the flask and stir on a magnetic stir plate until all of the solid particles are dissolved. Carefully add 1.0mL of phosphoric acid to acidify the solution to pH $\leq$2, let cool to room temperature. Bring to near full volume with reagent water and verify solution pH as $\leq$2. Bring to full volume with reagent water. **Refrigerate.** The expiration date of this solution is 1 year or less as described in SOP 300. Discard the solution if a precipitate forms or degradation is suspected.

6.9.3 Initial calibration standards: Prepared at a minimum of 5 levels to bracket the linear range of the detector. Prepared by diluting aliquots of the 1000mg/L TOC stock solution with reagent water. Calibration standards with concentrations of 10mg/L or greater can be stored for 1 year or as described in SOP 300. Standards with concentrations of less than 10mg/L are made daily upon use.

6.9.4 "Demand" TOC reference standard, second source: This is a stock standard solution obtained from a commercial vendor that is used to prepare the ICV/LCS standard. Alternately, the standard can be prepared in-house from sources independent of the calibration solutions, per the directions contained in the referenced method. The expiration date of this standard is the manufacturer's expiration date or 1 year from preparation ($\geq$10mg/L), whichever is shorter.

6.9.5 ICV/LCS (Initial Calibration Verification and Laboratory Control Sample): An aliquot of the "Demand" TOC reference stock standard is diluted with reagent water according to instructions provided by the vendor. The reference concentration of the prepared standard is provided by the vendor and may vary from lot number to lot number. The concentration of the ICV is typically different from the CCV and between 20-40mg/L.

6.9.6 CCV (Continuing Calibration Verification) standard: An aliquot of the TOC stock solution is diluted with reagent water to a concentration at or below the mid-point of the calibration range. The concentration of the

CCV is typically 30mg/L for a calibration range of 0.5-60mg/L. This standard expires in the shorter of 6 months or the expiration date of the standard it was prepared from.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 All samples should be collected according to an approved sampling plan.

7.2 Sampling and storage of samples in amber glass bottles is preferable. Plastic containers, such as conventional polyethylene and cubitainers, are permissible if it is established that the containers do not contribute contaminating organics to the sample or adsorb organics from the sample.

7.3 Methods EPA 415.1 and SW9060A provide for chemical preservation of samples using either hydrochloric (HCl) or sulfuric (H_2SO_4) acid. Method SM5310 C provides for chemical preservation of samples using either sulfuric or phosphoric acid (H_3PO_4). As discussed in Section 4.2, a technical note released by the instrument manufacturer (Tekmar-Dohrmann) recommends use of phosphoric acid to avoid possible instrumental interferences. Although ALSLG-FC can accept and process samples preserved with any of the three acids, it is ALSLG-FC's preference and practice to provide for phosphoric acid preservation to $pH \leq 2$.

7.4 The referenced methods do not prescribe a maximum holding time allowance. Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the time between collection of samples and analysis should be minimized. ALSLG-FC's policy is to analyze samples within 28 days of collection.

7.5 Samples should be kept cool (4±2°C) and protected from sunlight and atmospheric oxygen.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 INSTRUMENT SET UP

Prior to analyis, check to see each of the following are adequate for the amount of samples to be analyzed:

8.1.1 N_2 carrier gas, 500^+psi from cylinder.

8.1.2 Ample supplies of persulfate reagent, sparging acid, and reagent water.

8.1.3 Halogen scrubber, ample life.

8.1.4 Carrier gas flow rate (200cc/min, ±10%).

8.1.5 Gas/liquid separator water level filled to waste outlet.

8.1.6 Mist trap is empty, drain if necessary.

8.1.7 Thumbscrews of 8-port valve are hand tightened.

8.2 INITIAL CALIBRATION

8.2.1 Prepare calibration standards as described in Section 6.9 above. Typical concentrations comprising the calibration curve are 1.0, 4.0, 10, 20 and 40ppm.

8.2.2 Analyze the calibration standards on the instrument using the instrument software (TOC Talk™).

8.2.3 After analyzing the standards, the instrument software will calculate a linear equation to fit concentration with instrument response. To be acceptable, the coefficient of variation (r^2 or "r-squared" value on the output) must be 0.99 or greater.

8.3 CALIBRATION VERIFICATION

8.3.1 ICV: After an acceptable initial calibration has been established, an initial calibration verification (ICV) check standard must be analyzed. The ICV must be prepared from a parent source that is independent from that used to prepare the calibration standards. The ICV is typically prepared at a concentration near the midpoint of the calibration range, although other concentrations should be analyzed occasionally. See Section 6.9.4 above for preparation guidance, and QC Table following for acceptance criteria and corrective measures to be taken if necessary.

Since there is no sample preparation step involved in this analysis, the ICV check standard can serve a dual role as the laboratory control sample (LCS) for a quality control (QC) batch of 20 or fewer samples.

8.3.2 CCV: A CCV check standard is run at the beginning and conclusion of each analytical sequence and after every 10 samples in the sequence. **If running samples by SW9060A protocol, this CCV should be prepared from a source other than that used to prepare the ICAL (i.e., a second source)**. Preparation of the CCV is described in Section 6.9.5. Refer to QC Table following for acceptance criteria and corrective measures to be taken if necessary.

8.4 SAMPLE ANALYSIS

8.4.1 Samples must be analyzed for TOC in QC batches of 20 or fewer samples. See Section 9 for QC requirements (type and frequency). Confirm that pH is $\leq$2 for each sample prior to analysis and record the pH test result.

8.4.2 Prior to aliquoting, all samples should be homogenized by thorough shaking or agitation of the sample bottle.

8.4.3 For samples analyzed per Method SW9060A protocol, quadruplicate analyses must be performed for all field samples. Report the average

result of the four (4) analyses and the RSD (Relative Standard Deviation). The range of values may be obtained from the raw data.

8.4.4 If the TOC concentration of a sample exceeds the calibration range (i.e., exceeds the concentration of the highest calibration standard), the sample must be diluted and reanalyzed as necessary until the concentration is within range.

9. QUALITY CONTROL (QC)

See QC Table following for acceptance criteria and corrective measures to be taken if necessary.

9.1 METHOD BLANK

One method blank (MB) must be analyzed with every QC batch of 20 or fewer samples to demonstrate that potential contaminants within the analytical system are in control. The MB consists of an aliquot of reagent water.

9.2 LABORATORY CONTROL SAMPLES

One laboratory control sample (LCS) must be analyzed with every QC batch of 20 or fewer samples to demonstrate the effectiveness of the analytical system. The LCS composition is identical to that of the ICV check standard (see Section 6.9.4). Since there is no preparation step in this analysis, the ICV check standard at the beginning of an analytical sequence can serve a dual role as the LCS for a QC batch.

9.3 MATRIX SPIKES

Matrix spike (MS) samples consist of field samples into which known concentrations of target analytes have been introduced. Analysis of matrix spikes provides information on the effect of sample matrix on target analyte detection. A matrix spike duplicate (MSD) is typically run with the MS.

Sample volume permitting, one pair of matrix spike/matrix spike duplicate (MS/MSD) analyses must be performed for every 20 samples. The matrix spiked samples are prepared by spiking aliquots of a selected field sample in the preparation batch with aliquots of the 1000mg/L stock standard.

Analyte recovery for the MS and MSD is calculated as shown below:

$$\%R = \frac{(Conc._{Found} - Conc._{Sample})}{Conc._{Target}} \times 100$$

where:

$Conc_{Found}$ = analyte concentration found in the MS or MSD sample

$Conc_{Sample}$ = analyte concentration found in the field sample

$Conc_{Target}$ = target (anticipated) analyte concentration based on amount spiked

As a measure of precision, the relative percent difference (RPD) of the laboratory duplicate sample pair (or MS/MSD or LCS/LCSD pair) is calculated as shown below:

$$RPD\ (\%) = \frac{(Result_{MS} - Result_{MSD})}{(Result_{MS} + Result_{MSD}) / 2} \times 100$$

9.4 LABORATORY DUPLICATE

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. The LCS, MS, or both may be analyzed in duplicate to serve this purpose. Precision is expressed as Relative Percent Difference (RPD) (see above).

SW9060A protocol requires a "spike duplicate sample for every 10 samples". If analyzing samples by SW9060A protocol, include either an LCSD or (if sufficient sample volume is provided) an MSD *for every 10 samples analyzed*. If there is insufficient sample for the MSD, then either a second LCS/D pair can be analyzed in the latter half of the prep batch, or prep batches may be limited to 10 samples. **Note that this requirement does not apply to samples being analyzed by Method 415.1.**

9.5 METHOD DETECTION LIMIT STUDY

A method detection limit (MDL) study shall be conducted in the manner prescribed by SOP 329. The MDL study shall be performed as needed and at a minimum, annually.

10. DEVIATIONS FROM METHOD

See discussion in Sections 4.2 and 7.4 regarding acid preservation of samples. Methods 415.1 and SW9060A both describe the homogenization of samples by means of a blender. In order to protect the instrument from being clogged by particulate matter, this approach is not utilized at ALSLG-FC (see Section 8.4). This SOP contains no other known deviations from the promulgated methods.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP

11.1 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)
.

12. REFERENCES

12.1 USEPA, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, Method 415.1, "Total Organic Carbon by Combustion or Oxidation", 1983.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 9060A", Revision 1, November 2004.

12.3 Standard Methods for the Examination of Water and Wastewater, 20th Ed., 1999. "Total Organic Carbon, Persulfate-Ultraviolet Method", 5310 C.

12.4 Phoenix 8000 User Manual, Tekmar-Dohrmann, 1998.

12.5 Application Note, "TOC Analysis: The Acid Preservation Debate", Tekmar-Dohrmann, 2001.

12.6 "Method Development Study: Dissolved Organic Carbon (DOC)", Darryl Patrick, 2007. J:\QAOffice\Demonstrations\

Analytical Method: EPA 415.1; SW9060A, SM5310 C	**Parameter:** Total Organic Carbon (TOC) by Oxidation		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria **	Corrective Action
Initial Calibration, minimum 5-point	As needed (i.e., at on-set of analyses or when continuing calibration does not meet criteria)	r^2 must be ≥ 0.99	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Initial Calibration Verification (ICV), second source check standard run near mid-point of calibration curve (Because no sample preparation steps are involved, the ICV can also serve as the LCS for the initial QC batch of samples analyzed)	Once after each initial calibration	For Method 415.1 and SW9060A analyses, the ICV result must be within ±15% of the expected concentration	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV), run at or below midpoint of calibration; CCV concentration must be different from ICV concentration	Run after every 10 samples to begin and end an analytical sequence	For Method 415.1 and SW9060A analyses, the CCV result must agree within ±15% of the expected concentration	Check that calculations and preparation are correct, evaluate/ correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.
Laboratory Control Sample (LCS), second source standard run near mid-point of calibration curve (The ICV can also serve as the LCS for the initial QC batch of samples analyzed)	One LCS in every QC batch of 20 or fewer samples	For Method 415.1 and SW9060A analyses, the LCS result must be within ±15% of the expected concentration	Check calculations, spike preparation, and freshness of the standard used for spiking. Prepare another LCS and analyze. If LCS still fails, samples in QC batch must be reanalyzed.
Laboratory Duplicate (DUP)	For Method 415.1 and SW9060A, the LCSD & MSD both can serve as a laboratory duplicate analysis	For both Method 415.1 and SW9060A, the RPD between the duplicate pair should be ≤20%	For RPDs outside of QC limits, check all calculations for errors. Narrate.
Method Blank (MB)	One MB per every QC batch of 20 or fewer samples	For Method 415.1 and SW9060A analyses, the MB result must not exceed RL (usually 1mg/L TOC)	Prepare another MB and analyze. If MB still fails, samples in QC batch must be reanalyzed.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	Volume permitting, one MS/MSD pair per batch of ≤ 20 field samples	For Method 415.1 and SW9060A analyses, MS/MSD recoveries should meet advisory limits of ±20% (80-120% of the expected values) and RPD should be ≤ 20	Check for documentable errors (e.g., calculations and spike preparation). For Method 415.1 and SW9060A analyses, sample matrix effects are the most likely cause if no errors are found. Document and note in case narrative.

Analytical Method: EPA 415.1; SW9060A, SM5310 C	**Parameter:** Total Organic Carbon (TOC) by Oxidation		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria **	Corrective Action
Method Detection Limit (MDL) Study; run per guidance in SOP329	As needed and, at minimum, annually	Positive result < analyte reporting limit (usually 1.0PPM for both Method 415.1 and SW9060A analyses)	Determine the reason for failure and correct problem with system; then repeat study. If MDL study still not acceptable, discuss with Department and QA Managers, RL may be adjusted, if necessary.

**** Acceptance Limits are as stated within Table, or as otherwise specified in the applicable LIMS program specification.**

Appendix A

Preservation/Acidification and Filtration of Water Samples for TOC/DOC SOP 670 rev. __ ***ALS Laboratory Group***

Workorder ID / Sample ID	DOC							TOC / DOC			Comments
	Date	Initials	Samples Filtered by PAI or Client	Filtered Through 0.45um? (Y/N)	Filter Spec's LOT #	Volume of H_3PO_4 Added (mL)	Conc. H_3PO_4 LOT #	Date	Initials	pH at Time of Analysis	

Filter Blanks = Milli Q Double Deionized Water.

Reviewed by/date _____

Instrument SN 01-011007

Form 647r1.xls

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF HEXANE EXTRACTABLE MATERIAL (HEM) AND SILICA GEL TREATED HEXANE MATERIAL (SGT-HEM) BY EXTRACTION AND GRAVIMETRY FOR AQUEOUS SAMPLES
REFERENCED METHOD:	EPA 1664 A, AND SW9070A
SOP ID:	671
REV. NUMBER:	11
EFFECTIVE DATE:	APRIL 14, 2014

ALS Environmental

STANDARD OPERATING PROCEDURE 671 REVISION 10

TITLE: DETERMINATION OF N-HEXANE EXTRACTABLE MATERIAL (HEM) AND SILICA GEL TREATED HEXANE EXTRACTABLE MATERIAL (SGT-HEM) BY EXTRACTION AND GRAVIMETRY FOR AQUEOUS SAMPLES -- METHODS EPA 1664A AND SW9070A

FORMS: Appendix A

APPROVED BY:

PRIMARY AUTHOR___ _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- EPA 1664A and SW9070A -- are used to determine the content of n-hexane extractable material (HEM) in environmental water samples. Extractable materials that may be analyzed are relatively non-volatile hydrocarbons, vegetables oils, animal fats, waxes, soaps, greases and related materials. This method is not applicable to measurement of materials that volatilize at temperatures below approximately 85°C. The typical reporting limit is 5.0mg/L, which is equivalent to the Minimum Level (ML) required in Method 1664A. Note that SW-846 Method 9070A directs the reader to EPA Method 1664A, Publication No. EPA-821-R-98-002, for the method procedure. The suffix "A" and the method title were inadvertently omitted during the last promulgation as part of SW-846 Update IIIA.

2. SUMMARY

A 1L sample is acidified to pH $\leq$2 and serially extracted three times with n-hexane in a separatory funnel or solid phase extraction apparatus. The extract is dried over sodium sulfate. The solvent is evaporated from the extract and the HEM is desiccated and weighed. If the HEM is to be used for determination of SGT-HEM, the HEM is re-dissolved in n-hexane, then an amount of silica gel proportionate to the amount of HEM is added to remove polar materials. The solution is filtered to remove the silica gel, the solvent evaporated, and the SGT-HEM is desiccated and weighed.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing equipment are possible sources of contamination. Thorough cleaning of glassware is necessary. Materials used in the analysis must be demonstrated to be free of interferences by analyzing laboratory blanks.

4.2 Fine particulates suspended in the sample, as well as the sodium sulfate used in this procedure, could cause a positive interference by passing through the filter paper. If the filter paper is inadequate for removal of the fine particulates, then use of a 0.45µm filter is recommended.

4.3 Some crude oils and heavy fuel oils contain a significant percentage of materials that are not soluble in n-hexane (e.g., asphaltenes). Accordingly, recoveries of these materials may be low.

5. APPARATUS AND MATERIALS

5.1 Boiling flasks, 500mL for separatory funnel; 100 mL for SPE extraction

5.2 PTFE-boiling chips: pre-clean by rinsing with methylene chloride, then dry

5.3 Desiccator

5.4 Indicating Drierite™

5.5 Lint-free wiping cloths (e.g., KimWipes™), used to keep boiling flasks free of external contamination

5.6 Analytical balance, 0.0001g sensitivity, calibration verified per SOP 305

NOTE: A 2mg weight is used in addition to the standard (SOP 305) calibration verification (which includes the required 1000mg weight). This 2mg calibration verification is recorded on the benchsheet. The calibration of the balance must be verified **before** and **after** each analytical batch is weighed.

5.7 Separatory funnels, with PTFE stopcock and stopper, 1500mL

5.8 Graduated cylinder, 1L

5.9 Erlenmeyer flasks, 1500mL, or as appropriate

5.10 Funnels, glass, for holding filter paper

5.11 Filter paper, Whatman Glass Fiber Filter, GF/F, 142mm, #1825-142 or equivalent

5.12 Steam generator and S-Evap unit or Rapid Vap® evaporation unit

5.13 Pasteur pipets, disposable

5.14 Drying oven capable of maintaining 130-150ºC

5.15 Vacuum pump, with inlet hose

5.16 Stirring hot plate

5.17 PTFE-coated magnetic stir bars

5.18 Solid Phase Extraction Apparatus

- 5.18.1 SPE Manifold Setup (available from Environmental Express)
- 5.18.2 C18-47 mm and/or 90 mm SPE disks
- 5.18.3 Sodium Sulfate Drying Cartrides (5.5 g)

6. SOLVENTS

6.1 n-hexane, 85% purity, 99% minimum saturated C_6 isomers, residue less than 1mg/L.

6.2 Acetone, ACS grade, residue less than 1mg/L.

7. REAGENTS AND STANDARDS

7.1 Organic-free reagent water: laboratory deionized (DI) water is suitable.

7.2 Hydrochloric acid (HCL) or sulfuric acid (H_2SO_4), 1:1 Solution: Mix equal volumes of ACS grade concentrated HCl or H_2SO_4 into DI water.

7.3 Anhydrous sodium sulfate (Na_2SO_4), ACS grade, granular: Kiln for a minimum of 4 hours at 450 ^oC; cool to room temperature before use.

7.4 Silica gel, anhydrous, 75-150µm, Davisil Grade 923, Supelco #21447-7a or equivalent. Dry at 130-150ºC for a minimum of 24 hrs. This material is stored in the drying oven until use.

7.5 STANDARDS

7.5.1 Stearic acid, 98% minimum purity.

7.5.2 Hexadecane, 98% minimum purity.

7.5.3 All standards are maintained per ALS SOP 300. Unopened stock standards are valid until the manufacturer's expiration date and may be stored at room temperature in flame-sealed ampules or as otherwise recommended by the manufacturer. After opening ampules, the stock solution may be stored at room temperature in a tightly capped vial and retained for up to six months, however, the unused stock remaining after opening and initial use is typically discarded. Intermediate standards (e.g., OPR Spiking Solution) may also be stored in a tightly sealed container at room temperature and retained for up to six months. Standards may be replaced sooner if laboratory quality control (QC) analyses or other factors indicate deterioration.

Target analyte stock standards are generally purchased as certified solutions, and used to create calibration and spike standards. **Note that for this analysis, only *one* source of standard is required, a check standard from an independent (2nd) source is *not* required for this procedure.**

7.5.4 On-going Precision and Recovery (OPR) Spiking Solution (Hexadecane/Stearic acid): The OPR Spiking Solution is used to create laboratory control samples (LCSs), and is either purchased as a 4.0mg/mL in acetone commercial standard, or prepared as follows: Place 200±2mg of stearic acid and 200±2mg of hexadecane in a 100mL volumetric flask and fill to the mark with acetone. After the hexadecane and stearic acid have dissolved, transfer the solution to a 100-150mL glass container with a Teflon-lined cap; label appropriately. Verify concentration of spiking solution by removing 10.0mL of the solution and placing it in a tared weighing pan. Evaporate to dryness in a fume hood, then weigh. The mass of the residual materials in the pan must be 40±1mg.

7.5.5 All stock and intermediate standards are documented in ALS's Standards and Solutions database. The information recorded in the database facilitates reordering, provides documentation of purity or concentration of purchased materials, and also documents the concentration of each intermediate dilution (as well as the analyst who prepared the dilution), and ensures traceability to the manufacturer. Additionally, Certificates of Analysis are maintained by the applicable laboratory Department.

8. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

8.1 Samples should be collected according to an approved sampling plan.

8.2 A minimum of 1L of representative sample collected in a clean glass bottle is needed for analysis. If selected samples are to be used for matrix spike/matrix spike duplicate analysis, then additional aliquots must be collected accordingly.

8.3 If a sample is known or suspected to contain greater than 1,000mg/L of HEM, then a smaller volume of sample may be utilized for this analysis. **Note that this analysis does not allow subsampling of a water sample from the collection bottle at the laboratory. If smaller sample aliquots are desired, then they must be collected in the field, then shipped to the laboratory for analysis.**

8.4 The pH of the sample is adjusted to $\leq$2 with hydrochloric or sulfuric acid, at the time of collection. If a sample when checked for pH at the laboratory upon receipt yields a pH >2, the ALS Project Manager (PM) is notified, and will contact the client for instructions as to whether or not sample analysis should proceed.

8.5 Samples are stored at 4$\pm$2°C until analysis. No holding time has been established for this determination; ALS observes a 28-day holding time to extraction for this procedure.

9. PROCEDURE

9.1 A sufficient number of boiling flasks must be prepared to accommodate all field and QC samples (e.g., MB, LCS/LCSD, MS/MSD) in the batch. Label, add 3-5 boiling chips, wipe and weigh each flask on the analytical balance to the nearest 0.1mg. Record the weight on the benchsheet.

NOTE: Because the flasks are cleaned (SOP 334) and dried in a kiln, they do not need to be rinsed with hexane and dried prior to use. Store the flasks in a desiccator until needed.

One method blank (MB) and one OPR spike sample (LCS, see Section 8 above), must be prepared with each batch of twenty or fewer field samples. Though not required by the Methods, it is ALS's policy to prepare and analyze a duplicate laboratory control sample (LCSD), with each batch. Use a 1000mL aliquot of DI water for the MB, LCS and LCSD sample (place each DI water aliquot into an appropriately labeled separatory funnel). If the client provided sufficient sample volume, also prepare a matrix spike/matrix spike duplicate (MS/MSD) with each batch. Acidify all QC samples to pH<2. Spike LCS/LCSD and MS/MSD samples with OPR Spiking Solution to yield a target value of 40mg HEM/sample.

9.2 EXTRACTION – SEPARATORY FUNNEL

9.2.1 Using a marking pen, mark the sample's water level on the side of the sample container. Carefully pour the sample into a clean 1500mL separatory funnel.

9.2.2 Add 15mL of n-hexane to the emptied sample bottle, and re-cap with the container's lid. Shake the bottle to rinse the interior surfaces well with hexane. Pour the solvent rinse into the separatory funnel containing the sample. Repeat this Step.

9.2.3 For the MB, LCS/LCSD and MS/MSD QC samples, add 30mL hexane to each separatory funnel.

9.2.4 Re-fill the sample bottle to the mark with tap water, then pour the tap water into a graduated cylinder and measure what the volume of the original sample was. Record the volume (Vs in liters) on the benchsheet.

9.2.5 Extract the sample in the separatory funnel by shaking for two minutes, vent as needed to minimize pressure buildup.

9.2.6 Allow the organic phase (top solvent layer) to separate from the aqueous phase for a minimum of 10 minutes.

If an emulsion forms between the two phases, that is greater than ⅓ the volume of the solvent layer, break the emulsion using one of the following mechanical techniques:

- Gentle stirring
- Addition of sodium chloride
- Filtration through glass wool
- Drain emulsion into a glass centrifuge tube, spin to break emulsion
- Use an ultrasonic bath cooled with ice

The optimum technique to employ depends on the extent of the emulsion formed. Consult Supervisor for details as to how a particular technique should be performed.

9.2.7 Place approximately 10g of anhydrous sodium sulfate into a filter funnel with filter paper and rinse with a small portion of n-hexane. Discard the rinsate appropriately. Place a pre-weighed boiling flask containing boiling chips under the filter funnel.

9.2.8 Drain the water (aqueous phase) from the extracted sample in the separatory funnel, into a 1500mL Erlenmeyer flask and momentarily set aside.

Then, suspend the separatory funnel over the filter funnel containing the pre-wetted sodium sulfate, and drain the n-hexane layer (solvent phase) through the sodium sulfate (drying agent) and into the boiling flask.

Rinse the tip of the separatory funnel with a few mL of hexane, allowing the rinse to pass through the sodium sulfate and into the boiling flask.

9.2.9 Return the sample aqueous phase that was momentarily set aside to the separatory funnel; add 30mL of hexane.

Extract as before by shaking for two minutes, venting as needed to minimize pressure buildup.

Allow the phases to separate for a minimum of 10 minutes; treat emulsion, if formed, as necessary.

Drain the water phase from the separatory funnel into the same 1500mL Erlenmeyer flask and momentarily set aside.

Suspend the separatory funnel over the same filter funnel containing sodium sulfate, and drain the solvent phase through the sodium sulfate into the same boiling flask containing the first extracted solvent portion.

Rinse the tip of the separatory funnel with a few mL of hexane, allowing the rinse to pass through the sodium sulfate and into the boiling flask.

9.2.10 Repeat Step 9.2.5 again, resulting in a total of three extractions of the aqueous sample using 30mL portions of n-hexane, passing the post-extraction solvent portions through the sodium sulfate drying agent, and combining the dried solvent phases into the same pre-weighed boiling flask.

9.2.11 To ensure quantitative transfer of the HEM from the drying column (filter funnel with sodium sulfate), rinse the sodium sulfate with 10mL of hexane, collecting the rinse in the same boiling flask..

9.2.12 A milky extract (i.e., contents of the boiling flask) indicates the presence of water. If the extract is milky, allow the solution to stand for up to one hour to allow the water to settle. Decant the solvent (upper) layer through sodium sulfate (use funnel and filter paper setup as previously described) to remove any excess water. Collect in a clean pre-weighed boiling flask. Rinse the initial boiling flask, filter paper and sodium sulfate, with small portions of n-hexane to ensure a quantitative transfer.

9.3 DETERMINATION

9.3.1 Evaporate the hexane extract contained in the boiling flask on top of the S-Evap unit. The temperature of the S-Evap should be maintained at <85°C to ensure that the more volatile components extracted into the hexane are not lost (note that stearic acid begins to volatilize at 90°C).

9.3.2 Bring flask to dryness. Evacuate remaining hexane vapors using a vacuum pump with hose.

9.3.3 Move the boiling flasks to a desiccator and store for several hours.

9.3.4 Weigh each flask and record on benchsheet. Return flask to desiccator, store again for several hours, then re-weigh. Repeat this step until a constant weight is achieved (<5.0 mg of previous weight – see section 10.5.5).

9.3.5 If the residue does not look typical (i.e., affected by watery extract or contaminated with sodium sulfate), then re-dissolve the residue in n-

hexane, filter though a fresh drying funnel containing sodium sulfate, ensuring a quantitative transfer, and repeat evaporation process.

9.3.6 Calculate HEM per Section 11 below.

9.4 SILICA GEL TREATED HEM (SGT-HEM)

Silica gel cleanup is generally not performed unless the client specifies the need for separate data for HEM and SGT-HEM analytes. Also, if no HEM is detected, then there is no reason to proceed with the silica gel treatment.

9.4.1 If silica gel treatment is needed, then the MB and LCS (LCSD) must also be carried through the silica gel treatment. If the sample associated with the MS/MSD does not require silica gel treatment, then there is also no need to perform the silica gel treatment on the MS/MSD.

9.4.2 Re-dissolve the HEM in 80-90mL of n-hexane; swirl as necessary.

9.4.3 The amount of silica gel to use for treatment can be adjusted (up to 30g) based on the known weight of HEM. A 3.0g amount of silica gel can adsorb approximately 100mg of HEM. Add 3.0±0.3g of silica gel to the boiling flask for every 100mg of HEM measured. Add a PTFE-coated stir bar and stir on a magnetic stirrer for approximately 5 minutes.

9.4.4 Filter the solution through hexane-rinsed filter paper, into a pre- weighed boiling flask containing a few boiling chips. Rinse the sample flask, filter paper and funnel with a few aliquots of hexane (up to about 15mL total); evaporate the hexane on the S-Evap unit as described previously.

9.4.5 Cool in desiccator and determine constant weight as described previously (Steps 9.3.3. and 9.3.4).

9.4.6 Calculate SGT-HEM per Section 11 below.

10. SAMPLE EXTRACTION – SOLID PHASE EXTRACTION

10.1 Sample Preparation

10.1.1 All analytical samples should be brought to room temperature prior to analysis.

10.1.2 If sample is high in suspended solids, allow solids to settle prior to decanting the liquid portion.

10.1.3 Verify that the pH is < 2 for all samples (field and QC).

10.1.3.1 If the pH is not < 2, add 3ml of H_2SO_4 or HCl and mix thoroughly. Check the pH. If necessary, add more acid and retest.

10.2 SPE Disk Conditioning

10.2.1 Place the filter support screen on the unit head (the screen should be resting on the glass.

10.2.2 Place an extraction disk into the filter gasket (filter should be inside the gasket, mesh side down, and fibrous side up).

10.2.3 Place gasket w/filter on the screen and center funnel on the head. Secure the assembly with the clamp (handle to the rear).

10.2.4 Attach a solvent wash collection flask to the sample elution port.

10.2.5 Ensure that the 3-way valve is positioned for flow to the sample container.

10.2.6 Rinse the disk and reservoir with 20 ml of n-hexane (30 ml if using 90 mm disks). Ensure sufficient solvent is used to cover the top of the disk.

10.2.7 Apply vacuum and draw a minimal amount of hexane through the disk.

10.2.8 Release vacuum and allow disk to soak for 1 minute.

10.2.9 Apply vacuum and draw remaining hexane through the disk.

10.2.10 Repeat Sections 10.2.5 through 10.2.9.

10.2.11 Allow the disk to dry under vacuum for 1 minute.

10.2.12 Add 10 ml of methanol to the reservoir (30 ml if using 90 mm disks).

10.2.13 Apply vacuum, draw a few drops of methanol through the disk, and then release vacuum. Ensure sufficient methanol is available to cover the disk.

10.2.14 Allow disk to soak in the remaining methanol for 1 minute.

Note: If disk goes dry after the methanol conditioning, repeat the conditioning process.

10.3 Sample Extraction

10.3.1 Ensure that the 3-way valve is positioned for flow to the primary SPE waste container.

10.3.2 Pour/decant the sample into the SPE reservoir.

10.3.3 Apply vacuum and draw sample through the disk as quickly as possible. (Extract as much as liquid as possible prior to adding any sample sediment to the reservoir. Do not allow the disk to go dry prior to adding sediment.)

10.3.4 Allow disk to dry under vacuum for 3-4 minutes but no more than 5 minutes.

10.4 Sample Elution

10.4.1 Remove solvent wash collection flask (Section 13.2.4) from the solvent collection port.

10.4.2 Gently attach a Sodium Sulfate drying cartridge to the sample elution port.

10.4.3 Refer to Section 9.1 for flask preparation, then attach a clean, tared, appropriately labeled sample collection flask to the collection port.

10.4.4 Position the 3-way valve to allow flow into the sample collection flask.

10.4.5 Add 10 ml of hexane to the original sample container, swirl to rinse walls, cap, and invert 2-3 times.

10.4.6 Transfer the hexane to the disk. Ensure that reservoir walls are rinsed with hexane.

10.4.7 Apply vacuum, allow a few drops of hexane to pass through the disk, and then release vacuum.

10.4.8 Allow the remaining hexane to soak into the disk for no more than 2 minutes.

10.4.9 Carefully apply vacuum and slowly draw the remaining hexane drop wise through the disk into the collection flask.

10.4.10 Rinse the reservoir walls with 10 ml of hexane. Repeat steps 10.4.5 through 10.4.9 two to three (2-3) times, or until hexane level in collection flask is just below tip of sodium sulfate cartridge.

10.4.11 Apply vacuum and draw the hexane into the collection flask.

10.4.12 Allow the disk to dry for approximately 5 minutes before releasing vacuum.

10.5 Gravimetric Determination

10.5.1 Remove the sample collection flask from the manifold.

10.5.2 Place the collection flask in a Rapid Vap® evaportation unit heated to ~50 ^{o}C, for ~ 30 minutes.

10.5.3 When the flask appears to be dry, remove from Rapid Vap® unit, wipe dry, gently remove any remaining hexane with vacuum, and place in a dessicator.

10.5.4 Maintain flask in the dessicator for at least 30 minutes and then weigh.

10.5.5 Repeat Section 10.5.4 until the weight loss is less than 5.0 mg of the previous weight. Record the final weight in the logbook.

10.5.6 Measure the volume of sample as marked (see Section 9.2.1) in a 1L graduated cylinder.

11. CALCULATIONS

11.1 Calculate HEM as follows:

$$\text{SampleHEMConc.}(\text{mg}/\text{L}) = \frac{W_h\,(\text{mg})}{V_s\,(\text{L})}$$

where:

W_h = weight of extractable material (Step 10.3.4)

Vs = sample volume (Step 10.2.3)

11.2 Calculate the recovery of the OPR QC spike using the following equation:

$$\text{Recovery (\%R)} = \frac{\text{measuredconc.of HEM}}{\text{spikedconc.of HEM}} \text{ X } 100$$

See attached QC Table. Note that Method EPA 1664A provides different ongoing precision and recovery limits for the HEM vs SGT-HEM laboratory control sample.

11.3 Calculate the recovery of the MS/MSD QC samples as follows:

$$\text{Recovery (\%R)} = \frac{\text{HEM}_{\text{Found}} - \text{HEM}_{\text{Sample}}}{\text{HEM}_{\text{Target}}} \text{ X } 100$$

where:

$\text{HEM}_{\text{Found}}$ = calculated HEM concentration in the MS or MSD

$\text{HEM}_{\text{Sample}}$ = calculated HEM concentration in the field sample

$\text{HEM}_{\text{Target}}$ = the target concentration of the added HEM spike

11.4 Calculate the Relative Percent Deviation (RPD) of the MS/MSD QC samples using the following equation:

$$\text{RPD } (\%) = \frac{\left|HEM_{MS} - HEM_{MSD}\right|}{\left(\frac{HEM_{MS} + HEM_{MSD}}{2}\right)} \text{ X } 100$$

where:

HEM_{MS} = calculated HEM concentration in the MS

HEM_{MSD} = calculated HEM concentration in the MSD

11.5 Calculate the concentration of SGT-HEM as follows:

$$\text{Sample SGT} - \text{HEMConc.}(mg/L) = \frac{W_h\,(mg)}{V_s\,(L)}$$

where:

W_h = weight of silica gel treated extractable material (Step 10.4.5)

Vs = sample volume (Step 10.2.3)

12. QUALITY CONTROL

12.1 DEFINITION OF BATCH

A batch is defined as a group of ≤20 field samples of like matrix that is associated with one unique set of batch QC samples and processed as a unit. Batch QC samples are defined as the MB, LCS (LCSD) and matrix spike and duplicate (MS/MSD). All QC samples must be carried through all stages of the sample preparation and measurement steps.

12.2 METHOD BLANK

Method blanks are aliquots of clean matrix (i.e., laboratory DI water) that have been prepared and analyzed in the same manner as the associated field samples. To be acceptable, concentrations of analytes of interest detected (if any) in the MB must be below the analyte reporting limit, or as otherwise specified in the LIMS program specification. If this criterion is not met, analyses should be halted and the source of the contamination found and corrected. See also attached QC Table.

12.3 OPR QC SAMPLE

This sample is comprised of a known concentration of target analyte contained in a clean matrix. This laboratory control sample (LCS) is analyzed to measure the accuracy of the analytical system. LCS recovery is calculated as shown in Section 11. See QC Table for evaluation criteria.

12.4 MS AND DUPLICATE

Matrix spikes consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection and recovery. MS recovery is calculated as shown in Section 11, see QC Table for evaluation criteria.

A laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. To accomplish this analysis, either a field sample may be analyzed in duplicate (DUP), or the laboratory control sample (LCSD) or matrix

spike analysis (MSD) can be performed in duplicate. Recovery is calculated as shown in Section 11, and precision (see Section 11 for calculation) is evaluated in terms of RPD. See QC Table for acceptance limits.

Possible causes for matrix spiked failure include:

- Sample heterogeneity
- A sample matrix which inhibits extraction of spiked compounds
- High levels of HEM that "swamp out" the small amount of extractable materials added with the OPR spike.

NOTE: Typically, one MS and MS Duplicate pair is analyzed per batch. In the event that not enough sample volume is provided to generate MS and duplicate analyses, the requirement to perform these analyses is waived and an explanatory notation is made in the data package narrative.

For projects in which the client designates the MS/MSD samples, an analysis batch may not contain an MS/MSD pair because the required spikes were included in another batch. Where this occurs, a notation will be made in the data package narrative.

13. DEVIATIONS FROM METHOD

This procedure complies with the requirements of Methods EPA 1664A and SW9070A. There are no known deviations from the Methods.

14. SAFETY, HAZARDS AND WASTE DISPOSAL

14.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

14.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

15. REFERENCES

15.1 US EPA, EPA 821-R-98-002, February 1999, "N-Hexane Extractable Material (HEM: Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons)", Method 1664A, Revision A.

15.2 US EPA SW-846, Test Methods For Evaluating Solid Waste Physical/Chemical Methods, "Method 9070A", Update IIIA, April 1998.

Analytical Method: EPA 1664A; SW 9070A	**Parameter:** n-Hexane Extractable Material (HEM)		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria *	Corrective Action
* NOTE: Superceding criteria specified by the client and prescribed in the LIMS program specification may apply.			
Method Blank (MB)	One per each batch of ≤20 field samples; one each time a reagent is changed	MB must not yield HEM content above the 5.0 mg/L reporting limit (RL)	Check all calculations. If no computation errors are found, prepare a fresh MB and analyze, associated samples must also be re-extracted and re-analyzed (if possible).
On-going Precision and Recovery (OPR) Sample; Laboratory Control Sample (LCS)	One per batch of ≤20 field samples	Results obtained must be within 79-114% of expected (known) concentration of HEM,and 64-132 % for SGT-HEM	Check calculations and preparation for documentable errors. If no errors are found, reanalyze OPR, associated samples must also be re-extracted and re-analyzed (if possible). If samples cannot be re-extracted, narrate.
Matrix Spike (MS)	One per batch of ≤20 field samples	Results obtained should be within 79-114% of expected concentration of HEM, and 64-132 % SGT-HEM	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated OPR is within control limits, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike Duplicate (MSD)	One per batch of ≤20 field samples	(See MS recovery criteria above) HEM RPD should be ≤18%; SGT-HEM RPD ≤34%	See MS recovery corrective actions above. For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/Project/QA Managers.

APPENDIX A

Page ___ of ___

OIL & GREASE -- PREPARATION / ANALYSIS / CLEANUP – BENCHSHEET

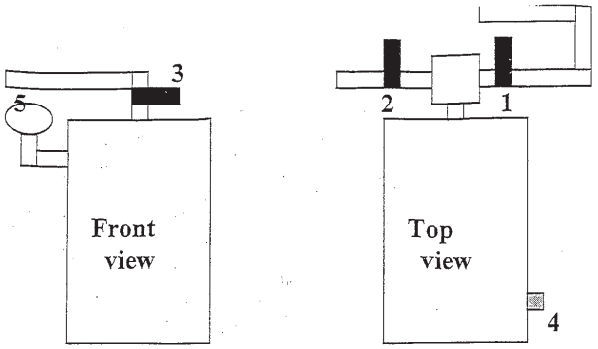
WO _____ AQ Solid _____ **Batch ID** _____ **M Spike Code** _____ **Analysis Code:** 9070A 9071 1664 Lipids **SOP**_____ **Rev**___ **Initials**_____
Bal. ID _____ **2 mg wt. ID #45458 #35069 2 mg check between 0.0019 – 0.0021g?** Y / N **Ig Check between 0.9900 – 1.0100g?** Y / N
Ex. Date/Time_____**(start)** **Date/Time**_____**(stop)**

Cleanup Code: (3630C) Silica Gel **Cleanup Date / Time** _____**SOP 644/Rev**____ **Initials**_____

SAMPLE					EXTRACT					COMMENTS
Sample No.	**Amount (g / mL)**	**Matrix Spike (mL)**	Matrix Spike	**Acidified? If so, amount used? (mL)**		**Silica Gel (g)**	**Beaker Initial (g)**	**Beaker Final (g)**	**Difference (g)**	

Reagent Lots: HCl _____ **H_2SO_4** _____ **Silica Gel**_____ **Hexane** _____ **$NaSO_4$** _____ **DCM** _____

Note: Each logbook page is copied as completed and included with the workorder/run documentation; reviewed subsequently.



1. Water inlet valve
2. Steam blowout valve
3. Steam valve
4. Boiler switch knob
5. Steam pressure gauge

Steam Generator Start up

1. Check to see if steam blowout valve[2] is closed. (perpendicular to pipe) If not close it.
2. Open water inlet valve[1]. (parallel to pipe)
3. Turn boiler switch knob[4] on to 250. If boiler switch light does not come on re-check in five minutes. Light should be on. If light is not coming on contact supervisor.
4. After approximately 30 minutes Steam pressure gauge[5] should read between 20-60 psi. If not, wait until desired pressure is reached.
5. Open steam valve[3]. (parallel to pipe)

Steam Generator Shutdown

Caution: Pipes and Generator will be hot!

1. Turn boiler switch knob[4] to OFF
2. Close water inlet valve[1]. (perpendicular to pipe)
3. Close steam valve[3]. (perpendicular to pipe)
4. **SLOWLY*** open steam blowout valve[2]. (parallel to pipe)

**Caution: Do not open blowout valve fully until a majority of the steam is released. The tubing carrying the steam outside may come out of the wall.*

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA EMISSION SPECTROSCOPY
REFERENCED METHOD:	EPA 200.7 (TRACE ICAP)
SOP ID:	807
REV. NUMBER:	13
EFFECTIVE DATE:	11/12/2013

STANDARD OPERATING PROCEDURE 807 REVISION 12

TITLE: DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA EMISSION SPECTROSCOPY -- METHOD EPA 200.7 (TRACE ICAP)

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the method it references -- Method EPA 200.7 -- is used to determine the concentration of total or dissolved metals in aqueous samples. Analytes are viewed axially, providing detection limits for many analytes similar to those that may be achieved using Graphite Furnace Atomic Absorption (GFAA) analysis.

The following elements are routinely determined using this method:

Aluminum (Al)	Antimony (Sb)	Arsenic (As)	Barium (Ba)	Beryllium (Be)
Cadmium Cd)	Calcium (Ca)	Chromium (Cr)	Cobalt (Co)	Copper (Cu)
Iron (Fe)	Lead (Pb)	Lithium (Li)	Magnesium (Mg)	Manganese (Mn)
Molybdenum (Mo)	Nickel (Ni)	Phosphorous (P)	Potassium (K)	Selenium (Se)
Silicon (Si)	Silver (Ag)	Sodium (Na)	Strontium (Sr)	Thallium (Tl)
Tin (Sn)	Titanium (Ti)	Uranium (U)	Vanadium (V)	Zinc (Zn)

2. SUMMARY OF METHOD

Samples are digested and prepared prior to analysis in accordance with Method EPA 200.7, per SOP 806. Filtered liquid samples, liquid samples containing low solids, or leachates may also be analyzed by direct aspiration into the ICAP instrument (i.e., without prior digestion). A computer-controlled Inductively Coupled Argon Plasma (ICAP) Trace Analyzer is used to accomplish the analyses.

Samples or sample digestates are aspirated into the ICAP instrument, into a high temperature argon plasma stream. Radio frequencies are generated to induce excitation of the plasma stream that causes constituent elements contained in the sample to emit light at characteristic wavelengths. A grating spectrometer is used to disperse the resulting spectra.

The light emissions are received by a photomultiplier tube, which in turn transmits a signal to the data acquisition system. The software of the data acquisition system interprets the signal by comparing it to a previously calibrated standard curve. The data are then further manipulated in the reporting process to incorporate such factors as dilution, etc.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALSALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALSALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 SPECTRAL INTERFERENCES

Potential spectral interferences include the following:

4.1.1 Overlap of a spectral line from another element at the analytical or background measurement wavelengths. Spectral overlap may be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element (see Section 8.8 for a description of this correction).

4.1.2 Unresolved overlap of molecular band spectra.

4.1.3 Background contribution from continuum or recombination phenomena.

4.1.4 Stray light from the line emission of high concentration elements. Background contribution and stray light may usually be compensated for by a background correction adjacent to the analyte line.

4.2 PHYSICAL INTERFERENCES
Effects associated with the sample nebulization and transport processes are considered physical interferences. Changes in viscosity and surface tension may cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the samples.

4.3 CHEMICAL INTERFERENCES
Molecular compound formation, ionization effects, and solute vaporization effects are chemical interferences. The most significant potential interference in the Trace ICAP instrument is ionization effects caused by varying levels of easily ionized elements in samples and standards. The ionization effects can be reduced by adding a constant amount of an easily ionized element (such as lithium) as an "ionization buffer" to all solutions. The lithium is added using a peristaltic pump that delivers a constant flow of lithium solution to the sample delivery tubing through a "T" connector.

5. APPARATUS AND MATERIALS

5.1 Autosampler: Thermo Jarrell Ash Model AS300 or equivalent

5.2 ICAP: An argon plasma trace analyzer (e.g., Thermo Jarrell Ash ICAP 61E Trace Analyzer), set to simultaneous operating conditions and containing the following:

- An axially mounted torch
- An R.F. (radio frequency) generator; set at 27.12MHz, 2kW (i.e., an inductively coupled argon plasma excitation source)
- Holographic grating, 2400 grooves/nm, blazed at 500nm
- A 0.75m Rowland Circle spectrometer (polychromator) or equivalent, with a Paschen-Runge mount and capable of accepting up to 63 channels
- An automated instrument control and data acquisition system (i.e., personal computer or equivalent) capable of providing various (i.e., background, interelemental) corrections
- Thermospec™ version 6.20 or higher or ICAP Manager™ version 6.10 or higher software, or equivalent

5.3 Volumetric flasks, various sizes, of suitable precision and accuracy

5.4 Volumetric pipets, fixed or adjustable, verified per SOP 321

6. REAGENTS AND STANDARDS

6.1 Hydrochloric acid (HCl), concentrated, JT Baker #9530-33 or equivalent

6.2 Nitric acid (HNO_3), concentrated, JT Baker #9598-34 or equivalent

6.3 Reagent water, deionized (DI) water obtained from the laboratory's DI water system (SOP 319)

6.4 Liquid Argon, 99.99% pure

6.5 STANDARDS

6.5.1 All standard solutions are prepared, documented, and stored in accordance with ALSALS SOP 300. All standard solutions are to be contained in fresh (previously unused) polypropylene bottles.

6.5.2 Detailed documentation of all standards associated with each ICAP acquisition (analytical sequence) is recorded in the Header Information of the ICAP software, and included with the associated raw data. This documentation includes the stock and intermediate standard identification numbers, dilutions performed to create the working standards, and the resulting concentrations of the working standards. (Further details pertaining to header information is provided in Section 8.4).

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Liquid samples are collected in plastic or glass containers, and must be chemically preserved with nitric acid to pH<2. Samples for dissolved metals analyses should be filtered in the field prior to chemical preservation. If samples are not preserved in the field, they may be acidified by the laboratory upon receipt, but must be held in their original container for a minimum of 16 hours before transfer or analysis of the sample.

7.3 Samples must be maintained at 4±2°C, and must be prepared and analyzed within 180 days of collection.

8. PROCEDURE

8.1 TYPICAL OPERATING CONDITIONS

Torch Gas:	High Flow
Auxiliary Gas:	Low
Nebulizer Gas:	25 PSI
RF Power:	1150 W
Pump Rate:	125 rpm
Sample Tubing:	Orange/Orange
Rinse Tubing:	Red/Red

8.2 The instrument is calibrated each day, by analyzing and processing multi-point calibration curves for each element quantitated. Second order (quadratic) calibration equations with at least 5 points are used to fit the calibration data and to determine concentration results.

8.3 The typical autosampler analytical sequence is listed below along with brief descriptions of each solution. Refer to attached quality control (QC) Table at end of SOP for performance criteria:

Autosampler Run Number	***Solution Name***	***Description***
1	Mix A	Reanalysis of the highest calibration standard for Mix A elements. Processed as a sample.
2	Mix B	Reanalysis of the highest calibration standard for Mix B elements. Processed as a sample.
3	Mix C	Reanalysis of the highest calibration standard for Mix C elements. Processed as a sample.
4	ICV	Initial Calibration Verification check standard for all elements (second source).
5	ICB	Initial Calibration Blank. Must be run following the multi-point calibration and before any samples are analyzed.
6	ICSA	Interference Check Solution A (contains high concentrations of Ca, Mg, Al, Fe).
7	ICSAB	Interference Check Solution B (contains high concentrations of Ca, Mg, Al, Fe and low concentrations of other elements).
8	CRI	Low concentration test solution containing analyte concentrations near the reporting limit; analysis of this solution is not described in Method 6010B. Run for informational purposes only, ALSALS does not control on CRI recovery, unless required by client LIMS program specification.
9	CCV	Continuing Calibration Verification check standard for all elements (second source).
10	CCB	Continuing Calibration Blank. Must follow CCV analyses.
11 thru 20	Samples	Additional analytical samples. Analytical samples include all samples analyzed on the instrument except ICV, ICB, CCV and CCB. The CRI, ICSA and ICSAB, along with all samples in the sequence and any dilutions, post-spikes (see Section 8.6.4), laboratory and matrix spikes, duplicates, serial dilutions and method/ preparation blanks, are all considered to be analytical samples. The sequence is closed out with the following solutions: CRI, ICSA, ICSAB (analyzed at the end of each analytical sequence, and every eight hours if the analytical sequence is longer than eight hours), followed by CCV, CCB.

Autosampler Run Number	*Solution Name*	*Description*
21	CCV	Continuing Calibration Verification check standard for all elements (second source).
22	CCB	Continuing Calibration Blank, must follow CCV analyses.
Repeat (11 through 22); the sequence continues with CCV and CCB analyzed after every 10 analytical samples.		

8.4 After the analytical sequence is complete, a "header and summary" section is produced which includes:

- Standard information including standard identifications, expiration dates, elements and concentrations, and preparation procedures
- Acid lot numbers
- Pipet identification numbers
- Dilution information and preparation procedures
- Analytical spike information and preparation procedures
- Daily and monthly maintenance items performed. (Maintenance is further discussed in Section 8.10).
- Summary page with analytical sequence and elements of interest

8.5 At the completion of the sequence, the instrument is shut down as follows:

8.5.1 Disconnect pump tubing.

8.5.2 Lower "purge optics" gas flow to approximately 1L/min.

8.5.3 Exit the software.

8.6 PREPARATION AND EVALUATION OF QUALITY CONTROL (QC) SAMPLES

8.6.1 Calibration Blank (STD-Blank): An aliquot of reagent water is acidified in the same manner as the sample digestates. This calibration blank is used as a component in establishing the calibration curve and is also analyzed repeatedly throughout the analytical sequence as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB). To be acceptable, the calibration blank cannot contain any analyte above the analyte reporting limit (or as otherwise specified in the applicable LIMS program specification). Refer to Section 10.1 for further discussion.

8.6.2 Method Blank (MB): Referred to as a reagent blank in Method 200.7 (40 CFR). One MB is prepared per batch of 20 or less field samples. The method blank consists of an aliquot of reagent water that has been digested and prepared in the same manner as the associated samples. To be acceptable, the method blank cannot contain any analyte above the

analyte reporting limit (or as otherwise specified in the applicable LIMS program specification). Method blank results are also acceptable if sample concentrations are greater than 10 times the concentration found in the method blank. Refer to Section 10.5 for further discussion.

8.6.3 Sample Duplicate: One sample duplicate is prepared per batch of 10 or less field samples. The control limit for duplicate precision is that the relative percent difference (RPD) must be ≤20%. RPD is calculated as shown below:

$$\text{RPD} = \left(\frac{\text{concentration sample} - \text{concentration duplicate}}{1/2\,(\text{concentration sample} + \text{concentration duplicate})} \right)(100)$$

The results are flagged if duplicate precision is greater than 20% RPD. Refer to Section 10.6 for further discussion.

8.6.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): One MS and MSD are prepared per batch of 10 or less field samples. MS and MSD samples consist of additional aliquots of a particular field sample that are spiked, digested and prepared in the same manner as the associated samples.

Matrix spike samples are evaluated in terms of recovery, calculated as follows:

$$\%\text{R} = \left(\frac{\text{concentration detected} - \text{conc. sample}}{\text{concentration spiked}} \right)(100)$$

Matrix spike recovery is not evaluated if the analyte concentration in the unspiked sample is greater than 4 times the spike level. The quality control limit for matrix spike recovery is 70 to 130%. The control limit for MS/MSD precision is that the RPD must be ≤20%. RPD for the MS/MSD is calculated the same as for the Sample Duplicate (see Section 8.6.3 above). Results are flagged if MS/MSD recovery or precision results are outside control limits.

A post digestion spike analytical spike (i.e., sample aliquot is spiked after digestion) should be performed when matrix spike recovery is outside the control limit. If recovery of the post digestion analytical spike is not within 90-110%, the result is flagged indicating that matrix interference is suspected. Refer to Section 10.8 for further discussion.

8.6.5 Laboratory Control Sample (LCS): One laboratory control sample is prepared per batch of 20 or less field samples. The LCS is a water sample with known analyte concentrations that is digested and prepared in the same manner as the associated samples. LCSs are evaluated in terms of recovery (%R), calculated as follows:

$$\%R = \left(\frac{\text{concentration detected}}{\text{concentration spiked}} \right)(100)$$

The control limits for LCS recovery are 85-115%. All samples associated with a failed LCS must be redigested and reanalyzed. A low level LCS will be digested and prepared for drinking waters; it will be prepped in the same manner as the associated samples. The analyte concentrations will be near the RL. The low level LCS recovery limits are 70-130%. Refer to Section 10.9 for further discussion.

8.6.6 Serial Dilution Sample: A 1:5 serial dilution of an additional sample digestate is prepared per 20 field samples of like matrix that are processed as a unit. If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within ±10 % of the undiluted results. Sample analyte results failing this test should be flagged indicating the existence of matrix interferences. Refer to Section 10.10 for further discussion.

8.7 LINEAR RANGE

The element concentrations in the Mix A, Mix B, and Mix C High Standards define the upper end of the analytical range. Samples whose concentrations exceed the calibration range must be diluted to bring their concentrations within the known calibration range of the instrument.

8.8 INTERELEMENT SPECTRAL INTERFERENCE CORRECTIONS

Interelement spectral interferences are determined by analyzing a solution that contains a high concentration of a potentially interfering element and observing the "apparent" concentration arising from the solution in other element channels. Interelement interference correction factors are calculated from these observations. *For example*: a 500ppm Al solution produces an apparent Pb concentration of 0.15ppm. The interelement correction factor K is calculated as follows:

$$K = \frac{Apparent\ Conc.\ in\ ppm}{Conc.\ of\ \ Interfering\ element\ in\ ppm}$$

In this example, K = 0.15 / 500 = 0.00030.

The interelement correction factor K is the amount of interference produced by 1ppm of the interfering element on the element being interfered with. Interference correction factors are used by the ICAP software to calculate corrected concentrations using the following equations:

$$Corrected\,Conc.(ppm) = Uncorrected\,Conc.(ppm) - \left(K * Conc.of\ Interfering\,Element(ppm)\right)$$

High concentrations of elements (such as Fe and Al in solid digestates) are the most likely sources of significant spectral interferences. The ICSA and ICSAB are

analyzed at the beginning and end of each analytical sequence to verify that the spectral interferences arising from Al, Fe, Ca, and Mg are being corrected properly.

An interelement interference study is conducted every six months by analyzing single element solutions of each analyte at high concentrations. The study is used to verify or update interelement interference correction factors. Because the instrument is a direct reading polychromator with fixed detectors, interelement interference correction factors usually remain quite constant.

8.9 In addition to the electronic run information provided by the instrument's output (analyst, date, time, sample ID, etc.), a hardcopy Run Log is maintained as an internal Departmental record of instrument throughput. Standard intensities for selected elements are noted as comments, these recordings are used to verify that operating conditions remain the same. If a variation or trend is noticed in intensity readings, the cause should be determined and corrected if necessary.

8.10 REGULAR MAINTENANCE ITEMS
The following items should be checked prior to each run to ensure the instrument is in good working order.

- Check argon level; order more as needed
- Check filters on rear of instrument and vacuum monthly
- Check water level in drain bottle and empty if necessary
- Check pump tubing -- replace when necessary
- Check that the previous day's work is properly recorded and processed

There is a section in the raw data header information where the Regular Maintenance items may be initialed as completed.

8.11 MAINTENANCE LOG
A maintenance logbook is used to record all information concerning instrument maintenance that is not covered by the daily and monthly maintenance items described previously. This logbook is used to document all repairs and the symptoms of the problems.

9. QUALITY CONTROL

9.1 Various quality control indicators are discussed in Sections 8.3 and 8.6.

9.2 A method detection limit (MDL) study consisting of the analysis of a minimum of seven replicate aliquots of target analytes at concentration levels 3-5 times the anticipated detection limit, shall be performed as needed, and at a minimum, annually. See SOP 329

9.3 Instrument detection limits (IDLs) reflect instrument capability and are determined per CLP protocol. See SOP 329 .

10. METHOD MODIFICATIONS

10.1 Section 12.1.1 of EPA Method 200.7 (40 CFR) recommends that the results for calibration blanks "should be within 2 standard deviations of the meal *(sic)* value." The intent of this statement is most likely that calibration blanks should be within ±2 standard deviations of the 'mean' value. ALSALS follows a different criteria for calibration blanks than the one recommended. ALSALS's criteria require that the calibration blank results be less than the reporting limit (ICB, CCB).

Method 200.7 (40 CFR) does not define the data set to be used for determining the control limits. ALSALS notes that several factors could significantly affect data used for calculation of control limits. Some examples include: different acid lot numbers; instrumentation changes (nebulizer, torch); the number of data points used; and the frequency of updating the control limits. A significant problem with the recommendation given in Section 12.1.1, is that a control limit of ±2 standard deviations of the mean value could allow blank results to be higher than the reporting limit. ALSALS's criteria for evaluating calibration blanks are clearly defined and allow for straightforward data review and validation.

10.2 Section 12.1.2 of EPA Method 200.7 (40 CFR) recommends that results for the interference check sample "should fall within the established control limits of 1.5 times the standard deviation of the mean value." A control limit of 1.5 times the standard deviation of the mean value shows that the interelement spectral interference correction routines are operating reproducibly, but this control limit does not provide assurance that the corrections are accurate. ALSALS analyzes 2 interference check samples (named ICSA and ICSAB) rather than one interference check sample as described in Method 200.7 (40 CFR). These solutions are analyzed at the beginning, end, and periodically throughout an analytical sequence. The ICSA solution contains Ca, Mg, Al and Fe at the upper analytical range concentration and no other analytes. Ca, Mg, Al and Fe are the elements most likely to cause significant spectral interferences in environmental samples. The control limits used by ALSALS for the ICSA results require that no analyte concentrations (other than Ca, Mg, Al and Fe) may exceed the absolute value of 2 times the reporting limit. The ICSAB solution contains Ca, Mg, Al and Fe at the upper analytical range concentration and the other analytes at low concentrations. The control limits used by ALSALS for the ICSAB results requires all analyte recoveries to be within ±20% of the known concentrations. The analysis of the ICSA and ICSAB solutions verifies that the spectral interference correction routines are functioning at concentrations near the reporting limit and also at concentrations above the reporting limit. ALSALS control limits for the ICSA and ICSAB solutions are clearly defined and allow for straightforward data review and validation.

10.3 ALSALS analyzes a CRI solution at the beginning and end of each sequence. This CRI solution provides assurance that the instrument sensitivity is adequate to support the reporting limit. Method 200.7 (40 CFR) does not describe the analysis of a low level test solution as part of an analytical sequence.

10.4 Section 12.1.1 in EPA Method 200.7 (40 CFR) states: "Analyze and *(sic)* appropriate instrument check standard at a frequency of 10%". This is assumed to mean that an appropriate instrument check standard should be analyzed. The control limit given in Method 200.7 (40 CFR) is ±5% of the true value. This control limit has been found to be too stringent to allow for the routine analysis of samples with widely varying matrix constituents. Instrumental drift is caused by samples containing high levels of dissolved constituents, which perturb the nebulizer, and changes in ambient temperature. ALSALS's control limit for CCVs is ±10% of the true value. The ±10% control limit is also given in EPA Method 200.7 (EPA 600). The ±10% control limit provides adequate assurance that instrumental drift has not significantly affected quantitation.

10.5 Section 11.1 of Method 200.7 (40 CFR) recommends, "reagent blanks should be subtracted from all samples". No criteria are given in Method 200.7 (40 CFR) defining the maximum allowable analyte concentration in reagent blanks. ALSALS does not subtract method blank results from samples. If reagent (method) blank results are either above the reporting limit or are greater than 1/10 the concentration found in samples, then all associated samples are re-digested and re-analyzed.

10.6 EPA Method 200.7 (40 CFR) does not describe the preparation and evaluation of duplicate samples. ALSALS performs duplicate analyses at a frequency of 10% to provide a measure of the precision of the analytical results.

10.7 Method 200.7 (40 CFR) does not discuss the preparation and evaluation of digested matrix spike and matrix spike duplicate samples. ALSALS prepares and analyzes matrix spike and matrix spike duplicate samples at a frequency of 10%.

10.8 Section 5.2.2 of Method 200.7 (40 CFR) recommends that a spike addition test should be performed whenever a new or unusual matrix is encountered. It is assumed that the spike addition is performed on a digested sample (i.e., a post digestion analytical spike) because it is discussed in the same section (5.2) as the serial dilution test. It is not likely that a commercial laboratory will be able to determine if a matrix is new or unusual. Therefore, ALSALS uses matrix spike recovery results in each preparation batch to evaluate matrix interferences. The matrix spike recovery control limits (70-130%) are taken from Method 200.7 (EPA 600). If matrix spike recovery is outside the control limit, then a post digestion spike is prepared and analyzed. ALSALS uses the 90-110% control limit for post digestion spike recovery recommended by Method 200.7 (40 CFR).

10.9 Method 200.7 (40 CFR) does not describe the preparation and evaluation of a digested laboratory control sample (LCS). ALSALS analyzes a LCS with each sample batch to provide assurance that the digestion and analysis is in control. ALSALS's LCS recovery limit is 85-115%, which is taken from Method 200.7 (EPA 600).

10.10 Section 5.2 of Method 200.7 (40 CFR) recommends that serial dilution results should agree within ±5% of the original determination. The amount that the sample should

be diluted for the test is not defined. The difference between undiluted and diluted results usually becomes larger as the level of dilution increases. A ±5% criterion seems too stringent for samples with widely varying levels of constituents. ALS flags data if the serial dilution (5 fold dilution) results do not agree within ±10% of the original determination. The ±10% criterion is also suggested by Method 200.7 (EPA 600).

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 EPA Method 200.7, 40 CFR, Appendix C to Part 136.

12.2 EPA Method 200.7, US EPA 600/R-94/111, Revision 4.4, May 1994.

12.3 Operator's Manual, ICAP Trace Analyzers.

Analytical Method: EPA 200.7	**Parameter**: ICAP Metals		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, using, at minimum, 4 standards and a blank	Daily	Correlation coefficient for all analytes >0.995	Correct problem and repeat initial calibration
Reanalysis of Mix-A, Mix-B and Mix C calibration standards as samples	Immediately after calibration	All analytes within ±5% of expected value	Correct problem then repeat initial calibration
ICV (Initial Calibration Verification) check standard (second source); at or below midpoint	Daily after initiation calibration	All analytes within ±5% of expected value	Correct problem then repeat initial calibration
ICB (Initial Calibration Blank)	Immediately following ICV	Absolute value of result for each analyte < reporting limit (or as specified in the applicable LIMS program specification)	Correct problem then re-analyze ICB
CCV (Continuing Calibration Verification) check standard; concentration of analytes must be different from the ICV	After every 10 samples and at the end of the analytical sequence	All analytes within ±10% of expected value Note: (where compliance samples are analyzed for regulatory reporting purposes, a ±5% CCV must be maintained).	Repeat calibration and reanalyze all samples since last successful CCV
CCB (Continuing Calibration Blank)	Immediately after every CCV	Absolute value of result for each analyte < reporting limit (or as specified in the applicable LIMS program specification)	Correct problem then analyze CCB and previous 10 samples
ICSA (Interference Check Solution A)	At the beginning and the end of an analytical run	All analytes within ±20% of expected value	Terminate analysis, correct problem, reanalyze ICSA, reanalyze all affected samples
ICSAB (Interference Check Solution B)	At the beginning and the end of an analytical run	All analytes within ±20% of expected value	Terminate analysis, correct problem, reanalyze ICSAB, reanalyze all affected samples
MB (Method blank)	One MB per batch of 20 or fewer field samples	Absolute value of result for each analyte < reporting limit (or as specified in the applicable LIMS program specification)	Correct problem then reprep and analyze MB and all samples processed with the associated MB
LCS (Laboratory Control Sample)	One LCS per batch of 20 or fewer field	Recovery limit 85-115% for each analyte	Correct problem then reanalyze. If still out, reprep and reanalyze the

Analytical Method: EPA 200.7	**Parameter**: ICAP Metals		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
	samples	A low level LCS will be digested and prepared for drinking waters; it will be prepped in the same manner as the associated samples. The analyte concentrations will be near the RL. The low level LCS recovery limits are 70-130%.	LCS and all samples in the affected batch
Sample Duplicate	One sample duplicate per batch of 10 or fewer field samples	For each analyte RPD ≤20%	Flag results if RPD >20%.
MS/MSD (Matrix Spike/Matrix Spike Duplicate)	One MS/MSD pair per batch of 10 or fewer field samples	Recovery limit 70-130% for each analyte, not calculated if analyte conc >4X the spike level. For each analyte RPD ≤20%	Flag results if MS/MSD recovery or precision results are outside control limits, perform post digestion analytical spike as applicable
Post Digestion Spike	Performed when MS/MSD recovery is outside ± 30% (unless analyte conc > 4X the spike level)	Recovery limit 90-110% for each analyte	Flag results if post spike recovery or precision results are outside control limits
Serial Dilution sample analysis	Performed on one sample per batch of 20 or fewer field samples, where analyte concentrations exceed 50X IDL	Results should agree within ±10% of undiluted results	Flag results if outside criteria
Method Detection Limit (MDL) Study	As needed, at minimum, annually	Concentrations for the MDL study shall be at a level ≤ to that of the reporting limit	Determine the reason for failure and fix problem with system; repeat study for those analytes that did not meet criteria. If criteria still not met, discuss with Department and QA Managers (RL may be adjusted, if needed).

ALS Standard Operating Procedure

DOCUMENT TITLE:	PREPARATION AND DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY
REFERENCED METHOD:	SW7471A, EPA 245
SOP ID:	812
REV. NUMBER:	16
EFFECTIVE DATE:	08/28/2014

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 812 REVISION 16

TITLE: PREPARATION AND DETERMINATION OF MERCURY BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY -- METHODS SW7470A, SW7471A, EPA 245.1,
FORM NUMBERS: Appendix A and B

APPROVED BY:

PRIMARY AUTHOR____ _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the digestion procedures and instrument analysis of all matrices for mercury.

2. SUMMARY

Waters and TCLP leachates analyzed by methods SW7470A, EPA 245.1, are digested using the same basic procedure. A weighed portion of sample is digested using acids, potassium permanganate and potassium persulfate for liquid samples. Aqua regia and potassium permanganate are used for digesting SW7471A soil samples. After digestion, and the addition of hydroxlyamine sulfate, the digestate is analyzed using a cold vapor mercury atomic absorption spectrometer (CVAA). The mercury (Hg) is reduced to the elemental state by the addition of stannous chloride. The mercury vapor passes through a cell in the light path of an atomic absorption lamp that emits light at 253.7nm. The 253.7nm light is absorbed in proportion to the concentration of Hg atoms in the cell. The absorbance is measured as a function of mercury concentration by running a series of standards with each batch of samples. The typical reporting limits for this method are 0.0002mg/L and 0.0006mg/Kg.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 It is the responsibility of all personnel who work with samples or data involving this method to consult the applicable LIMS Program Specification for client-specific requirements prior to initiating handling of samples or data.

3.3 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, performance of precision and accuracy tests, or the successful analysis of an unknown proficiency test sample.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file indicates that this review for precision accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20mg/L or 20mg/kg of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

4.2 Copper has also been reported to interfere, however, copper concentrations as high as 10mg/L or 10mg/kg had no effect on recovery of mercury from spiked samples.

4.3 Samples that contain relatively high amounts of chlorides or oxidizable organic material require additional permanganate (as much as 25mL in a 100mL final volume) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7nm. Care must be taken to ensure 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 sulfate reagent (25mL).

4.4 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

4.5 Low level mercury sample preparation, digestion, and analysis may be subject to environmental contamination if performed in areas with high ambient backgrounds where mercury was previously employed as an analytical reagent in analyses such as total Kjeldahl nitrogen (TKN) or chemical oxygen demand (COD).

5. APPARATUS AND MATERIALS

- Mercury analyzer, automated, CETAC M-6000A, with QuickTrace software, version 1.6.5, or equivalent. Refer to QAM Appendix E1 for listing of instruments and software.
- Mercury vapor lamp, electro-optically regulated, low pressure, high frequency, thermally stabilized. Clean/replace as necessary.

- Nafion drying cartridge and mercury trap ($KMnO_4$), CETAC SP5894 or equivalent. Replace as necessary.
- Hot Blocks -- capable of maintaining a temperature of 95±5°C
- Thermometer -- for monitoring hot plate or water bath temperature. **Use only a mercury-free type of thermometer.**
- Beakers, polypropylene, disposable, 250mL - - used for soils
- SCP tubes with caps, disposable, 50mL, or 15 mL 17x100 polypropylene test tubes with caps - - used for liquids
- pH test strips, low range, acidic – capable of testing solutions < pH2
- Pipette(s), adjustable, 0.01-5.0mL
- Laboratory balance -- capable of weighing to 0.01g, verified per SOP 305
- Test tubes, 16X100mm
- Spatulas, wooden, disposable
- Boiling chips, Teflon™, ChemWare PTFE Chips, VWR Cat. No. 26397-103 or equivalent

6. REAGENTS

NOTE: Only trace metals grade acids may be used. The lot # must be verified by the Metals Department prior to the preparation/analysis of samples with any new acids. Each lot of acid must be certified (no metals detected at or above the MDL).

6.1 Reagent water/deionized (DI) water, interference free

6.2 Liquid argon, 99.99% pure

6.3 Sulfuric acid (H_2SO_4), concentrated, EMD SX1247-2 or equivalent

6.4 Nitric acid (HNO_3), concentrated, JT Baker 9598-34 or equivalent

6.5 Hydrochloric acid (HCl), concentrated, JT Baker 9530-33 or equivalent

6.6 Stannous chloride solution (10% w/v): CCI 5515AL or equivalent. Dissolve a ratio of 25g stannous chloride with 25mL HCl up to a 250mL final volume with reagent water for a final concentration of 10%.

6.7 Sodium chloride - hydroxylamine sulfate solution (12% w/v): EMD SX0420-5 and GFS Chemicals 144 or equivalents. Dissolve a ratio of 12g sodium chloride and 12g hydroxylamine sulfate in reagent water and dilute to 100mL for a final concentration of 12%.

6.8 Potassium permanganate, mercury-free (5% solution w/v): JT Baker 3227-01 or equivalent. Dissolve a ratio of 5g potassium permanganate in 100mL of reagent water for a final concentration of 5%.

6.9 Potassium persulfate (5% solution w/v): JT Baker 3238-01 or equivalent. Dissolve a ratio of 5g potassium persulfate in 100mL of reagent water for a final concentration of 5%.

6.10 Aqua regia: 3 parts concentrated HCl to 1 part concentrated HNO_3. *Made immediately before using.*

6.11 Mercury stock solution; 1000ug/mL: Purchased from two separate sources.

6.12 Intermediate mercury standards, 10ug/mL: **Two** standards are made, one using the primary source mercury stock solution, and the second using the secondary source mercury stock solution. Use 10% HNO_3 to dilute 1mL mercury stock solution to a final volume of 100mL.

6.13 Spiking solutions (working standards):

6.13.1 Spiking Solution A (100ug/L Hg): This solution is *made fresh daily* by diluting the first source 10ug/mL intermediate mercury standard 1:100 with DI water.

6.13.2 Spiking Solution B (100ug/L Hg): This solution is *made fresh daily* by diluting the second source 10ug/mL intermediate mercury standard 1:100 with DI water.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Samples should be collected according to an approved sampling plan.

7.2 Use sample containers obtained from the vendor pre-washed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

7.3 Aqueous samples must be acidified to a pH<2 with HNO_3. Typically, a 1L sample is collected. Analysis of the sample cannot begin until it has been acidified for at least 24hrs. The maximum holding time for an aqueous sample is 28 days from collection.

7.4 Non-aqueous samples are not chemically preserved, but are required to be refrigerated at $4\pm2^{o}C$ until analyzed. Typically, 200g of solid or waste is collected.

8. PROCEDURE

8.1 DIGESTION LOGBOOK

8.1.1 Two types of digestion logbooks are used, one for liquid samples and another for soil or solid samples. The most recent iteration of the mercury benchsheet (Form 808) is to be used. At the time of digestion, the analyst should complete the header and footer information prompted in the digestion logbook (e.g., digestion date, method, digestion analyst, etc.)

8.1.2 Standard/Verification information -- The information needed to prepare the standard curve is pre-printed in the digestion logbook. The calibration curve consists of at least 5 points plus a blank point.

Also pre-printed in the digestion/analysis logbook are the ICV (initial calibration verification), ICB (initial calibration blank), CCV (continuing calibration verification), CCB (continuing calibration blank), and the CRA (low level check standard). Note that a calibration verification and blank must bracket every ten analytical samples (i.e., any sample except a CCV or CCB), and that a CCV and CCB must also be run to closeout the analytical sequence.

IPC -- For method 245.1, an initial performance check (IPC) solution is analyzed immediately following calibration. The IPC solution should be prepared from the same source used for the calibration standards. The IPC concentration should be near the midpoint of the calibration range. The percent recovery of the IPC must be 95-105% before sample analysis can begin.

8.1.3 Sample/Digestate information -- A 'Preparation Batch' is created in LIMS that lists the specific client samples to be digested, and well as the associated QC samples (Blanks, Blank Spikes, Laboratory Control Samples, Duplicates, Matrix Spikes and Matrix Spike Duplicates) that are digested for analysis. A Serial Dilution QC sample is also analyzed per batch, but this sample is not digested.

8.1.4 Additional documentation -- Reagent lots, support equipment ID, etc. are also recorded in the digestion/analysis logbook.

8.2 SAMPLE PREPARATION AND DIGESTION (Soils, Solids, and Wastes)

8.2.1 Label 100mL disposable polypropylene beakers with solution IDs as prepared in the digestion logbook.

8.2.2 Mix the sample by stirring with a disposable wooden spatula in order to obtain a representative aliquot. The aliquot should have a similar particle size distribution as the bulk sample. For very heterogeneous or unusual matrices, consult the Department Manager and Project Manager. If special processing prior to taking an aliquot was required, document in the comments section of the benchsheet.

8.2.3 For SW7471A digestions, weigh approximately a 0.60g aliquot of sample into the appropriately labeled beaker. . Record the aliquot size on the digestion benchsheet to the nearest 0.01g.

8.2.4 For soil/solid digestions, the method blank is prepared by digesting approximately 0.60g of Teflon™ chips for method SW7471A, Record the weight to the nearest 0.01g on the laboratory benchsheet.

8.2.5 For SW7471A digestions, the Laboratory Control Sample (LCS) is approximately 0.60g of Teflon™ boiling chips (record on benchsheet to nearest 0.01g) with a known amount of spike added.

8.2.6 Spike the standards, check standards and matrix spike samples as described in the digestion logbook (Spike Solution and Volume).

8.2.7 Serial Dilution Sample: A 1:5 serial dilution of an additional sample digestate is prepared per 20 field samples of like matrix that are processed as a unit. If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within ±10 % of the undiluted results. Sample analyte results failing this test should be flagged indicating the existence of matrix interferences.

8.2.8 For SW7471A digestions, add 5.0mL of Aqua regia to each beaker. Place beakers in the hot block for 2 minutes. Remove the beakers and cool to room temperature.

8.2.9 Add 50.0mL of DI water.

8.2.10 For SW7471A digestions, add 15mL of 5% potassium permanganate solution.

8.2.11 Loosely cover the beakers with polypropylene lids and mix well. Return the beakers to the water bath for 30 minutes. Remove the beakers and allow to cool.

8.2.12 Add 6mL of 12% sodium chloride hydroxylamine sulfate solution. Mix well until the color from the potassium permanganate has disappeared. Bring all standards and samples to 100mL with DI water. The samples are now ready for analysis.

8.3 SAMPLE PREPARATION AND DIGESTION (Aqueous Samples)

8.3.1 Label 50mL/15mL tubes with solution ID's as noted in the prepared digestion logbook.

8.3.2 For each sample in the batch use a test strip to verify that the solution pH is <2. Record the result of the pH test on the benchsheet (Form 824). **If the pH of a sample is found to be >2, concentrated nitric acid (trace metals grade) must be added and the sample held *in its original container* for a minimum of *24 hours* until verified to be pH<2.**

8.3.3 Transfer a well mixed 20g/10g aliquot (or smaller aliquot diluted to 20mL/10mL with DI water) into the appropriately labeled tube. TCLP leachates are usually digested at a ten-fold dilution (2.0mL/1.0mL of leachate and 18.0mL/9.0mL of DI water). Transfer the required amount of

DI water as described in the digestion logbook, into the appropriately labeled tubes for standards and quality control (QC) samples.

8.3.4 For aqueous digestions, the method blank (MB) is prepared by digesting an aliquot of reagent water.

8.3.5 A 1:5 serial dilution of an additional sample digestate is prepared per 20 field samples of like matrix that are processed as a unit. If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within +- 10% of the undiluted results. Sample analyte results failing this test should be flagged indication the existence of matrix interferences.

8.3.6 Spike the standards, check standards and matrix spike sample as described in the digestion logbook (Spike Solution and Volume).

8.3.7 Add 1.0mL/0.5mL of concentrated sulfuric acid to each tube.

8.3.8 Add 0.5mL/0.25mL of concentrated nitric acid to each tube.

8.3.9 Add 3.0mL/1.5mL of 5% potassium permanganate solution to each tube.

8.3.10 Add 1.6mL/0.8mL of 5% potassium persulfate solution to each tube.

8.3.11 Cap the tubes tightly and mix well making sure the purple color persists for at least 15 minutes. If necessary, add equal amounts of permanganate solution to all tubes in the batch until color persists for at least 15 minutes, or re-aliquot the sample at a dilution, or re-aliquot the sample and put in a separate batch.

8.3.12 Heat the tubes in a pre-heated (95+-2 C) Hot-Block for 2 hours. Remove the tubes from Hot-Block and allow to cool.

8.3.13 Add 1.2mL/0.6mL of 12% Sodium chloride hydroxylamine sulfate solution to each tube. Mix well until the color from the potassium permanganate has disappeared. The samples are now ready for analysis and should be analyzed within 24 hours of the addition of the hydroxylamine sulfate.

8.3.1 .

8.4 MERCURY ANALYSIS (using CETAC M-6000A or CETAC 7500 automated mercury analyzer)

8.4.1 Complete the digestion logbook by filling in the analytical filename, date analyzed and analyst information.

8.4.2 Preparing the System

- Turn on the mercury lamp and auto sampler and allow to warm up for at least 10 minutes (if in stand by mode). The mercury lamp is equipped with an oven to keep it at a constant temperature of 70^{o}C. If the system has been completely shut down, the instrument requires a warm up time of 90 minutes.
- Open the mercury software by clicking on the icon and open the appropriate worksheet.
- Stannous chloride is added using a peristaltic pump that delivers a constant flow of a 10% stannous chloride solution to the sample delivery tubing through a "T" connector. Clamp the pump tubing and turn the pump on. The pump line should be in 10% stannous chloride solution and the rinse tank should be filled with 1% hydrochloric acid and 1% nitric acid solution. Allow flow through all lines for about fifteen minutes before running the analytical sequence.
- Next, lower the autosampler tip. For the M6000 instrument, This is done by going to the "Instrument" option, and then going to "M6000 Controls". Then pick the "Autosampler" tab, and click the "Park" button. For the CETAC 7500 instrument, go to the "Autosampler" tab, then click "down".
- Turn the gas on at the main tank.
- Change the peristaltic pump tubing and Nafion drying cartridge as needed.

8.4.3 Calibrating the CETAC 7500 System

- Make sure that the six standards (five concentrations and a blank) have been loaded into the standards rack.
- A calibration is pre-programmed into the template. Click on the "analyze single sample" button, select high standard tube, then click "OK" (the autosampler will go to std. 10 and give a signal and peak reading. This is to make sure the system is operating correctly). After you have checked the instrument, run the calibration curve.
- Calibration results are automatically stored, and displayed at the bottom right of the screen.

- Determination of acceptable calibration curve -- An r^2 value greater than 0.995 is acceptable. If the curve is acceptable, begin running samples.
- If the curve is not acceptable, click on the stop button. Try running the calibration sequence again and check to be certain the solutions have been mixed well. At times the instrument requires a second calibration sequence to allow for warm up time. The analytical batch must be re-digested if an acceptable calibration curve cannot be obtained.

8.4.4 Running Samples

- After the field and QC samples are loaded into their designated tubes and the racks are placed on the autosampler, the instrument can begin running samples. Note that the instrument usually starts to read samples immediately after calibration, unless the analyst stops it due to a failed calibration curve.
- ICV -- Analyze an initial calibration verification (ICV) after an acceptable calibration. This is a second source check standard. Analyze an ICB immediately following. The ICV concentration must be different from the CCV. Either the ICV or CCV must be at a concentration below the midpoint of the calibration curve. For SW-846 methods and EPA 245.1, the ICV recovery must be within 90-110%. Cause of failure must be determined and corrected and the instrument recalibrated.
- CRA -- Analyze this reporting limit standard after the ICB. The results of this standard analysis are not formally assessed, unless otherwise directed in the applicable LIMS program specification. The purpose of this standard analysis is simply to verify the analyte reporting limit.
- CCV -- Analyze a continuing calibration verification (CCV) after every ten analytical samples. An analytical sample is every solution analyzed on the instrument except ICV/CCV or ICB/CCB. A CCV is also analyzed after the last analytical sample. Percent recovery of the CCV must be 80-120% of the true value (SW-846 methods and CLP methods). If samples are being analyzed by method 245.1, percent recovery of the CCV must by 90-110%. Cause of failure must be determined and corrected. Samples following the last acceptable CCV must be reanalyzed. All samples must be bracketed by satisfactory CCVs.
- The autosampler will follow the run log you entered under the "Labels" tab, this will be the order in which the samples are

analyzed. The instrument will store the data in the folder (the data are stored by date and output sequence).

8.4.5 Shutdown Procedures

- After the sample run is complete, pull up the autosampler tip and change the pump tubing from stannous chloride over to 10% nitric acid, and then deionized water. Let both run for approximately 5 minutes.
- Shut the mercury lamp off.
- When the pump has run DI water for 5 minutes, remove all lines so that air is pumped through the system for 5 minutes. Turn pump switch off and unclamp tubing.
- Turn the gas off at the main tank.

9. QUALITY CONTROL

9.1 DEFINITION OF BATCH

An analysis batch is defined as a group of twenty (20) or less field samples of like matrix that is associated with one unique set of batch QC samples and processed as a unit. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike and duplicate (MS/MSD), and serial dilution sample. All QC samples must be carried through all stages of the sample preparation and measurement steps.

9.2 BLANKS

Method blanks (MBs) are run to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Each time a batch of samples is analyzed or there is a change in reagents, a method blank must be processed. The blank concentration found must be less than the analyte reporting limit, or as otherwise specified in the applicable LIMS program specification.

SPLP, CalWet and TCLP leachates have associated blanks that are carried through the tumbling process with designated samples. These blanks are analyzed as though they are samples. Calculated results for these blanks must be less than the reporting limit, or as otherwise specified in the applicable LIMS program specification.

ICB -- Analyze an initial calibration blank (ICB) after the ICV. The calculated result of the ICB must be less than the reporting limit, or as otherwise specified in the applicable LIMS program specification. CCB -- Analyze a continuing calibration blank after every CCV. The calculated result of the CCB must be less than the reporting limit, or as otherwise specified in the applicable LIMS program specification.

9.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the

method. One laboratory control sample (LCS) is analyzed with each batch of 20 or fewer field samples. Percent recovery of the LCS must be 80-120% unless solid LCS is used in which case a range will be given by the vendor. If samples are being analyzed by EPA 245.1, percent recovery of the LCS must be 85-115%. Samples associated with a failed LCS must be re-digested and reanalyzed. Other limits may apply, consult applicable LIMS program specification.

Blank spikes (LCSs) are prepared for SPLP, Cal Wet and TCLP leachate samples using the blanks that were carried through the tumbling process. Two different fluids (determined by pH of the sample) may be used for TCLP extraction. These fluids are termed Fluid #1 and Fluid #2. One TCLP blank spike is analyzed for each fluid type present per batch of 20 samples or less.

9.4 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE

Matrix spikes (MS/MSDs) consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection. One MS/MSD are analyzed with each batch of 20 or fewer field samples (SW846). One MS/MSD is analyzed with each batch of 10 or fewer field samples for EPA method 245.1. For SPLP, Cal Wet and TCLP leachates, an MS/MSD set are run for each leachate/fluid fluid type present per batch of 20 samples or less.

Analyte recovery for matrix spikes is calculated as shown below:

$$\text{MS \% Recovery} = \frac{(C_{found} - C_{native})}{C_{added}} \times 100$$

where:

C_{found}	=	analyte concentration found in the spiked sample
C_{native}	=	native analyte concentration found in the unspiked sample
C_{added}	=	spike added analyte concentration

The MSD is analyzed as a measure of the precision of the analytical results generated as is expressed as Relative Percent Difference (RPD):

$$RPD = \frac{| Result_x - Result_{Dup} |}{(Result_x + Result_{Dup}) / 2} \times 100$$

For SW-846 methods, the recovery for the matrix spiked analytes should be within $\pm$20% of the expected value For EPA 245.1, the recovery of the matrix spiked analytes should be within $\pm$30% of the expected value. Other limits may apply, consult applicable LIMS program specification. Failures will be flagged in

reports.

The RPD between the matrix spike and the matrix spike duplicate (all methods) should be <20, unless otherwise specified in the applicable LIMS program specification. Failures will be flagged in reports.

9.5 SAMPLE DUPLICATE

One sample duplicate (Duplicate) is analyzed with each batch of 20 or fewer field samples (SW846). One sample duplicate is analyzed with each batch of 10 or fewer field samples (EPA 245.1). The RPD between the sample and duplicate (all methods) should be <20.

9.6 SERIAL DILUTION

To assist in the assessment of possible matrix interferences, a 1:5 serial dilution of an additional sample digestate is prepared per 20 field samples of like matrix that are processed as a unit. If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within ±10 % of the undiluted results, or as otherwise specified in the applicable LIMS program specification. Sample analyte results failing this test should be flagged indicating the existence of matrix interferences.

9.7 A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven replicate analyses at a concentration level near to the capabilities of the method and below the analyte reporting limit (RL). The MDL study shall be performed as needed (i.e., whenever there is a significant change in operator, background, or instrument response) and at a minimum, every year.

10. DEVIATIONS FROM METHODS

This SOP meets the requirements of methods SW846 7470A/7471A, EPA 245.1 The laboratory performs one known deviation from method SW7471A as follows: instead of weighing out three 0.20g aliquots of soil for triplicate analysis, the laboratory weighs out approximately 0.60g for a single analysis to ensure a representative sample.

11. SAFETY, HAZARDS, AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Mgt Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical

Methods, 3rd Edition, Volume 1A, "Method 7470A" and "Method 7471A", Revision 1, September 1994.

12.2 USEPA/600/4-91/010, Methods for the Determination of Metals in Environmental Samples, June 1991, "Method 245.1", Revision 2.3, April 1991.

Analytical Method: SW 7470A/7471A; EPA 245.1	**Parameter:** Mercury		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point (plus blank)	Daily at on-set of analyses or when corrective action for CCV failure does not resolve calibration verification non-compliance	Correlation coefficient (r^2) for linear regression must be $\geq$ 0.995	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze calibration standards. If quality control acceptance criterion still not met, analyses cannot proceed; a new suite of calibration standards must be prepared and analyzed. Analyses cannot proceed until an acceptable initial calibration curve is generated.
Initial Performance Check (IPC) Standard; first source (run for Method 245.1 only)	Immediately following initial calibration	Results must agree within $\pm$5% of expected value	If QC criterion not met, analyze again. If IPC still fails, IPC and initial calibration standards must be re-digested and reanalyzed.
Independent Calibration Verification (ICV); second source; run at a concentration at or below the midpoint of the calibration curve	Once after each initial calibration	Response must agree within $\pm$10% of initial calibration for SW7470A/7471A and EPA 245.1	If QC criterion not met, analyze again. If ICV still fails, ICV and initial calibration standards must be redigested and reanalyzed.
Blanks: Preparation (Method), Initial and Continuing Calibration Blank (ICB and CCB)	ICB run following the ICV One method blank per matrix type processed and analyzed per batch of twenty or less environmental samples processed. CCB run following the CCV to bracket a set of 10 analyses and to close a run sequence	Blank value must be less than reporting limit (RL), or as otherwise specified in LIMS program specification	If QC criterion not met for ICB, locate and correct problem; repeat initial calibration. If QC criterion not met for method blank, the method blank and all associated samples must be redigested and reanalyzed. If QC criterion not met for CCB, locate and correct the problem; all samples analyzed since last acceptable CCB must be reanalyzed.
CRA Standard -- Low-Level Reporting Limit Standard	Run immediately following the ICB	No acceptance criteria applicable	No corrective actions required.

Analytical Method: SW 7470A/7471A; EPA 245.1	**Parameter:** Mercury		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV); may be first or second source; run at a concentration at or below the midpoint of the calibration curve	Run to bracket a set of 10 analyses, and to end any run sequence (must be followed by a CCB analysis)	Response must agree within ±20% of expected value (SW7470A/7471A); response must agree within ±10% (EPA 245.1).	Check for calculation errors. If no calculation errors are found, analyze again. If CCV still fails, evaluate/correct instrument malfunctions; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after last acceptable CCV must be reanalyzed.
Laboratory Control Sample (LCS)	One prepared and analyzed per matrix type per batch of 20 or less field samples	Recovery must be within ±15% of expected value (EPA 245.1). For SW7470A recovery for aqueous LCS must agree within ±20% of expected value. For SW7471A, the recovery for the solid matrix LCS must agree within ±20% of expected value. Other client-specified criteria may apply, consult applicable LIMS program specification.	Check for documentable errors (e.g., calculations and spike preparation) If no computation errors are found, all associated field and quality control samples must be redigested and analyzed.
Matrix Spike (MS)	One prepared and analyzed per matrix type per batch of 20 or less field samples for SW7470A/ 7471A One prepared and analyzed per matrix type per batch of 10 or less field samples for Method 245.1	For SW7470A/7471A, recovery should agree within ±20% of expected value. For EPA 245.1, recovery must agree within ±30% of expected value. Other client-specified criteria may apply, consult applicable LIMS program specification.	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative and flag results appropriately.
Matrix Spike Duplicate (MSD)	One prepared and analyzed per matrix type per batch of 20 or less field samples for SW7470A/ 7471A . One prepared and analyzed per matrix type per batch of 10 or less field samples for EPA 245.1	(See MS recovery criteria above). RPD should be < = 20. Other client-specified criteria may apply, consult applicable LIMS program specification.	Check for documentable errors (e.g., calculations and spike preparation) If no errors are found, then sample matrix effects are the most likely cause. Note in narrative and flag results appropriately.
Laboratory Duplicate	One prepared and analyzed per matrix type per batch of 20 or less field samples for SW7470A/ 7471A	RPD should be less than or equal to 20. Other client-specified criteria may apply, consult applicable	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then

Analytical Method: SW 7470A/7471A; EPA 245.1	**Parameter:** Mercury		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
	One prepared and analyzed per matrix type per batch of 10 or less field samples for EPA 245.1	LIMS program specification.	sample heterogeneity is the most likely cause. Note in narrative and flag results appropriately.
Serial Dilution Test (1:5 dilution), analyzed to assist in the assessment of possible matrix interferences	One prepared and analyzed per matrix type per batch of 20 or less field samples	If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within ±10 % of the undiluted results, or as otherwise specified in the applicable LIMS program specification	Sample analyte results failing this test should be flagged indicating the existence of matrix interferences.
Method Detection Limit (MDL) Study; run at an analyte concentration lower than the reporting limit (RL)	As needed; at minimum annually	Must yield a positive result < than the analyte reporting limit (RL).	Determine the reason for failure and fix problem with the system. Repeat the MDL study. If criteria still not met, discuss with QA Manager (RL may be adjusted if required).

APPENDIX A
EXAMPLE
MERCURY DIGESTION - WATER/TCLP

Method _____ SOP 812/Rev __ Date Analyzed _____ File _____ *** Init. ___ (prep.) ___ (analysis)

Digestion Date _____ Spike Witness N/A Time Start _____ Time Finish _____ Bath Temp ___°C

Tube #	Solution ID	Spike * Solution	Spike Volume (mL)	Final ** Volume (mL)	Comments
STD 1	0 ppb	-	-	20.0	
2	0.2 ppb	A	0.04	20.0	
3	0.5 ppb	A	0.1	20.0	
4	1.0 ppb	A	0.2	20.0	
5	2.0 ppb	A	0.4	20.0	
6	5.0 ppb	A	1.0	20.0	
7	10.0 ppb	A	2.0	20.0	
	ICV	B	0.2	20.0	
	ICB	-	-	20.0	
	CRA-0.2 ppb	A	0.04	20.0	
	IPC (245.1 only)	A	0.4	20.0	
	SAMPLES -- **Prep. Batch ID(s)** _____				(see LIMs Prep. Batch report for sample info. (IDs, Aliquots, etc.)
	CCVs	A	0.4	20.0	___ # prepared
	CCBs	-	-	20.0	___ # prepared

*** See run report for run log information.

** Laboratory DI water used to make-up to final volume.

*A: 100 ppb Hg solution made from 100x dilution (1 mL/100 mL) of _____ ID

*B: 100 ppb Hg solution made from 100x dilution (1 mL/100 mL) of _____ ID (2nd source)

See run header for maintenance performed.

Digestion Cups: _____

Reagents: H_2SO_4 _____ HNO_3 _____ $KMnO_4$- _____ $K_2S_2O_8$- _____

SnCl- _____ Hydroxylamine _____

Balance(s) Used: _____

Pipet(s) Used: _____

Note: Each page is copied as completed and included with the workorder/run documentation; reviewed subsequently.

APPENDIX A
EXAMPLE
MERCURY DIGESTION - SOIL

Method _____ SOP 812/Rev __ Date Analyzed _____ File _____ *** Init. ___ (prep.) ___ (analysis)

Digestion Date _____ Spike Witness N/A Time Start _____ Time Finish _____ Bath Temp ___°C

Tube #	Solution ID	Spike * Solution	Spike Volume (mL)	Sample **** Aliquot (g)	Final ** Volume (mL)	Comments
STD 1	0 ppb	-	-	-	100.0	
2	0.2 ppb	A	0.2	-	100.0	
3	0.5 ppb	A	0.5	-	100.0	
4	1.0 ppb	A	1.0	-	100.0	
5	2.0 ppb	A	2.0	-	100.0	
6	5.0 ppb	A	5.0	-	100.0	
7	10.0 ppb	A	10.0	-	100.0	
	ICV	B	1.0	-	100.0	
	ICB	-	-	-	100.0	
	CRA-0.2 ppb	A	0.2	-	100.0	
	SAMPLES -- **Prep. Batch ID(s)** _____					(see LIMs Prep. Batch report for sample info. (IDs, Aliquots, etc.)
	CCVs	A	2.0	-	100.0	___ # prepared
	CCBs	-	-	-	100.0	___ # prepared

**** Automated balance entry into LIMS.

*** See run report for run log information.

** Laboratory DI water used to make-up to final volume.

*A: 100 ppb Hg solution made from 100x dilution (1 mL/100 mL) of _____ ID

*B: 100 ppb Hg solution made from 100x dilution (1 mL/100 mL) of _____ ID (2nd source)

See run header for maintenance performed.

Digestion Cups: _____

Reagents: HNO_3 _____ HCl _____ SnCl- _____ $KMnO_4$- _____ Hydroxylamine _____

Balance(s) Used: _____

Pipet(s) Used: _____

Note: Each page is copied as completed and included with the workorder/run documentation; reviewed subsequently

Form 808r20(b).doc (8/29/2011)

APPENDIX B

Documentation of Acidification and/or Filtration of Water Samples in Metals Department

Sample ID	Initial Sample pH	Initial Sample Volume (mL)	Filtered through 0.45 µm ? (Y / N)	Volume HNO_3 Added (mL)	Lot # for Conc. HNO_3	Date HNO_3 Added	Time HNO_3 Added	Initials	Sample pH after 24 hrs	Initials	Comments
				Use current iteration of benchsheet.							

Reviewed by / date _____

Form 824r1.doc (8/1/07)

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATIN OF ELEMENTS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY
REFERENCED METHOD:	_EPA 200.8 AND SW6020A
SOP ID:	827
REV. NUMBER:	10
EFFECTIVE DATE:	_09/11/2015

ALS
STANDARD OPERATING PROCEDURE 827 REVISION 10

TITLE: DETERMINATION OF ELEMENTS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY -- METHODS EPA 200.8 AND SW6020 and 6020A

FORMS: NONE

APPROVED BY:

PRIMARY AUTHOR _____ DATE 9/9/2015

QUALITY ASSURANCE MANAGER _____ DATE 9/24/15

LABORATORY MANAGER _____ DATE 9/9/2015

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- EPA 200.8 and SW6020 -- are used to determine the concentration of total or dissolved elements in prepared liquid and solid samples. This SOP is applicable to a large number of elements in waters and wastes after appropriate sample preparation steps are taken. Sensitivities and linear ranges for these elements will vary with the matrices and operating conditions. Reporting limits in relatively simple matrices will generally be below 0.1µg/L. Less sensitive elements (such as As and Se) and desensitised major elements may have reporting limits of 1.0µg/L or higher.

2. SUMMARY

This SOP describes the multi-element determination of trace elements by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS). Sample material in solution is introduced by pneumatic nebulization into a radiofrequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface into an octopole-based radiofrequency lens (collision cell). Reaction gases such as helium and hydrogen are introduced into the collision cell to facilitate ion beam focusing and minimization of molecular ion interferences. The ions are then introduced into a mass spectrometer where they are sorted according to their mass-to-charge ratios (m/z) and quantified with a detector. Interferences relating to the technique must be recognized and corrected. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift as well as suppressions or enhancements of instrument response caused by sample matrix must be corrected for by the use of internal standards.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 ALS uses custom Program Specifications which are directives and controls programmed into LIMS that govern the acquisition and reporting of project data. It is the responsibility of all personnel who work with samples or data involving this method to consult the applicable LIMS Program Specification prior to initiating handling of samples or data.

3.3 Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing these methods. This demonstration may come in the form of supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.4 Final review and sign-off of the data are performed by the Department Supervisor or designee. Initialing and dating the file indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Isobaric elemental interferences occur when isotopes of different elements form singly or doubly charged ions of the same nominal mass-to-charge ratio, and which cannot be resolved by the mass spectrometer (e.g., Mo98 and Ru98). A data system must be used to evaluate and correct for these interferences when they are present. Corrections for isobaric interferences may be made by measuring the intensity (response) due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

4.2 Abundance sensitivity is a property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interference may occur when a small ion peak is being measured adjacent to a large one. The potential for these interferences should be recognized and the spectrometer resolution adjusted to minimize them.

4.3 Isobaric molecular interferences are caused by ions consisting of more than one atom or charge which have the same nominal mass-to-charge ratio as the isotope of interest, and which cannot be resolved by the mass spectrometer (e.g., ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.). These ions are commonly formed in the plasma or interface system from support gases or sample components. Predictions about the type of molecular interferences may be made using knowledge about the sample matrix. Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of Ar_40Cl_{35} on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 (Ar_40Cl_{37}) and converting to the apparent intensity of ArCl at mass 75 by using the isotope ratio of Cl_{37} to Cl_{35}. Corrections for these interferences may be made based on the natural isotope ratios of the molecular ion (as described above) or by measuring the interference that occurs when the interferent is present.

4.4 Physical interferences are associated with the physical processes that govern the transport of the sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass spectrometer interface. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension), or during the excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute to deposits of material on the extraction and/or skimmer cones. Deposits can reduce the effective diameter of the orifices and therefore ion transmission. Dissolved solid levels not exceeding 0.2% (w/v) have been recommended to reduce such effects. Internal standardization may be effectively used to compensate for many physical interference effects. Internal standards ideally should have similar analytical behavior to the elements being determined.

4.5 Memory interferences result when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects, or carryover, can result from sample deposition on the sampler and skimmer cones, as well as from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them.

5. EQUIPMENT AND SUPPLIES

5.1 Agilent 7700x inductively coupled plasma mass spectrometer (icp-ms) with agilent mass hunter software version b.01.01or equivalent. System must be capable of providing resolution, less than or equal to 0.75amu at 5% peak height from 6-253amu and must be equipped with a data system that allows corrections for isobaric

interferences and the application of the internal standard technique. Refer to qam appendix e1 for complete listing of instruments and software**.**

5.2 Autosampler, Cetac ASX-510 or equivalent

5.3 Radiofrequency generator compliant with FCC regulations

5.4 A variable speed peristaltic pump is required for solution delivery to the nebulizer

5.5 A mass flow controller on the nebulizer gas supply is required

5.6 Volumetric flasks of suitable precision and accuracy

5.7 Volumetric pipets of suitable precision and accuracy

6. REAGENTS

6.1 Hydrochloric Acid (HCl), concentrated, trace metals grade

6.2 Nitric Acid (HNO_3), concentrated, trace metals grade

6.3 Reagent water

6.4 Liquid Argon, 99.999% pure or better

6.5 Helium, high purity, 99.999% or better

6.6 Hydrogen, high purity, 99.999% or better

6.7 STANDARDS

NOTE: All standard solutions are prepared, documented, and stored in accordance with SOP 300. All standard solutions are to be contained in fresh (previously unused) polypropylene bottles.

6.7.1 Individual Elemental Standards, 10,000µg/ML,5,000µg/mL or 1,000µg/mL: First source, purchased as certified solutions of 99.99% purity or greater. Element stocks should be checked for the presence of impurities that might influence the accuracy of the standard. Records of vendor supplied certificates of analysis must be maintained. Shelf life = Manufacturer's date of expiration; replaced sooner if degradation occurs.

6.7.2 Intermediate Standard Solutions (single- or multi-element): Made by the dilution of vendor purchased individual elemental standards. Care must be taken in the preparation of multi-element standards that the elements are compatible and stable. Freshly prepared solutions should be transferred to acid cleaned bottles for storage and monitored periodically for stability.

6.7.3 Calibration Standards: Prepared by diluting the individual elemental standards or the intermediate standard solutions to levels appropriate to the operating range of the instrument, using reagent water, for example, containing 1% HNO_3 and 1% HCl. Dilutions should be made with a diluent that best matches the acid strength in the samples, digestates, or their dilutions. Calibration standards should be prepared at a minimum of three concentrations, one of which should be at the reporting limit.

6.7.4 ICSA (Interference Check Standard-A): Made daily by diluting 0.1mL of ICSA stock solution (vendor purchased) up to a 10mL final volume.

6.7.5 ICSAB (Interference Check Standard-AB): Made daily by diluting 0.1mL of ICSA Stock solution (vendor purchased) and 2.0mL of the high calibration standard up to a 10mL final volume. T

6.7.6 ICV (**second source** Initial Calibration Verification Standard): Prepared by diluting 2.0mLof a second source intermediate (dilution from a second source stock) up to a 10mL final volume. The ICV working solution contains elements of interest with concentrations near the midpoint of the linear range and at a concentration other than that used for instrument calibration.

6.7.7 CCV (**first source** Continuing Calibration Verification Standard): prepared by dilution of intermediate or calibration standard solutions. The concentrations for each element found in the CCV should be at or near the mid-point of the calibration curve.

6.7.8 CRI (**first source**, low leveldetection limit check standard): Prepared by dilution of intermediate or calibration standards solutions. The concentrations for each element found in the CRI's are at the ALS detection limit .

6.7.9 LLICV/LLCCV (**first** source, low level initial/continuing calibration verification): (prepared by dilution of intermediate. The concentrations for each element found in the LLICV/LLCCV are at the standard ALS reporting limit.

6.8 Internal Standard Solutions: An internal standard intermediate containing 2ppm Ga, Ge and Pt, 1 ppm Rh and In and 0.5 ppm Bi1,000ppm individual element standards. This intermediate is added to all standards and samples in the same proportion of 1:100. Most often this dilution is performed by adding 0.05mL of the internal standard intermediate to 5mL of sample or standard. Internal standards can be introduced continuously using a peristallic pump.

6.9 Mass Spectrometer Tuning Solution: Consists of a solution containing elements representing all of the mass regions of interest. A vendor purchased stock (containing for example, 10mg/L of Be, Bi, Ce, Co, In, Mg, Ni, Pb and U) is diluted 1000X. The concentration of these elements in the working solution is 10ppb. Concentration of the tuning solution is not critical as the tuning solution is used to verify that the resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability.

6.10 BLANKS

6.10.1 Calibration blank: Consists of, for example, 1% (v/v) nitric acid and 1% (v/v) hydrochloric acid in reagent water. This blank is the diluent used in

preparing the calibration standards, and should match (as close as possible) the strength in the samples, digestates, or their dilutions.

6.10.2 Method blank: Contains all the reagents in the same volumes as used in processing the samples. The method blank must be carried through the same entire preparation scheme as the samples, including digestion.

6.10.3 Rinse blank: Consists of 1% (v/v) nitric acid and 1% (v/v) hydrochloric acid in reagent grade water.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 Samples should be collected according to an approved sampling plan.

7.2 Liquid samples are collected in plastic or glass containers and must be chemically preserved with nitric acid to a pH<2. The acidified sample must remain in its original container for a minimum of 24hrs before analyses can begin. Samples for dissolved metals analyses should be filtered in the field prior to chemical preservation. Unfiltered samples must be maintained at 4±2°C. Aqueous metals samples must be prepared and analyzed within 180 days of collection.

7.3 Solid samples are collected in plastic or glass containers. Solid samples are not chemically preserved and must be maintained at 4±2°C. Solid metals samples must be prepared and anlayzed within 180 days of collection.

8. PROCEDURE

(See SOP 337 for further calibration and calculation details)

8.1 INSTRUMENT START-UP

Verify that the cooling water for the instrument is flowing. Make sure that all gas supplies are connected and switched on. Make sure that the ventilation system is running. The instrument software will check the instrument communications, including any peripheral devices before commands can be issued. Open the instrument control page and establish a plasma following the manufacturer's instructions. An automatic start-up procedure has also been programmed into the software. Allow a period of not less than 30 minutes for the instrument to warm up.

8.2 Tune the instrument following the manufacturer's instructions. Instrument parameters are controlled from the tune page in the software. Optimize the instrument’s sensitivity, stability, oxide levels, etc., by aspirating the tuning solution and adjusting, if necessary, any parameters. Some of these parameters include: torch position in the X, Y, and Z axis, various gas flow rates, cone voltages, etc.

8.3 PRECALIBRATION ROUTINE

Follow the pre-calibration routine below before completing the calibration of the instrument:

8.3.1 Instrument stability is demonstrated by running the tuning solution five (5) times with resulting standard deviations of absolute signals for all analytes

of less than 5% (Reference 12.3). Data from this stability test is included with the daily raw data.

NOTE: To meet the requirements of Section 10.2.2 of EPA Method 200.8, the tuning solution shall be run a minimum of five times with resulting standard deviations of absolute signals for all analytes of less than 5%, when analyzing wastewater samples for State of California compliance monitoring,

8.3.2 Conduct mass calibration and resolution checks . Resolution at low mass is indicated by magnesium isotopes 24, 25, 26. Resolution at high mass is indicated by lead isotopes 206, 207, 208. Adjust mass calibration if it has shifted by more than 0.1amu from unit mass. Adjust the spectrometer resolution, if needed, to produce a peak width <0.9amu full width at 10% peak height (SW6020A). Data showing these criteria were met are included with the data package upon client request.

NOTE: To meet the requirements of Section 10.2.1 of EPA Method 200.8, the spectrometer resolution, shall be adjusted, if needed, to produce a peak width less than 0.75amu at 5% peak height, when analyzing wastewater samples for State of California compliance monitoring.

8.4 INTERNAL STANDARDIZATION

Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full range mass scans, a minimum of three internal standards shall be used. Generally, an internal standard should be no more than 50amu removed from the analyte. Internal standards shall be present in all samples, standards, and blanks at identical levels. This is achieved by directly adding an aliquot of the internal standard intermediate to each sample, standard, and blank. The concentration of the internal standard should be sufficiently high for good precision and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Internal standards should be added to samples, standards, and blanks in a similar manner, in order for dilution effects to be disregarded. .

8.5 CALIBRATION

Before initial calibration, set up proper instrument software routines for quantitative analysis. Calibrate the instrument for the analytes of interest for the selected isotopes using the calibration blank and at least three calibration standards according to the manufacturer's recommended procedures. Flush the system with the rinse blank for at least 30 seconds between each standard solution and sample. Use the average of multiple integrations for both standardization and sample analysis. All masses that could affect data quality should be monitored to determine potential effects from matrix components on the analyte peaks. These masses should be monitored either simultaneously in a separate scan or at the same time quantification occurs.

8.6 ANALYTICAL SEQUENCE

The typical autosampler analytical sequence after the initial calibration is listed below along with brief descriptions of each solution. See also Section 9 and the QC Summary Table for further details.

8.6.1 ICV: Initial calibration verification check standard (second source). Results must be within 90-110% of the expected concentration. Cause of failure must be determined and corrected and the instrument recalibrated, if needed.

8.6.2 ICB: Initial calibration blank. Analyzed immediately after the ICV. The absolute value of the blank results must be less than the analyte reporting limit, or as specified in the applicable LIMS program specification. Demonstrates that the analytical system is free from interferences and is in control.

8.6.3 CRI: Low level (detection limit) check standard. Unless otherwise specified by LIMS program specification, run for informational purposes only. ALS does not typically control on CRI recovery.

8.6.4 LLICV: Low Level Initial Calibration Verification. Check standard (first source). Results must be within 70-130% of the expected concentrations, or according to client program specifications. Cause of failure must be determined and corrected and the instrument recalibrated if needed

8.6.5 ICSA: Interference check standard A. Contains high concentrations of possible interfering elements. Analyzed at the beginning of an analytical sequence or once every 12 hours, whichever is more frequent. Analyzed to verify the magnitude of elemental and molecular-ion isobaric interferences and the accuracy of any corrections made. Results for this check standard analysis should not contain analyte concentrations (i.e., analytes susceptible to interference) above twice the analyte reporting limit (2X MDL per DOD), consult applicable LIMS program specification.

8.6.6 ICSAB: Interference check standard B. Contains all elements in the ICSA. In addition, contains low concentrations of the elements of interest. Analyzed at the beginning of an analytical sequence or once every 12 hours, whichever is more frequent. Analyzed to verify the magnitude of elemental and molecular-ion isobaric interferences and the accuracy of any corrections made. Results for this check standard analysis should be within 80-120% of the expected concentrations for the elements of interest.

8.6.7 SAMPLES: Additional analytical samples include all samples analyzed on the instrument except ICV, ICB, CCV, LLICV, LLCCV and CCB. The CRI, ICSA and ICSAB along with all samples in the sequence and

any dilutions, post-spikes, laboratory and matrix spikes, duplicates, serial dilutions and method/preparation blanks are all considered to be analytical samples.

8.6.8 CCV: Continuing calibration check standard (first source). A CCV is analyzed at a frequency of 10% or every two hours whichever is more frequent. The CCV is also analyzed after the last analytical sample. Results must be within 90-110% of the expected concentration. Cause of the failure must be determined and corrected and the instrument recalibrated, if necessary. Samples following the last acceptable CCV must be reanalyzed.

8.6.9 CCB: Continuing calibration blank. Must follow CCV analyses. The absolute value of the blank results cannot exceed the analyte reporting limit, or as specified in the applicable LIMS program specification. Demonstrates that the analytical system is free from interferences and is in control.

8.6.10 LLCCV: Low Level Continuing Calibration Verification: Check standard (first source). At a minimum, run a LLCCV after the last analytical sample (more frequently is recommended to reduce reanalysis of data). Results must be within 70-130%of the expected concentration, or according to client program specifications. Cause of failure must be determined and corrected. Samples following the last acceptable LLICV/LLCCV must be reanalysed.

The sequence continues with CCV and CCB analyzed after every 10 analytical samples and at the conclusion of the sequence.

8.7 After the analytical sequence is complete, a "header and summary" section is produced which includes:

- standard information including standard identifications, expiration dates, elements and concentrations, and preparation procedures
- acid lot numbers
- pipet identification numbers
- dilution information and preparation procedures
- analytical spike information and preparation procedures
- daily and monthly maintenance items performed
- summary page with analytical sequence and elements of interest

8.8 In addition to the electronic run information provided by the instrument's output, a hardcopy Run Log is maintained as an internal Departmental record of instrument throughput.

8.9 REGULAR MAINTENANCE ITEMS

Check the following items prior to each run to ensure the instrument is in good working order:

- argon, hydrogen and helium levels; order more as needed
- printer and paper supply
- water in recirculating coolers -- fill as necessary
- pump tubing -- replace when necessary
- empty the drain containers
- tune instrument per manufacturer's procedures
- perform ten minute stability test (include results with data package)
- check / clean torch and cones for deposits
- check / clean nebulizer and spray chamber
- check / fill vacuum pump oil

There is a section in the raw data header information where the Regular Maintenance items may be initialled as completed.

8.10 MAINTENANCE LOG

The instrument maintenance software module is used to record information concerning instrument maintenance that is not covered by the daily and monthly maintenance items described above.

9. QUALITY CONTROL

The quality control requirements for analyses by ICP-MS consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and calibration solutions as a continuing check on performance. In addition to this Section, various quality control samples and acceptance criteria are discussed in Section 8.6 and in the QC Table at the end of this SOP.

9.1 MDL/DL limits determinations are completed annually and as defined by the reference method. A MDL/DL study must also be performed as a component of method validation or whenever the basic chemistry of a procedure changes. See ALS SOP 329 for guidance on detection limits. ALS uses RVS samples run with each batch to assess the method sensitivity on an ongoing basis and to calculate detection limits as needed.

9.2 Instrument detection limits (IDLs) reflect instrument capability and are determined per ALS SOP 329.

9.3 Tune Standard: The tune standard shall be prepared in the same acid matrix as the calibration standards and analyzed at least five times consecutively. If the peak width at 10% peak height is not <0.9amu, the mass calibration is not within 0.1amu,

or the percent relative standard deviation (%RSD) of the absolute signals of the analytes exceeds 5% (discussed in Section 8.3.1), the analysis shall be terminated, the problem corrected, and the instrument re-tuned. All sample results reported must be associated with an instrument tune that meets these requirements

9.4 Method Blank (MB): One method blank is prepared per batch of 20 or less field samples. The method blank consists of an aliquot of reagent water that has been digested and prepared in the same manner as the associated samples.

For drinking water matrix samples analyzed for regulatory compliance purposes, values in the method blank cannot exceed 2.2 times the analyte MDL. If this criterion is not met, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable method blank values have been obtained.

For non-drinking water matrix sample analyses, the method blank cannot contain any analyte above the analyte reporting limit, or as specified in the applicable LIMS program specification. Method blank results are also acceptable if sample concentrations are greater than 10 times the concentration found in the method blank.

9.5 Laboratory Control Sample (LCS): One laboratory control sample is prepared per batch of 20 or less field samples. The LCS is a water sample with known analyte concentrations that is digested and prepared in the same manner as the associated samples. The control limits for LCS recovery are 80-120%, or as otherwise specified in the applicable LIMS program specification. All samples associated with a failed LCS must be redigested and reanalyzed. A low level LCS will be digested and prepared for drinking waters; it will be prepped in the same manner as the associated samples. The analyte concentrations will be near the RL. The low level LCS recovery limits are 70-130%.

9.6 Sample Duplicate: One sample duplicate must be prepared per batch of 20 or less field samples. The control limit for duplicate precision is that the relative percent difference (RPD) must be ≤20%, or as otherwise specified in the applicable LIMS program specification. RPD is calculated as shown below:

$$RPD\ (\%) = \frac{(Result_x - Result_{dup})}{(Result_x + Result_{dup}) / 2} \times 100$$

The results are flagged if the RPD exceeds 20%.

9.7 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): One MS and MSD are prepared per batch of 20 or less field samples. MS and MSD samples consist of additional aliquots of a particular field sample that are spiked, digested and prepared in the same manner as the associated samples. Matrix spike samples are evaluated in terms of recovery, calculated as follows:

$$\%R = \frac{(Conc_{Found} - Conc_{Sample})}{Conc_{Target}} \quad X \quad 100$$

where:

$Conc_{Found}$ = analyte concentration found in the MS or MSD sample

$Conc_{Sample}$ = analyte concentration found in the field sample

$Conc_{Target}$ = target (anticipated) analyte concentration based on amount spiked

Matrix spike recovery is not evaluated if the analyte concentration in the unspiked sample is greater than 4 times the spike level. The quality control limit for matrix spike recovery is 75-125%, and the control limit for MS/MSD precision is that the RPD must be $\leq$20%, unless otherwise specified in the applicable LIMS program specification. RPD for the MS/MSD is calculated the same as for the Sample Duplicate (see Section 9.6 above). Results are flagged if MS/MSD recovery or precision results are outside of control limits. A post digestion spike analytical spike (i.e., sample aliquot spiked after digestion) should be performed when matrix spike recovery is outside the control limits. If recovery of the post digestion analytical spike is not within 75-125% (or other specified limits), the result is flagged indicating that matrix interference is suspected.

9.8 Serial Dilution Sample: A 1:5 serial dilution of an additional sample digestate is prepared per 20 field samples of like matrix that are processed as a unit. If analyte concentrations are sufficiently high (at least 50X the instrument detection limit), the results of the dilution test should agree within ±10 % of the undiluted results. Other client-specified control limits may apply, consult applicable LIMS program specification. Sample analyte results failing this test should be flagged indicating the existence of matrix interferences.

9.9 Linear Range: The linear range of the instrument is determined by analysing a solution with concentrations ten times the level in the highest calibration standards. If results are within 90-110% of the known concentration, then the upper end of the analytical range (linear range) is its concentration (ten times the level in the highest calibration standard). . Samples whose concentrations exceed the linear calibration range must be diluted to bring their concentrations within the known calibration range of the instrument. The linear range is verified every six months or whenever significant instrument components have been replaced or repaired.

9.10 Internal Standard Responses: The responses from the internal standards should be monitored throughout the analytical sequence. Ratios of the internal standards responses against each other should also be monitored routinely. This information may be used to detect potential problems caused by mass dependent drift, errors incurred in adding the internal standards or increases in the concentrations of individual internal standards caused by background contributions from the sample.

For drinking water samples analyzed for regulatory compliance purposes, the absolute response of any one internal standard must not deviate more than 60-125% of the original response in the calibration blank. For non-drinking water matrix sample analyses, the absolute response of any one internal standard must not fall below 30% of the intensity of that internal standard in the initial calibration blank (ALS evaluates against the zero standard), *or as specified in the applicable Program Specification.* If deviations greater than these are observed, flush the instrument with the rinse blank and monitor the responses in the calibration blank (CCB). If the responses of the internal standards in the calibration blank (CCB) are within limits, dilution and reanalysis of the sample is required. If the response of the internal standards in the calibration blank (CCB) is outside the control limits, terminate the analysis and determine the cause of the drift. Possible causes of drift may be a partially blocked sampling cone or a change in the tuning condition of the instrument.

10. DEVIATIONS FROM METHOD

10.1 There is variability between the two methods referenced by this SOP regarding the preparation and analysis of interference check standards (ICSA and ICSAB); interference check standards are not discussed in Method EPA 200.8 at all. Hence, in this SOP, the ICSA and ICSAB standards are prepared according to the specifications of Method 6020 & A, with the exception that the solution concentration of all the constituents is ten fold less, in accordance with the sensitivity characteristics of the ICP-MS instrument used at ALS.

10.2 Section 9.4.5 of Method 200.8 stipulates control limits of 60-125% for internal standard response, Sections 4.4 and 9.3 of SW6020A discuss internal standards response in terms of suppression (below 30%) due to physical interferences. Unless otherwise directed by LIMS Program Specification, ALS observes SW6020 A guidance for evaluating internal standards performance as it has been ALS's experience that the data and performance of quality control samples are not impacted when internal standard response is enhanced (i.e., above 100%).

10.3 Section 9.3.4 of Method 200.8 provides for ±15% CCV criteria. In concurrence with SW6020A, ALS follows the more stringent CCV criteria of ±10%.

10.4 Both Methods 6020A and 200.8 calculate IDLs based on the analysis of reagent blank solution. ALS follows the procedure listed in ALS SOP 329..

10.5 SW6020A requires dilution of samples when post spike criteria is not met. ALS's policy is to flag results and narrate if post spike criteria is not met.

10.6 SW6020A requires reanalysis of samples that fail duplicate precision criteria. ALS's policy is to flag results and narrate if duplicate precision criteria is not met.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 Methods for the Determination of Metals in Environmental Samples - Supplement I, EPA-600/R-94-111, May 1994. "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry", USEPA Method 200.8, Revision 5.4, EMMC Version.

12.2 USEPA SW-846, Test Methods For Evaluating Solid Waste - Physical/Chemical Methods, "Method 6020A", Revision 1, February, 2007..

12.3 USEPA CLP SOW ILM05.2, "Method 6020 CLP-M", version 9.0.

12.4 Operator's Manual: Agilent Model 7700X, User's Guide.

Analytical Methods: EPA 200.8, SW6020A	**Parameter**: ICP-MS Elements		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Tune Standard; analyzed at least 5 times consecutively	Daily before the initial calibration	Peak width <0.9amu at 10% peak height, mass calibration within 0.1amu and %RSD of replicates <5% (unless otherwise noted in Program Specification)	Correct problem and repeat tune standard routine.
Initial Calibration; uses at least 3 standards and a blank	Daily	Correlation coefficient (r^2) for all analytes >0.998	Correct problem and repeat initial calibration.
Initial Calibration. Verification (ICV); second source	Daily after initiation calibration	All analytes within ±10% of expected value	Correct problem then repeat initial calibration.
Initial Calibration Blank (ICB)	Immediately following ICV	Absolute value of result for each analyte <reporting limit (RL), or as specified in applicable LIMS program specification	Correct problem then repeat initial calibration.
Continuing Calibration Verification (CCV); conc. of analytes must be different from the ICV	After every 10 samples and at the end of the analytical sequence	All analytes within ±10% of expected value	Correct problem and reanalyze all samples since last successful CCV.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of result for each analyte <RL, or as specified in applicable LIMS program specification	Correct problem then analyze CCB and previous 10 samples
Low Level Initial Calibration Verification (LLICV)	Run immediately following CRI	All Analytes within 30% of expected value	Re-analyze failed analyte. If still outside control limit, halt analysis, correct problem and recalibrate.
CRI (reporting limit check) Standard	Run immediately following the ICV/ICB	ALS does not typically control on CRI Standard response. However, if so specified by LIMS program specification, ALS will observe the following performance criteria: recovery within 50-150% for Sb, Pb and Tl; recovery within 70-130% for all other analytes.	Reanalyze failed analytes. If still outside of control limits, halt analyses, correct problem and recalibrate. Analyses may not proceed until an acceptable CRI Standard has been analyzed.
ICSA (Interference Check Solution A) and ICSAB (Interference	At the beginning of an analytical run or every 12 hours whichever is more	ICSA should not contain non-spiked analytes at concentrations above twice	No directives are given in the referenced methods for this QC check. The limits indicated in

Analytical Methods: EPA 200.8, SW6020A	**Parameter**: ICP-MS Elements		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action**
Check Solution B)	frequent	the analyte RL, or as otherwise specified in the applicable LIMS program specification. ISCAB: all analytes of interest should be within ±20% of expected value.	this Table are used as ALS guidelines; no corrective actions are taken on an analytical batch basis.
Method blank (MB)	One MB per batch of 20 or fewer field samples	For drinking water compliance samples, blank values must be <2.2 times the analyte MDL For non-drinking water matrix sample analyses, absolute values in MB <analyte RL, or as specified in applicable LIMS program specification	Correct problem then reprep and analyze MB and all samples processed with the contaminated blank.
Laboratory Control Sample (LCS)	One LCS per batch of 20 or fewer field samples	For drinking water compliance samples, analyte recoveries must be within ±15% of expected values. For non-drinking water matrix sample analyses, analyte recoveries must be within 80-120% of expected values for each analyte. A low level LCS will be digested and prepared for drinking waters; it will be prepped in the same manner as the associated samples. The analyte concentrations will be near the RL. The low level LCS recovery limits are 70-130%. Other client-specified criteria may apply, consult applicable LIMS program specification	Correct problem then reanalyze. If still out reprep and reanalyze the LCS and all samples in the affected batch.
Sample Duplicate (DUP)	One sample duplicate per batch of 20 or fewer field samples	For each analyte RPD ≤20%, or as otherwise specified in applicable LIMS program specification	Flag results if RPD > 20%.
Low Level Continuing Calibration Verification (LLCCV)	Run at MINIUMUM following the final field sample/ CRI recommended more often	All Analuytes witin 30% of expected values (unless program specifications require otherwise.	Correct problem and re-analyze all samples since last successful LLICV/LLCCV

Analytical Methods: EPA 200.8, SW6020A	**Parameter**: ICP-MS Elements		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	**Frequency**	**Acceptance Criteria**	**Corrective Action**
	to reduce number of samples requiring re-analysis		
Matrix Spike/Matrix Spike Duplicate MS/MSD (MS/MSD)	One MS/MSD pair per batch of 20 or fewer field samples	Recovery limit 75-125% for each analyte, not calculated if analyte conc > 4X the spike level. For each analyte RPD ≤20% Other client-specified criteria may apply, consult applicable LIMS program specification	Flag results if MS/MSD recovery or precision results are outside control limits, perform post digestion analytical spike.
Post digestion analytical spike	Performed when MS/MSD recovery is outside ± 25% (unless analyte conc > 4X the spike level)	Recovery limit 75-125% for each analyte, or as specified in applicable LIMS program specification	Flag results if post spike recovery or precision results are outside control limits.
Internal standard responses	Monitored throughout the analytical run	For drinking water compliance samples, response of internal standard in sample must be within 60-125% of response in initial calibration blank For non-drinking water matrix sample analyses, response of internal standard for samples must be >70% of the original response in the calibration blank, or as specified in the applicable LIMS program specification (note that per DOE client specification, IS response not to exceed 155%)	Dilute samples until internal standard response is within criteria.
Serial Dilution	Performed on one sample per batch of 20 or fewer field samples	Results should agree within ±10% of undiluted results if analyte concentrations are sufficiently high (at least 4X the RL)	Flag results if outside criteria.
RVS (Method Detection Limit (MDL) Study); run per method requirements and at an analyte concentration near the minimum detection capabilities of the method	As needed and, at minimum, annually	Positive result < analyte RL	Determine the reason for failure and correct problem with system; then repeat study for analytes that did not meet criteria. MDL determination follows procedures specified in ALS SOP 329.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF TOTAL AND AMENDABLE CYANIDE (DISTILLATION)
REFERENCED METHOD:	_SW9010C, SW9013, EPA 335.1, EPA 335.2, CLP INORGANIC SOW (ILM04.0); DETERMINTION OF WEAK AND DISSOCIABLE CYANIDE – SM4500-CN I_
SOP ID:	1110
REV. NUMBER:	15
EFFECTIVE DATE:	10/11/2013

ALS
STANDARD OPERATING PROCEDURE 1110 REVISION 15

TITLE: DETERMINATION OF TOTAL AND AMENABLE CYANIDE (DISTILLATION) -- METHODS SW9010C, SW9013, SW9014, EPA 335.1, EPA 335.2 AND CLP INORGANIC SOW (ILMO4.0)

DETERMINATION OF WEAK AND DISSOCIABLE CYANIDE – METHOD SM4500-CN I

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY DIRECTOR _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- SW9010C, SW9013, SW9014; EPA 335.1, EPA 335.2; the CLP Inorganic SOW ILMO4.0; and SM4500-CN I -- describe procedures to extract and quantify cyanide in waters, soils, and various wastes and waste leachates. Cyanide that is present either as soluble salts or in complexed forms is detected.

Total cyanide, total cyanide extractable from solids and oils, cyanide amenable to chlorination, and/or weak and dissociable cyanide can be determined by the procedures outlined in this SOP. These procedures are written to address trace analyses (cyanide concentrations <1000ppm), but may also be used to determine minor (1000-10,000ppm) and major (>10,000ppm) concentrations of cyanide by adapting the sample preparation techniques (e.g., employment of sample dilution) or determinative techniques (e.g., use of the titration procedure provided for in Methods EPA 335.2, SW9010C and SW9014, in lieu of colorimetric development, where cyanide concentrations are significant).

Procedures presented in the CLP Inorganic SOW ILMO4.0 are based on total cyanide Method EPA 335.2. The CLP Inorganic SOW also provides for the direct analysis (i.e., without extraction) of solid matrix samples. The extraction procedure for total cyanides in solid or oil matrices contained in this SOP references Method SW9013. The reflux-distillation preparation for liquid samples or extracts described by this SOP references Methods SW9010C and EPA 335.2. The spectrophotometric determination of cyanide described herein references Methods SW9010C; SW9014 and EPA 335.2.

Procedures for the determination of cyanide amenable to chlorination contained in this SOP reference Methods SW9010C and EPA 335.1. The procedures for the determination of weak and dissociable cyanide described by this SOP reference Method SM4500-CN I.

2. SUMMARY

Cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation process conducted under acidic conditions. The released cyanide is absorbed in a scrubber containing sodium hydroxide (NaOH) solution. It is critical that the amount of sodium hydroxide solution in the scrubber is the same for all standards and samples processed. The amount of cyanide contained in this basic scrubber solution is then determined colorimetrically. The colorimetric reaction involves the formation of cyanogen chloride (CNCl) by reaction with chloramine-T. It is essential that the same amount of chloramine-T is added to all standards and samples processed. Then pyridine-barbituric acid is added as a color development reagent. The colored complex is detected by measuring absorbance at 578nm with a spectrophotometer. Methods SW9010 C; SW9014 and EPA 335.2 alternately provide for the use of pyridine-pyrazolone (instead of pyridine-barbituric acid) as the color development reagent. If pyridine-pyrazolone is used, the time required for color development is longer (40 minutes rather than 8 minutes) and the samples must be read at 620nm (rather than 578nm).

To determine cyanide amenable to chlorination, two sample aliquots (one carried through a chlorination process, and one that is not) are processed. The amount of cyanide amenable to chlorination is determined by calculation (i.e., amount CN present in unchlorinated aliquot - amount CN remaining in chlorinated aliquot).

Liquid samples or extracts for determination of weak and dissociable cyanide are prepared by distillation under less acidic conditions than those established for total and amenable cyanide determination. Different preparatory reagents are also used. Colorimetric reaction, development and reading are the same as with total and amenable cyanide determination.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Oxidizing agents such as chlorine decompose most cyanides. Interference from chlorine can be removed by adding an excess of sodium arsenite ($NaAsO_2$) to the sample prior to preservation and storage. Sodium arsenite reduces the chlorine to chloride, which does not interfere. Alternatively, an excess of ascorbic acid may be used to remove the residual chlorine.

4.2 Sulfides interfere by producing hydrogen sulfide (H_2S) during distillation. Sulfide interference can be removed by adding an excess of bismuth nitrate [$Bi(NO_3)_3$] to the sample before distillation (to precipitate the sulfide). Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation, should be treated by the addition of bismuth nitrate.

4.3 Nitrate and nitrite at levels higher than 10mg/L may react with some organic compounds to generate hydrogen cyanide (HCN) yielding high results. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds, once formed, will decompose under test conditions to generate HCN. The possibility of interference from nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation.

4.4 Thiocyanate has been reported to be an interference when present at very high levels. Concentrations of 10mg/L were not found to interfere.

4.5 Where titration (rather that colorimetric development) is used as the determinative step, fatty acids, detergents, surfactants, and other compounds may interfere. When these substances are present in large concentrations, foaming may occur during distillation, thus making the endpoint of the titration difficult to detect. Section 6.8 of Method SW9010C and Section 5.3 of Method EPA 335.2 describe procedures for the removal of these interferent substances.

4.6 Some unidentified organic chemicals may oxidize or form breakdown products during chlorination, yielding higher results for cyanide *after* chlorination than before chlorination. This may lead to a negative value for cyanides amenable to chlorination after distillation for some wastes (e.g., steel industry, petroleum

refining, pulp and paper processing). Method 4500CN I (Dissociable Cyanide) is recommended where these interferences occur.

4.7 Some metal-cyanide complexes may be sensitive to ultraviolet light. Sample processing under incandescent light is recommended to prevent photodecomposition.

5. APPARATUS AND MATERIALS

5.1 Midi-Dist™ cyanide distillation system, Model 110-10R or equivalent, containing Midi glassware (e.g., reflux impingers, tubes and cold fingers; absorption impingers and tubes), all connective tubing, a flow meter and a heating block

5.2 Vacuum source for operation of the Midi-Dist™ system

5.3 Water source for operation of the Midi-Dist™ system

5.4 Spectrophotometer, Sequoia-Turner, Model 340 or equivalent

5.5 Cuvettes, optically matching, 1 inch diameter, Bausch & Lomb or equivalent

5.6 Cuvette stand

5.7 Analytical balance, capable of weighing to 0.0001 gram, and verified per SOP 305

5.8 Top-loading balance, capable of weighing to 0.01 gram, and verified per SOP 305

5.9 Volumetric flasks, glass, Class A, 100mL, 500mL and 1L sizes

5.10 Magnetic stir plate

5.11 Magnetic stir bars, Teflon™-coated

5.12 Pipets, variable Eppendorf™ or equivalent and disposable pipet tips, and operated per SOP 321

5.13 Pipets, plastic, disposable

5.14 Beakers, glass, 250mL

5.15 Beakers, plastic, disposable, 100mL

5.16 pH paper, narrow-range (acidic) and narrow-range (basic), or wide-range (0-14)

5.17 Indicator paper, KI-starch

5.18 Test paper, lead acetate

5.19 Centrifuge tubes, 50 and 250mLs, disposable (used in the extraction of cyanide from solid and oil matrices)

5.20 Centrifuge, capable of producing 3500rpm rotation

5.21 Tumbler, rotary

5.22 Filtration apparatus (vacuum pump, funnel, filter discs or glass wool)

6. REAGENTS AND STANDARDS

6.1 Reagent water, laboratory deionized (DI)

6.2 Clean silica sand, or Ottawa sand

6.3 Ascorbic acid, reagent grade, fine powder

6.4 Sodium hydroxide solution, 10N: Used for sample preservation and pH adjustment during sample preparation. Dissolve 40g NaOH in a final volume of 100mL using DI water. Shelf Life = 1 year.

6.5 Calcium hypochlorite solution, 0.35M: Used in determining cyanide amenable to chlorination. Use DI water to dissolve 5g $Ca(OCl)_2$ and bring to full volume in a 100mL Class A volumetric flask. **Store in an amber bottle; shake well before using**. Shelf Life = 1 month.

6.6 Magnesium chloride solution, 2.5M, 51% (w/v): Used as an anti-foaming agent during distillation. Obtained from a commercial vendor or made in-house by dissolving 510g $MgCl_2 \bullet 6H_2O$ in DI water and bringing to full volume in a 1L Class A volumetric flask. Shelf Life = 1 year.

6.7 Bismuth nitrate solution, 0.062M: Used prior to distillation to remove sulfide interferences. Dissolve 30g $Bi(NO_3)_3$ in 100mL of DI water. Add 250mL glacial acetic acid, stir. Bring to full volume in a 1L Class A volumetric flask. Shelf Life = 1 year.

6.8 Sulfamic acid solution, 0.4N: Used for the removal of nitrate/nitrite interferences. Use DI water to dissolve 40g NH_2SO_3H and bring to full volume in a 1L Class A volumetric flask. Shelf Life = 1 year.

6.9 Sodium hydroxide solution, 0.25N: Used as the scrubber solution and scrubber solution diluent; also used to prepare the working calibration standards. Dissolve 10g NaOH in a final volume of 1L using DI water. Shelf Life = 1 year.

6.10 Sulfuric acid solution 18N, 50% (v/v): Used to establish acidic conditions for distillation. Carefully add 100mL concentrated H_2SO_4 to 100mL DI water. Shelf Life = 1 year.

6.11 Sodium phosphate monobasic buffer solution, 1M: Dissolve 138g of $NaH_2PO_4 \bullet H_2O$ in 1L of DI water. ***Refrigerate.*** Shelf Life = 1 year.

6.12 Standard silver nitrate titrant, 0.0192N: Obtained from a commercial vendor or prepared in-house. Use DI water to dissolve 3.2647g dried $AgNO_3$ and bring to full volume in a 1L Class A volumetric flask. Shelf Life = 1 year.

6.13 p-Dimethylaminobenzalrhodanine indicator, reagent grade: Obtained from a commercial vendor or made in-house by dissolving 0.020g p-dimethylamino-benzalrhodanine in a final volume of 100mL acetone (spectral grade). Shelf Life = 1 year.

6.14 Stock potassium cyanide standard, 1000mg/L CN: Use 0.25N NaOH solution to dissolve 0.2504g potassium cyanide (KCN) salt and bring to final volume in a 100mL Class A volumetric flask. ***Refrigerate.*** Shelf Life = 1 year.

NOTE: Two stock solutions (one each) are prepared from two independent sources of KCN salt (i.e., "first source" and "second source").

The concentration of each of the stock potassium cyanide standard solutions (i.e., **first source** and **second source**) must be verified when made. Use the standardization procedure following to verify the stock solutions (*Note: this titration standardization procedure is detailed in SM4500-CN D)*:

6.14.1 Transfer 10.0mL of potassium cyanide stock standard to a 250mL beaker. Dilute to about 100mL using DI water.

6.14.2 Add a few drops of the p-dimethylaminobenzalrhodanine indicator solution.

6.14.3 Add a clean magnetic stir bar to the beaker and place the beaker + contents on the analytical balance; tare the balance.

6.14.4 Put the beaker + contents on a magnetic stir plate. Begin agitation.

6.14.5 Titrate with 0.0192N $AgNO_3$, adding dropwise, to the first change in color (i.e., from yellow to salmon). Reweigh the beaker + contents and record the weight change in the appropriate logbook (weight change equals amount of titrant added).

6.14.6 Titrate three replicate aliquots for each stock standard and average the results for each.

6.14.7 An aliquot of DI water (DI water blank) is also titrated according to the procedure described above.

6.14.8 Use the average result obtained for each stock standard to calculate the CN concentration as follows:

$$\text{mg } CN^- / L = \frac{(A - B) \times 1000^*}{C}$$

where:

A = mL standard 0.0192N $AgNO_3$ required for aliquot of stock
B = mL standard 0.0192N $AgNO_3$ required for DI water blank
C = mL CN- stock standard titrated**

* NOTE: 1.0mL 0.0192N $AgNO_3$ = 1.0mg CN^-

** (C = 10.0mL, if this SOP is followed without deviation)

6.14.9 The titration verification must yield results within ±2% (i.e., between 980 and 1020mg/L CN) in order for the stock standards to be acceptable. If this criterion is not met, the stock standards must be remade.

6.15 Intermediate potassium cyanide standard solution, 10mg/L: Add 1.00mL of 1000mg/L cyanide stock solution to 99.00mL of 0.25N NaOH. ***Store in a refrigerator (4±2°C).*** *Shelf Life = 3 months.*

NOTE: Prepare intermediate potassium cyanide standards from both of the first and second source stock KCN standard solutions. The first source 10mg/L intermediate potassium cyanide standard solution is used to spike the MS/MSD samples and to create the low and high distillation check standards. The second source 10mg/L intermediate potassium cyanide standard solution is used to prepare the ICV standard.

6.16 See Section 8 for directions for making the daily working calibration standards.

6.17 Chloramine-T solution: Dissolve 1g of chloramine-T in 100mL of DI water. *Prepare fresh each day of use.* ***Refrigerate until ready to use.***

6.18 Pyridine-barbituric acid solution: Place 7.5g of barbituric acid in a graduated container (i.e., marked at 125mL) and add a minimal amount of DI water necessary to wash the sides of the flask and wet the barbituric acid. Add 37.5mL of pyridine and mix. Add 7.5mL of concentrated HCl, mix and cool to room temperature. Dilute to 125mL with DI water and mix. ***Store in an amber glass bottle and refrigerate.*** *This reagent is stable for about 6 months; if a precipitate forms on the bottom of the container, discard and prepare fresh reagent*

6.19 Methyl red indicator: Used in the determination of weak and dissociable cyanide. Dissolve 0.10g methyl red sodium salt in 100mL of DI water. Shelf Life = 1 year.

6.20 Acetic acid solution, 1:9: Used in the determination of weak and dissociable cyanide. Mix 1 volume of glacial acetic acid with 9 volumes of DI water. Shelf Life = 1 year.

6.21 Acetate buffer: Used in the determination of weak and dissociable cyanide. Dissolve 41.0g sodium acetate trihydrate ($NaC_2H_3O_2 \bullet 3H_2O$) in 50mL DI water. Add glacial acetic acid (approximately 50mL) to yield a solution of pH 4.5. Shelf Life = 1 year.

6.22 Zinc acetate solution, 100g/L: Used in the determination of weak and dissociable cyanide. In a 100mL volumetric flask, dissolve 12g zinc acetate [$Zn(C_2H_3O_2)\bullet H_2O$], and bring to full volume with DI water. Shelf Life = 1 year.

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIME

7.1 All samples should be collected according to an approved sampling plan.

7.2 Plastic or glass containers may be used.

7.3 Oxidizing agents, such as chlorine, decompose most cyanide forms. To determine whether oxidizing agents are present, test a drop of sample with KI-starch indicator paper. A blue color indicates the need for treatment. To treat, add 0.1N sodium arsenite ($NaAsO_2$) solution, a few mL at a time, until a drop of sample produces no color on the KI-starch indicator paper. Add an additional 5mL of the 0.1N $NaAsO_2$ solution per each liter of sample. Ascorbic acid can be used as an alternative, although it is not as effective as sodium arsenite. Add a few crystals of ascorbic acid at a time (gently swirl after each addition), until a drop of sample produces no color on KI-starch indicator paper. Then add an additional 0.5g of ascorbic acid powder per each liter of sample volume.

7.4 Aqueous samples must be preserved by adding 2mL of 10N NaOH per L of sample volume (Method EPA 335.2) or sufficient 50% NaOH solution (Methods SW9010C and SM4500-CN B) until the pH of the sample is equal to or greater than 12 at the time of collection.

Carbonate in high concentration may affect the distillation procedure by causing the violent release of carbon dioxide with excessive foaming when reflux-distilled under acid conditions, thereby causing the pH of the absorption solution to be reduced. Calcium hydroxide (CaOH) should be used in lieu of NaOH to preserve samples suspected to contain significant carbonate amounts.

7.5 Samples should be kept cool (i.e., 4±2°C).

7.6 To meet ALS's holding time requirements, analyses must be completed within 14 days of sample collection.

8. PROCEDURE

8.1 EXTRACTION PROCEDURE FOR SOLID SAMPLES - Method SW9013

8.1.1 EXCEPTIONS

8.1.1.1 If the sample is a homogeneous fluid or slurry that does not separate or settle in the distillation flask when agitated but mixes so that the solids are entirely suspended, then the sample may be analyzed directly (per Method SW9010C) without an extraction step.

8.1.1.2 The sample may be analyzed directly (without extraction) per Method SW9010C if it is known to contain 50µg/g cyanide or

greater. Direct preparation for analysis can be accomplished by placing 1g of sample in the distillation flask and diluting to 50mL with 0.25NaOH.

8.1.2 Samples that contain free water may be filtered and separated into aqueous and solid components. The non-aqueous component may then be extracted and an aliquot of the extract combined with an aliquot of the filtrate in proportion to the composition of the sample (see Method SW9013, Section 7.0 for details). Alternately, the components may be analyzed separately with cyanide concentrations reported for each component.

8.1.3 The solid sample preparation procedure outlined below must be followed for clients requesting SW9013 - Cyanide Extraction Procedure for Solids and Oils. If Method SW9013 preparation for solid samples is not specified, the direct analysis alternative procedure (i.e., 1.0g sample added directly to the Midi-Dist™ apparatus as provided for in the Section 7.0 of the CLP SOW ILMO4.0) may be followed.

8.1.3.1 Place 10g of representative solid sample into a clean, labeled 250mL disposable centrifuge tube.

8.1.3.2 Prepare a method blank (MB) by placing 10g clean sand into a clean, labeled 250mL disposable centrifuge tube.

8.1.3.3 Add 195mL DI water and 5mL of 10N NaOH to each centrifuge tube.

NOTE: If heavy grease is present, 20mL of reagent grade n-hexane may be substituted for 20mL DI water in order to prevent an emulsion from forming.

8.1.3.4 Cap tube and shake for 1 minute. Check pH using indicator paper.

8.1.3.5 If pH <12, add 10N NaOH dropwise until pH is at least 12. Recap tube and shake for 1 minute. Check pH. Repeat this step as necessary until pH does not drop.

8.1.3.6 Place sample tubes into a 2L plastic jar(s) so that they can be placed onto the rotary tumbler. Extract by tumbling for 16 hours.

8.1.3.7 Centrifuge tubes at 3500rpm for 15 minutes or filter the extract using the vacuum pump, buchner funnel and glass fiber filter setup.

8.1.3.8 Proceed with pretreatment for cyanides amenable to chlorination (Section 8.3) or proceed with the distillation and

analysis of weak and dissociable cyanide (Section 8.5) or proceed directly with total cyanide analysis (Section 8.4), as applicable.

8.2 VERIFICATION OF AQUEOUS SAMPLE pH

Test the pH of each aqueous field sample by using a clean disposable transfer pipet to place a drop of aliquot onto a piece of pH test paper. Compare the paper's color to the pH packet's color standard chart and determine the sample's pH. Record result on LIMS benchsheet. The sample pH must be 12 or greater. If not, consult with the Project Manager for client direction as to how to proceed.

8.3 PRETREATMENT FOR CYANIDE AMENABLE TO CHLORINATION

8.3.1 Because cyanides amenable to chlorination is determined by subtracting the sample's total cyanides result from the amenable cyanides result, two identical sample aliquots are required to determine cyanides amenable to chlorination. Prepare two 50mL sample aliquots (or sample aliquots diluted to 50mL with DI water), into two labeled 100mL disposable beakers. *Only one of the sample aliquots is subjected to alkaline chlorination.*

A chlorinated batch duplicate (DUP) must also be prepared. Choose one sample per batch of twenty and prepare another 50mL aliquot for alkaline chlorination for that sample.

8.3.2 Dispense 50mL of DI water into a labeled 100mL disposable beaker to serve as the method blank (MB).

NOTE: The following procedures must be performed under incandescent light only because $K_3[Fe(CN)_6]$ may decompose under fluorescent light causing a false positive response for cyanide amenable to chlorination. For this reason and because of the possibility of toxic gases being evolved, this procedure must be performed in a fume hood.

8.3.3 Place a Teflon™-coated magnetic stir bar into each beaker to be subjected to alkaline chlorination, put the beakers on a stir plate and begin gentle agitation.

8.3.4 Add calcium hypochlorite solution dropwise (while maintaining a pH between 11 and 12 by adding 10N NaOH dropwise) until an excess of chlorine is present -- indicated by KI-starch paper turning blue upon testing. Maintain an excess of chlorine for one hour while gently agitating the sample continuously. Periodically test for excess chlorine with KI-starch paper and add additional calcium hypochlorite as necessary.

Also, periodically test pH using pH test paper to ensure that a pH between 11 and 12 is maintained; add 10N NaOH dropwise as necessary.

8.3.5 After one hour, add 0.05g portions of ascorbic acid until the KI-starch paper shows no residual chlorine (i.e., does not turn blue). Add 0.05g of excess ascorbic acid to ensure the presence of excess reducing agent.

8.3.6 Distill each chlorinated sample aliquot (along with the other aliquot of sample that was not subjected to alkaline chlorination) per Section 8.3 below.

Following spectrophotometric determination, the difference of cyanide content in the unchlorinated sample aliquot minus that of the chlorinated sample aliquot is the amount of cyanide amenable to chlorination for that sample.

8.4 DISTILLATION (TOTAL AND AMENABLE CYANIDE)

8.4.1 One method blank and one sample duplicate were prepared per each batch of twenty samples subjected to alkaline chlorination.

One sample duplicate must also be prepared for total cyanides by CLP SOW ILMO4.0. Select one sample per batch of twenty and dispense an additional aliquot to serve as the sample duplicate.

One method blank and one matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for each batch of twenty samples to be distilled for total cyanide analysis. Sample volume permitting, select one sample per batch of twenty (aqueous or solid samples) and dispense two additional sample aliquots to serve as the basis for the MS/MSD set. For solid samples not specified to be prepared by Method SW9013, weigh two 1.0g sample aliquots into two cyanide reflux tubes and add 0.25N NaOH to the 50mL marking for each to serve as the basis for the MS/MSD set.

To create a method blank for solid samples not specified to be prepared by Method SW9013 and/or solid samples processed via CLP SOW ILMO4.0, place 1.0g clean sand into a separate labeled cyanide reflux tube and fill with 0.25N NaOH to the 50mL marking.

8.4.2 Sample aliquots are tested for interferences and treated as needed. Sample aliquots are then distilled, followed by color development and measurement using a spectrophotometer.

8.4.2.1 Sulfide Test To test for the presence of sulfide interfering compounds in the sample, use a clean disposable transfer pipet to place a drop of sample on a piece of lead acetate paper. If the paper's color turns black, the presence of sulfide interference is indicated and the sample needs to be treated. Record the sulfide test result on the LIMS benchsheet.

For each sample yielding positive sulfide interference test results, add 1mL of 0.062M bismuth nitrate solution to each

dispensed aliquot. Add a Teflon™-coated magnetic stir bar to the beakers, transfer to a stir plate, and carefully mix for about three minutes. Repeat the lead acetate paper test.

Add additional bismuth nitrate solution, mix and repeat the lead acetate paper test as necessary until all sulfide interferences are removed

If a sample was treated with bismuth nitrate to remove sulfide interferences, add 5mL of 0.4N sulfamic acid through the air inlet tube. Allow to mix for about three minutes.

8.4.2.2 Oxidizing Agent Test To test for the presence of oxidizing agents in the sample, use a clean disposable transfer pipet to place a drop of sample on a piece of KI-starch indicator paper. A blue color indicates the presence of oxidizing agent interference and the need for sample treatment. Record the oxidizing agent test result on the LIMS benchsheet.

For each sample yielding positive oxidizing agent interference test results, add 0.05g ascorbic acid to each dispensed aliquot. Add a Teflon™-coated magnetic stir bar to the beakers, transfer to a stir plate, and carefully mix for about three minutes. Repeat the KI-starch indicator paper test.

Add 0.05g increments of ascorbic acid and repeat the KI-starch indicator paper test as necessary until a drop of aliquot produces no color on the KI-starch indicator paper. Then add an excess of 0.05g of additional ascorbic acid.

8.4.3 Place each prepared sample aliquot, including chlorinated sample aliquots, method blanks, duplicates and MS/MSDs, into separate, labeled cyanide reflux tubes.

8.4.4 Spike all the MS/MSD aliquots with 0.5mL of **first source** 10mg/L intermediate potassium cyanide standard solution. Expected concentrations for the MS/MSD samples is 0.10mg/L CN (aqueous samples), 5.0mg/Kg CN (solid samples)

8.4.5 ALS prepares both high and low distilled standards with each batch of cyanide samples processed as a check of the effectiveness of the distillation procedure. Concentrations of these distilled standards are compared to similar values on the calibration curve to ensure that the distillation technique is reliable.

Prepare the aqueous and Method SW9013 extract low (0.2mg/L) and high (0.4mg/L) distillation standards (which also serve as the laboratory control samples, LCSs) by pipetting 1.0mL and 2.0mL of the **first source** 10mg/L

intermediate potassium cyanide standard solution, respectively, into separate, labeled cyanide reflux tubes. Dilute to the 50mL mark with 0.25N NaOH.

To create the low (5.0mg/Kg CN) and high (20.0mg/Kg CN) distillation standards for solid matrices prepared by CLP SOW ILMO4.0, add 1.0mL and 2.0mL of the first source 10mg/L intermediate potassium cyanide standard solution to two separate 1.0g aliquots of clean sand placed into separate, labeled cyanide reflux tubes. Dilute to the 50mL mark with 0.25N NaOH.

NOTE: Additional distilled quality control samples (e.g., independent calibration verification standard, ICV) may be required based on client request. A distilled ICV is required by CLP SOW ILMO4.0 protocol.

To prepare the ICV for distillation, pipet 0.5mL of **second source** 10mg/L intermediate potassium cyanide standard solution into a labeled cyanide reflux tube and dilute to the 50mL mark with 0.25N NaOH. The expected concentration of this ICV standard is 0.10mg/L CN.

In addition to the distilled ICV, CLP SOW ILMO4.0 protocol requires that a reference soil sample with certified CN^- concentration be analyzed with each sample batch. A 1.0g aliquot of this reference soil (brought to the 50mL mark with 0.25N NaOH) is distilled and analyzed.

8.4.6 To all prepared aqueous sample or aqueous extract aliquots, add 1.0g of clean Ottawa sand. The clean sand is used in lieu of Teflon™ boiling chips; on-going successful Performance Testing (PT) analysis results show no adverse impact to method performance. The sand is not reused. No Ottawa sand needs to be added to prepared field or quality control samples already containing 1.0g of soil or Ottawa sand.

8.4.7 Add 50mL of 0.25N NaOH to each gas absorber tube.

8.4.8 Connect the reflux impingers, absorption impingers and cold fingers. Recheck all fittings.

8.4.9 Turn on the cooling water and adjust the flow rate indicated on the flow meter to 60GPH across the Midi-Dist™ unit.

8.4.10 Turn on the vacuum and adjust the valve until the level of bubbles in the gas absorber tube at each station is about 12cm above the 50mL mark.

8.4.11 If a sample is known or suspected to contain nitrate or nitrite, or if the sample aliquot was treated with bismuth nitrate to remove sulfide

interferences, add 5mL of 0.4N sulfamic acid through the air inlet tube. Allow to mix for about three minutes.

8.4.12 Use a pre-calibrated repeat pipettor to slowly inject 5mL of 50% (v/v) H_2SO_4 solution through each air inlet. Wait approximately 5 minutes while the acid mixes with the sample.

Sufficient acid must be added to bring the sample solution pH to <2. Very basic or highly buffered samples may require additional acid.

Add an additional 3mL of 50% (v/v) H_2SO_4 solution or check the pH if the sample aliquot's pH is suspect.

8.4.13 To control excessive foaming during distillation, add 51% MgCl2 solution to the sample through the air inlet, as needed.

8.4.14 Turn on the heating block (red switch). This will heat the block to 123-125°C. Adjust the timer to 105 minutes. The timer will automatically turn off the block heater.

8.4.15 After the heaters have turned off, allow the unit to cool 15 minutes (continue the vacuum).

8.4.16 Lift the fritted absorber impinger from the absorption tube. Disconnect the absorber-to-reflux connection. Disconnect the absorption impinger from the vacuum. Repeat for all Midi-Dist™ stations.

8.4.17 Turn off the vacuum valve; turn off water.

8.4.18 Seal the absorber tubes and store at 4±2 °C until colorimetric development and analysis. Make sure each tube is properly labeled with sample identity.

8.4.19 Disconnect the Midi-Dist™ glassware and clean the distillation apparatus per the manufacturer's instructions.

8.5 DISTILLATION (WEAK AND DISSOCIABLE CYANIDE) - METHOD SM 4500-CN I
The distillation for weak and dissociable cyanide is carried out under *slightly* acidic (pH 4.5 - 6.0) conditions. Preparative steps such as checking pH, checking for interferences, preparation of quality control samples, staging aliquots in CN reflux tubes, and other set-up procedures are conducted as described in the previous Sections. Continue by following the distillation procedure outlined in Section 8.4 above with the following exceptions:

8.5.1 Do not add sulfamic acid solution because NO_2^- and NO_3^- do not interfere.

8.5.2 Do not add H_2SO_4 or $MgCl_2$, instead, add 1mL each of the acetate buffer and zinc acetate solutions, followed by 2-3 drops of methyl red indicator.

8.5.3 Rinse the air inlet tube with DI water and allow the air to mix the reflux tube's contents. If the solution is not pink, add 1:9 acetic acid dropwise through the air inlet tube until a pink color persists.

8.6 Complete the distillation as described in Section 8.4 above., MANUAL SPECTROPHOTOMETRIC DETERMINATION
A series of calibration standards are prepared then developed colorimetrically. Absorbance at 578nm is read on a spectrophotometer. A standard curve is then calculated by plotting the absorbance for each standard vs. the standard's concentration.

8.6.1 Standard Curve Use 0.25N NaOH to prepare the calibration standards (made each day of use) in optically matching 1-inch diameter spectrophotometer cuvettes as described in the chart below:

Standard Concentration (mg/L)	Volume of 10 mg/L Intermediate Standard (mL)	Final Volume[ii] (mL)
0	0	10
0.01	0.010	10
0.05	0.050	10
0.10	0.10	10
0.20 (CCV)[i]	0.20	10
0.30	0.30	10
0.40	0.40	10
0.50	0.50	10
0.10 (ICV)[iii]	0.10	10

[i] From first source 10mg/L intermediate potassium cyanide standard solution.

[ii] Note that calibration standards are prepared directly in 1-inch, optically matched cuvettes.

[iii] From second source 10mg/L intermediate potassium cyanide standard solution.

8.6.2 Standard Curve for Samples Containing Sulfide Per all method references, the method of standard additions (MSA) must be used for the analysis of all samples that suffer from matrix interferences (e.g., samples that contain sulfides). MSA is not required where interferences have been successfully removed during pretreatment. Details regarding MSA

performance are provided in Method SW9014 (Section 7.4) and Method EPA 335.2 (Section 8.9).

8.6.3 Color Development and Absorbance Measurements

NOTE: The steps below *must* be performed in a fume hood.

8.6.3.1 For each distilled sample, pipet 10mL of scrubber solution (contained in the sealed absorber tube) into a pre-labeled 1-inch diameter, optically matched spectrophotometer cuvette.

8.6.3.2 Color development and absorbance measurements are performed in the following sequence:

1. 0.00 mg/L cal std
2. 0.01 mg/L cal std
3. 0.05 mg/L cal std
4. 0.10 mg/L cal std
5. 0.20 mg/L cal std
6. 0.30 mg/L cal std
7. 0.40 mg/L cal std
8. 0.50 mg/L cal std
9. ICV
10. ICB (calibration blank)
11. Preparation blank (distilled method blank)
12. LCS (distilled standards low and high; followed by the known reference material)
13. MS
14. MSD
15. Chlorinated amenable cyanide sample

16-22 Up to 4 field samples.

23. CCV (0.20 mg/L calibration standard, 1st Source)
24. CCB (calibration blank)
25. Up to ten more field or QC samples, then CCV, CCB (repeated until all samples are analyzed)

NOTE: No more than ten field or QC samples may be analyzed between the ICV/ICB and first CCV/CCB

set, or between successive CCV/CCB sets. A CCV/CCB set must close out the run sequence.

8.6.3.3 Add 3.0mL of 1M NaH_2PO_4 buffer solution to each cuvette.

NOTE: After this solution is added, the cyanide in the sample is no longer stable and the color development steps must be continued without delay.

8.6.3.4 Add 1.0mL of chloramine-T reagent to each cuvette and mix well.

8.6.3.5 After 1 to 2 minutes, add 1.0mL of pyridine-barbituric acid and 5.0mL of DI water; mix well immediately.

NOTE: All spectrophotometer checks must be performed per manufacturer's instructions prior to use.

8.6.3.6 After the red color has fully developed (usually about 8 to 15 minutes), read absorbance at 578nm using the spectrophotometer. Establish an instrument range from 0 to 100 % T or 0.00 to 2.000 absorbance.

8.6.4 Plotting the Standard Curve Prepare the standard curve by plotting the absorbance of each standard versus the standard's cyanide concentration (mg/L). This may be done using a spreadsheet program on a personal computer. Proceed with standard curve evaluation and calculations as described in the following Section.

8.7 CALCULATIONS

Perform a linear regression analysis of the plotted standard curve. The regression equation for the standard curve must have a correlation coefficient (r^2) that is $\geq$ 0.995. If this criterion is not met, check the standards preparation data, plotting and computation for errors. If no errors are found, the calibration standards can be rerun. If the criterion is still not met, the analysis must be halted, new calibration standards prepared, and a new calibration curve generated.

8.7.1 Calculate the cyanide concentration in aqueous samples as follows:

$$\text{Concentration of CN (mg/L)} = A \times D$$

where:

A = mg/L of CN in distillate as determined from the regression analysis

D = dilution factor (if dilution was necessary to produce a response within the calibration range)

8.7.2 Calculate the cyanide concentration in solid samples as follows:

$$\text{Concentration of CN (mg/Kg)} = \frac{A \times D \times V}{W \times E}$$

where:

A = mg/L of CN in distillate as determined from the regression analysis

D = dilution factor (if dilution was necessary to produce a response within the calibration range)

V = volume of distillate solution (liters); if this SOP is followed exactly, V = 0.050 L

W = wet sample weight (g)

E = correction factor for moisture content (i.e., % solids/ 100, as determined per SOP 642)

8.7.3 Once the concentration of total cyanide and amenable cyanide have been determined via the calculations shown above, the concentration of cyanide amenable to chlorination can be determined as follows:

Concentration CN Amenable to Chlorination	=	Concentration of Total CN (Unchlorinated aliquot)	-	Concentration of Total CN (Chlorinated aliquot)

9. QUALITY CONTROL (QC)

9.1 BLANKS

One preparation (i.e., method blank) must be carried through the entire preparation, reflux-distillation and analytical processes to determine if any contamination or memory effects are occurring. For this procedure, the preparation blank consists of DI water + clean sand (solid matrix samples not processed by Method SW9013 extraction). A clean sand (+ 50mL DI water and NaOH solution (0.25) solid matrix method blank is prepared and processed during the Method SW9013 extraction procedure. No blank may yield positive results greater than the analyte reporting limit (usually 0.01mg/L CN; 0.50mg/Kg CN). If this criterion is exceeded, the analyst should determine the cause of the problem, correct it and reanalyze the sample batch.

9.2 SPIKE RECOVERIES

Laboratory control samples (LCSs) consist of clean matrices (i.e., DI water and clean sand), which are spiked with a known amount of target compound. In some cases (e.g., CLP SOW ILMO4.0), pre-spiked reference samples are commercially available for use. LCS analyses are performed to evaluate the accuracy of the analytical system.

Matrix spike (MS) samples consist of additional sample aliquots, which are spiked with a known amount of target compound. MS analyses are performed to determine the effect of sample matrix interferences.

Spiked samples are evaluated based on percent recovery (%R), which is a calculation of the amount of target analyte yielded vs. the amount of target analyte anticipated. The following equation is used to calculate spike recovery:

$$\%R = \frac{Concentration_{Found} - Concentration_{Aliquot}}{Concentration_{Anticipated}} \times 100$$

where:

$Conc_{Found}$ = amount of target analyte yielded by the spiked analysis

$Conc_{Aliquot}$ = amount of target analyte found in the *unspiked* aliquot (i.e., "zero" for clean LCS matrix aliquots; or concentration of target analyte calculated to be present in the unspiked sample matrix aliquot)

$Conc_{Anticipated}$ = amount of target analyte expected to be yielded based on known amount spiked

The spiked LCS results should agree within ±15% of the anticipated value for aqueous samples (i.e., 85-115 % recovery), and within vendor-specified limits for solid reference samples. *Note that other acceptance criteria may be applicable as requested by the client.* The advisory control limits for the matrix spiked analyses are set at 75-125% recovery.

If established spike recovery quality control criteria are not met, check all calculations and spike preparations for errors. If no errors are found, the sample batch must be reanalyzed for LCS exceedances. Narrate if sample matrix interference is suspected as the cause of MS (or MSD) exceedance.

9.3 DUPLICATE PRECISION

A sample duplicate is prepared for amenable cyanide determinations and for cyanide analyses per CLP SOW ILMO4.0 protocol. Also, generally the matrix spike sample is prepared and analyzed in duplicate. Duplicates are analyzed to measure analytical precision. Precision is evaluated in terms of Relative Percent Difference (RPD), which is calculated as follows:

$$RPD\ (\%) = \frac{|Result_{Sample} - Result_{Dup}|}{\frac{(Result_{Sample} + Result_{Dup})}{2}} \times 100$$

Generally, an RPD of ≤20 is set as the quality control limit. This RPD limit is not applicable for matrix spiked duplicates whose native (i.e., unspiked) concentration of target analyte is greater than 5X the analyte's reporting limit.

If the RPD quality control criterion is not met, check all calculations and spike preparations for errors. Consult with the Department, Project and Quality Assurance Managers if the RPD criterion is not met for duplicates. Narrate if sample matrix interference is suspected as the cause of the RPD limit not being met for an MS/MSD set.

9.4 METHOD DETECTION LIMIT STUDY

The Detection Limit (DL/LOD) is performed as needed, at a minimum, annually, following the guidance of SOP 329.DEVIATIONS FROM METHODS

10.1 Note that ALS utilizes a Midi-Dist™ apparatus for distillation in lieu of a round bottom flask setup.

10.2 Section 7.7 of Method SW9013 describes the use of 50% NaOH (added in 5mL increments as needed) to maintain a pH of 12 or greater during the extraction of cyanide in solid and oil matrices. Instead, this SOP prescribes the addition of 10N NaOH dropwise to achieve adequate pH adjustment.

10.3 Section 4.1 of Method EPA 335.1 and Section 7.1.2 of Method SW9010C describe the use of 1.25N NaOH to maintain a pH between 11 and 12 during the chlorination process for cyanides amenable to chlorination. Instead, this SOP prescribes the addition of 10N NaOH dropwise to achieve adequate pH adjustment.

10.4 Section 7.1.4 of Method SW9010C discusses the use of sodium arsenite to remove all residual chlorine after the amenable cyanide chlorination process. As described in Section 4.3 of Method EPA 335.1, ALS uses ascorbic acid to neutralize all residual chlorine generated in the amenable cyanide chlorination process.

10.5 Section 8.0 of Method EPA 335.2 and Section 7.2.1 of Method SW9010C indicate that 1.25N NaOH is to be used as the reflux scrubber solution. ALS utilizes 0.25N NaOH as the reflux scrubber solution. Successful Proficiency Test (PT) sample studies show that this practice does not impair sample results.

10.6 Per Section 8.8 of Method EPA 335.2, 1.25N NaOH is used in the creation of working cyanide standards; 1N NaOH is used in the creation of working cyanide standards per Sections 5.3.7 of Method SW9014. ALS uses 0.25N NaOH to create the working cyanide standard solutions. This provides for the same matrix as the distillates that are trapped in 0.25N NaOH.

10.7 To remove sulfide interferences, Section 8.2 of Method EPA 335.2 describes the addition of lead acetate to the reflux scrubber and Section 4.2 of the CLP SOW ILMO4.0 discusses the use of powdered cadmium carbonate. Per Method SW9010C (Sections 3.3 and 7.2.3), this SOP prescribes the use of 0.062M bismuth nitrate solution to remove sulfide interferences.

10.8 The referenced methods discuss addition of sulfamic acid powder to the sample prior to distillation for the removal of nitrate/nitrite interferences. To provide for better delivery into the Midi-Dist™ system, ALS uses a 0.4N sulfamic acid solution instead.

10.9 The referenced methods describe the use of Teflon™ boiling chips during the distillation process. ALS uses 1.0g of clean silica sand in lieu of boiling chips. On-going successful Performance Testing (PT) performance has shown no adverse impact to method performance.

10.10 Section 8.7.1 of Method EPA 335.2 and Section 7.2.2 of Method SW9014 cite the addition of 2mL chloramine-T reagent and 5mL pyridine-barbituric reagent during the color development process (ALS uses 1mL each because smaller volumes are

developed colorimetrically). Also, Section 7.2.2 of Method SW9014 discusses using KI-starch paper to test for excess chlorine following the addition of the 2mL chloramine-T reagent, with additional increments of 0.5mL chloramine-T added until chlorine residual is achieved and with a final excess of 0.5mL chloramine-T added. ALS does not test for chlorine residual. The generation of successful Proficiency Test (PT) sample results have shown that these practices do not adversely affect sample results.

10.11 Section 8.8.2 of Method EPA 335.2 and Section 8.6 of Method SW9010C recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. Both method references state that if the distilled standards do not agree within ±10% of the undistilled standards, the analyst should find the cause of the apparent error before proceeding. It is ALS's policy (per Section 8.3 of Method SW9010C and Section 7.2.2.1 of the CLP SOW ILMO4.0), that the distilled check standards must yield values within ±15% of the expected value. It is ALS's interpretation that the conflicting criterion given in Method SW9010C (Section 8.6 vs Section 8.3) be resolved in favor of the looser ±15% criterion.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

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11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 9010C", Revision 3, November 2004.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Final Update IV, "Method 9013A", Revision 1, November 2004.

12.3 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 3rd Edition, Final Update III, Chapter 5, Method 9014, "Titrimetric and Manual Spectrophotometric Determinative Methods for Cyanide", Revision 0, December 1996.

12.4 US EPA, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, Method 335.1, "Cyanides, Amenable to Chlorination", 1974.

12.5 US EPA, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, Method 335.2, "Cyanide, Total (Titrimetric, Spectrophotometric)", 1980.

12.6 US EPA, EPA-540/R95/121, Contract Laboratory Program (CLP), Statement of Work (SOW) for Inorganics Analysis, Multi-media, Multi-concentration, ILMO 4.0.

12.7 Standard Methods for the Examination of Water and Wastewater, 20th Edition, Part 4000, Method 4500-CN I, "Weak and Dissociable Cyanide", 1998.

Analytical Method: SW 9010,C, 9013, 9014; E 335.1, 335.2; OLMO4.0; SM4500-CN I	**Parameter:** Total and Amenable Cyanide by Distillation; Weak and Dissociable Cyanide		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 7-point (plus blank)	As needed (i.e., at on-set of analyses or when continuing calibration does not meet criteria)	Correlation coefficient (r^2) for linear regression must be $\geq$ 0.995	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze calibration standards. If quality control acceptance criterion still not met, analyses cannot proceed; a new suite of calibration standards must be prepared and analyzed. Analyses cannot proceed until an acceptable initial calibration curve is generated.
Independent Calibration Verification (ICV); second source standard; at or below midpoint	Once after each initial calibration	Results must agree within $\pm$15% of corresponding value generated by the initial calibration	If QC criterion not met, prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV); first source; at or below midpoint	Run to bracket a set of 10 analyses, and to end any run sequence (must be followed by a CCB analysis)	Response must agree within $\pm$15% of corresponding value generated by the initial calibration	Check for preparation and calculation errors; evaluate/correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.
Blanks: Method (Preparation, MB) Also run as an Initial Calibration Blank (ICB) and Continuing Calibration Blanks (CCBs)	One method blank per batch of twenty or less environmental samples processed. ICB run immediately following calibration curve. CCB run following the CCV to bracket a set of ten analyses and to close a run sequence	CN content of the blank must be less than the analyte reporting limit (RL); RL usually 0.01mg/L CN; 0.50mg/Kg CN	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.
Distilled LCS (low and	One low and one high	Distilled LCS result must	Check data for preparation or

Analytical Method: SW 9010,C, 9013, 9014; E 335.1, 335.2; OLMO4.0; SM4500-CN I	**Parameter:** Total and Amenable Cyanide by Distillation; Weak and Dissociable Cyanide		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
high distillation check standards)	prepared and analyzed per batch of ≤ 20 field samples	agree within ±15% of non-distilled ICV result	calculation errors. If no errors are found, the distillation of these standards and all associated samples in the batch must be repeated.
Distilled ICV; second source ICV subjected to reflux-distillation (run per CLP protocol and by client request); at or below midpoint	Once per analytical run sequence	Distilled ICV result must agree within ±15% of non-distilled ICV result (see Section 10.0 - Deviations, of this SOP)	If QC criterion not met, check for preparation errors or instrument malfunction. If distilled low and high LCS criteria also not met, repeat distillation for all samples in batch.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set prepared and analyzed per batch of ≤20 field samples	Recoveries should meet control limits of 75-125 % RPD between duplicates should be ≤20	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.
Sample Duplicate (DUP), only for Method 335.1	One prepared and analyzed per batch of ≤20 field samples.	RPD should be ≤20	For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/Project/QA Managers (narrate if sample matrix interferences are suspected as the cause of the RPD limit not being met for an MS/MSD set).
Method Detection Limit (MDL) Study; run at an analyte concentration near to but lower than the reporting limit (RL) and per SOP329	As needed; at a minimum annually	Positive result < the analyte reporting limit (RL)	Determine the reason for failure and fix problem with the system. Repeat the MDL study. If criteria still not met, discuss with QA Manager (RL may be adjusted if required).

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY
REFERENCED METHOD:	METHODS EPA 300.0 AND SW9056
SOP ID:	1113
REV. NUMBER:	14
EFFECTIVE DATE:	JANURY 16, 2015

ALS Environmental

ALS
STANDARD OPERATING PROCEDURE 1113 REVISION 14

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY -- METHODS EPA 300.0 AND SW9056

FORMS: APPENDIX A

APPROVED BY:
PRIMARY AUTHOR _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY DIRECTOR _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- Methods EPA 300.0 and SW9056 -- are used to determine the concentration of selected ions in environmental water samples and in aqueous extracts of environmental solid samples. An Ion Chromatograph (IC) is used to separate and detect the anions. ALS typically uses this procedure to determine the following anions: Chloride (Cl^-), Bromide (Br^-), Fluoride (F^-), Nitrate (NO_3^-), Nitrite (NO_2^-), Sulfate ($SO_4^{=}$), and Orthophosphate (PO_4^{3-}).

2. SUMMARY

The anions are separated and measured using an analytical system consisting of an autosampler, pump, and an IC containing ion exchange columns, a suppressor device, and a conductivity detector.

Small volumes of sample (typically 5mL) are injected via the autosampler into the IC to flush and fill the 25µL sample loop. The contents of the filled sample loop are then injected into a bicarbonate-carbonate eluent stream, which is pumped through a series of two ion exchange columns. The anions of interest are separated by the two ion exchange columns and then carried by the eluent stream through a third (suppressor) column. The suppressor column reduces the anions to their acid form, and also reduces the background conductivity of the eluent. Next, the eluent stream passes through a conductivity detector, and its signals are captured and maintained by a microprocessor (PC). The anions are identified based on retention times and are quantitated using an external standard calibration method.

Solid samples must first be extracted using deionized (DI) water prior to determination.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the ability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of

Supervisory/training review, results of precision and accuracy tests performed, or by the successful analysis of a proficiency test sample.

3.3 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the workorder file indicate that this review for precision, accuracy and completeness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work, and documentation of measures taken to remediate the data.

3.4 It is the responsibility of all personnel who work with samples or data involving this method to consult the applicable LIMS Program Specification for client-specific requirements prior to initiating handling of samples or data.

3.5 It is the responsibility of all personnel who work with samples involving these methods to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Samples that contain particles larger than 0.45µm and reagent solutions that contain particles larger than 0.20µm require filtration to prevent damage to instrument columns and flow systems.

4.2 Constituents that elute at retention times close to the retention times of analytes may interfere. Sample spiking with the analyte of interest can be used to solve most interference problems associated with retention times.

4.3 Large concentrations of an anion can cause interference by causing poor resolution or by overloading the capacity of the column. Sample dilution can be used to solve most interference problems associated with column overloading.

4.4 Several of the anions (NO_3^-, Cl^-, $SO_4^{=}$) are commonly found in the laboratory in concentrated acids and can easily cause contamination. Clean glassware and reagents are critical in order to prevent the occurrence of a noisy baseline or contamination of samples.

5. APPARATUS AND MATERIALS

5.1 Dionex DX-120 Ion Chromatograph (IC), or equivalent, equipped with an autosampler, eluent pump and conductivity detector

5.2 Dionex AS-14 or AS-14A anion exchange analytical columns with AG-14 or AG-14A guard columns, or equivalents

5.3 Anion ASRS-Ultra self-regenerating suppressor, 4mm, or equivalent

5.4 Dionex *PeakNet* software, or equivalent

5.5 Dionex auto sampler vials ("PolyVials") with filter caps, 5mL, with appropriate 6-vial cassette holder, or equivalent

5.6 centrifuge tubes, polypropylene, disposable, 50mL

5.7 filter disks, 0.45

5.8 insertion tool for the PolyVial filter caps

5.9 Eppendorf™ adjustable pipettors, or equivalent, operated per SOP 321 requirements

5.10 volumetric flasks, various sizes, Class A

5.11 analytical balance, capable of weighing to 0.0001g, verified per SOP 305

5.12 conductivity meter

5.13 TCLP-type rotary tumbler

5.14 PolyVials, disposable, 5mL with caps

5.15 vortex mixer

5.16 Disposable 14 mL polypropylene tubes

6. REAGENTS – Only reagent grade or better chemicals may be used!

6.1 Deionized (DI) water, obtained from the laboratory's deionized water system

6.2 Helium (He) compressed gas, high purity grade or better

6.3 Eluent Solution Concentrate, 0.1M $NaHCO_3$ + 0.35M Na_2CO_3 (for AS-14/ AG-14): Dissolve 8.4g of sodium bicarbonate ($NaHCO_3$) and 37.1g sodium carbonate (Na_2CO_3) in DI water and dilute using DI water to 1000mL final volume. *Store at room temperature. Shelf Life = 1 year.*

0.1M $NaHCO_3$ + 0.8M Na_2CO_3 (for AS-14A/ AG-14A): Dissolve 8.4g of Sodium bicarbonate ($NaHCO_3$) and 84.8g sodium carbonate (Na_2CO_3) in DI water and dilute using DI water to 1000mL final volume. *Store at room temperature. Shelf Life = 1 year.*

6.4 Eluent Working Solution: Dilute the eluent concentrate solution 100-fold using DI water. Usually, 10mL of concentrate is diluted to a final volume of 1L. *Make fresh daily. Store at room temperature.*

7. STANDARDS

Two Stock Solutions (i.e., "first source" and "second source") are required for each analyte to create the necessary calibration and spiking solutions. Standard and spiking solutions may be prepared in-house from ACS reagent grade materials or can be purchased as certified solutions from a commercial vendor. ALS's Standards and Solutions database is used to document and manage all standards in use at ALS. **All**

anion standards are to be kept refrigerated at 4±2°C and Nitrite standards have shelf life of one month. *Consult the Standards and Solutions database for the concentrations and traceability of the Nitrite (NO_2^-) and anion (Cl^-, Br^-, F^-, NO_3^-, $SO_4^=$, PO_4^{3-}) standards and solutions used in this procedure.*

8. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

8.1 Samples should be collected according to an approved sampling plan.

8.2 Samples should be collected in clean plastic or glass bottles. The volume collected should be sufficient to provide a representative sample and to allow for the analysis of quality control samples (i.e., matrix spike and duplicate).

8.3 Samples requiring analysis of nitrate, nitrite, and orthophosphate must be kept cool (4±2°C) and must be analyzed within 48 hours of collection.

8.4 Samples requiring analysis of bromide, chloride, fluoride and sulfate should be kept cool (4±2°C) and must be analyzed within 28 days of collection.

9. PROCEDURE

9.1 PREPARATION OF CALIBRATION STANDARDS

Calibration standards (see compositions below) are prepared by diluting the first source anion intermediate standard at five different levels using eluent working solution as the diluent. An eluent working solution blank is used as the 6th calibration point.

ANION CALIBRATION STANDARDS (mg/L)

Analyte Standard	#1 1000X	#2 100X	#3 25X	#4 10X	#5 5X
F^-	0.05	0.5	2	5	10
Cl^-	0.1	1	4	10	20
Br^-	0.1	1	4	10	20
NO_2^- as N	0.05	0.5	2	5	10
NO_3^- as N	0.1	1	4	10	20
PO_4^{3-} as P	0.1	1	4	10	20
$SO_4^=$	0.5	5	20	50	100

Calibration standard #4 (first source) is also used as the continuing calibration verification (CCV) standard. The "second source" intermediate anion standard is used to prepare the independent calibration verification (ICV) standard. With the exception of Nitrite, the ICV is made at concentrations half that of the CCV (concentration of Nitrite is 2.0mg/L).

9.2 SYSTEM START-UP AND SHUT-DOWN

9.2.1 Turn on gas and set He regulator to 2psi.

9.2.2 Turn on power by depressing the white button on the front of the instrument.

9.2.3 From the "Main Menu" window in *PeakNet*, click on the "**PEAKNET RUN**" icon. In the Peak Net Run window, select the "**DIRECT CONTROL**" icon. In the Direct Control window, click to start the pump, eluent pressure and SRS/cell.

9.2.4 The eluent flow rate should be set at 1.20mL/min for AS-14 or 1.00mL/min for AS-14A column/guard column. Check this manually by using a graduated cylinder to collect eluent from the waste line and calculate the flow rate from the volume expelled over the time period collected.

9.2.5 Allow the pressure to stabilize for about 30 minutes before starting an analytical sequence.

9.2.6 To shut down system, turn off the pump, eluent pressure and SRS/cell from the "**DIRECT CONTROL**" window. Turn off power (white button on main panel) and close gas tank valve OR:

NOTE: Turn on and off by pressing Local/Remote on front panel to highlight Local and manually press pump, eluent pressure and SRS/ cell.

9.3 EXTRACTION OF SOLID SAMPLES

9.3.1 Weigh 4.0g of sample into a 50mL polypropylene centrifuge tube. Add 40mL DI water.

9.3.2 Mix the suspension thoroughly and tumble using a rotary tumbler (TCLP-type) for about 60 minutes.

9.3.3 Centrifuge the resulting slurry for about 15 minutes.

9.3.4 Filter the supernatant using a 0.45µm filter disk.

9.4 SCREENING AQUEOUS SAMPLES AND SOLID SAMPLE EXTRACTS

Samples should be screened prior to analysis using a conductivity meter. Record screening results in logbook (Form 1116). The electrical conductivity of an aqueous solution is directly related to the concentration of total dissolved solids (TDS). The type of meter used is capable of converting conductivity to estimated TDS in mg/L.

9.4.1 It may be necessary to dilute a sample/extract prior to injection into the IC in order to protect the instrument and provide for sample analysis within the instrument's calibrated linear range. Samples/extracts should be diluted with eluent working solution as necessary so that solutions injected into the instrument do not exceed 1000mg/L

estimated TDS (the sample screening information is used to determine the extent of dilution required). Allow several hours (overnight if possible) for the precipitate to form and then filter before injecting. Visual appearance of the sample/extract, historical data, or additional information provided by the client may also provide information used to determine the extent of dilution needed.

NOTE: Use a small amount of eluent concentrate instead of eluent working solution when a dilution is not required, in order to remove the "water dip" before the fluoride peak. .

9.4.2 Prepare aliquots of each pre-filtered sample or extract for analysis in 5mL Dionex vials; dilute as necessary per discussion above. Place filter caps half way down on vials and vortex. Use insertion tool to push cap down fully on vials. Load vials into cassettes for the autosampler.

9.5 SEQUENCE SET-UP

9.5.1 Analytical sequences are set-up using the *PeakNet* Chromatography Software. A new "**SCHEDULE**" is created for each day. Typically this schedule is renamed from the previous day's schedule and then edited to create the new schedule. Sample, sample type, method, and data file must be filled out correctly to save properly.

9.5.2 Each day's data are stored in a new subdirectory named for the day, using a yymmdd format. A typical daily analytical sequence is shown below:

1 - 6	calibration standards (when ICAL is performed)
7	ICV
8	ICB
9	MB
10	LCS
11 – 18	8 samples, may include MS/MSD
19	CCV
20	CCB

An analytical sequence can continue indefinitely by repeating Steps 9 – 20, so long as the quality control criteria are met. No more than 10 samples/extracts (including QC samples) may be analyzed between calibration verifications (i.e., CCV, CCB set). One Method Blank, one LCS must be analyzed per twenty environmental samples of like matrix. A CCV, CCB set must close the analytical sequence. A CCV, CCB is used to initiate analysis when an ICAL is not performed.

At the end of the sequence, the autosampler stops, but the eluent pressure and SRS/cell pump will continue to run until stopped

manually; unless a stop method (Stop.met) is placed at the end of the schedule in the method column.

9.6 PEAKNET METHOD

A "**METHOD**" is created in *PeakNet* when calibration is needed. The method is a subprogram, which controls the events that take place throughout the analytical sequence (trigger autosampler, operate injection valve, start and end data acquisition, etc.) The method contains the integration parameters that are used in processing the raw data. The method also contains a table with the calibration curves and retention time windows for each analyte. This table is updated each time a new calibration is established. However, retention times may be updated as necessary using the information obtained from the first calibration verification check run that day.

9.7 DATA ACQUISITION AND PROCESSING

9.7.1 AUTOSAMPLER OPERATION

- Place cassette on autosampler facing inward.
- Press **"**Hold/Run**"** button (on autosampler) to advance cassette to injection port.
- Then press "Load" (on autosampler).
- After the autosampler "Load" button light begins to blink, click on the "Start" icon in the *PeakNet* "**RUN**" window.

Usually, a new injection is made every 10 minutes. Each chromatogram is stored in a separate file. Filenames are automatically given to each chromatogram based on information that is entered when the sequence is created (i.e., a filename prefix, and a value for the counter). The filename prefix is the date of acquisition, and the counter is set to add one for each injection (e.g., yy0209_005.dxd designates the 5th injection made on February 9^{th,} 20xx.

9.7.2 Data processing (integration, peak identification, quantitation) is performed after the chromatograms have been acquired. It is important to review the integration to ensure that baselines are drawn correctly. In cases where it is necessary to modify a baseline, the raw data must include printouts of the chromatogram before and after the baseline correction (refer to SOP 939 for further directives). Only values that fall between the lowest and highest calibration standards are reported. Samples yielding results that exceed the highest standards should be diluted and reanalyzed. Results are reported in mg/L. Report NO_2^- as N, NO_3^- as N, and PO_4^{3-} as P.

9.8 RUN LOG

A record of each day's analyses is entered into the instrument "**SCHEDULE**". The following information is recorded:

- Date of analysis
- Analyst's initials
- Solution ID (including any dilutions)
- Filename
- Schedule ID

9.9 MAINTENANCE AND TROUBLESHOOTING

9.9.1 A maintenance logbook is used to record all information concerning instrument maintenance. The logbook documents all repairs and symptoms of problems.

9.9.2 To reduce the accumulation of metal hydroxide precipitates, components of the autosampler need periodic cleaning. This cleaning should be performed when a new guard column is installed. Components include

- the tubing carrying the sample from the autosampler to the injection valve
- The injection valve
- the sample loop

9.9.3 Cleaning Procedure

9.9.3.1 Disconnect guard column from injection valve.

9.9.3.2 Place a beaker in an appropriate position to collect waste solution exiting the injection valve.

9.9.3.3 Start eluent pump

9.9.3.4 Load 10 sample vials with 0.1 M HCI and inject using the autosampler

9.9.3.5 Load 5 sample vials with deionized water and inject using the autosampler

9.9.3.6 Connect guard column to injection valve

9.9.4 The fritted disks (sometimes called "bed supports") at the ends of the guard and analytical columns should be changed periodically. The system backpressure will gradually increase over a period of many days or weeks as the fritted disks become clogged with small particles.

9.9.5 In blanks (ICBs, CCBs, MBs), small peaks may be observed in the retention time windows for various anions after analyzing samples high in dissolved salts. This problem is most frequently encountered with orthophosphate. Carryover effect can be addressed via the following:

- Carryover may be lessened or prevented by setting up the autosampler to pass an aliquot of eluent working solution through the sample loop between sample injections. To accomplish this, a vial of eluent working solution is placed between each vial containing a sample or standard in the autosampler. The autosampler recognizes the added vial to be a "rinse vial" because the filter cap is pushed down to the first position using the special insertion tool.

- The sample loop and the tubing that carries the sample solution from the autosampler to the IC can be cleaned with 0.1N HCl using a syringe with a luer lock connector. All traces of HCl must be removed from the system by rinsing the line thoroughly with DI water before analyzing samples.

9.9.6 The guard column will need to be replaced more frequently than the analytical column. Generally, the guard column is replaced about three times for each analytical column that is used over approximately a 1-year period. Overlap of peak retention times is a sign that the analytical column needs replacing.

10. QUALITY CONTROL (QC)

10.1 DEFINITION OF ANALYSIS BATCH

An analysis batch is defined as a group of twenty (20) or less field samples of like matrix that is associated with one unique set of batch QC samples and processed as a unit. Batch QC samples are defined as the method blank (MB), laboratory control sample (LCS), matrix spike and duplicate (MS/MSD). All QC samples must be carried through all stages of the sample preparation and measurement steps.

10.2 METHOD BLANKS

Method blanks (MBs) are run to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Each time a batch of samples is analyzed or there is a change in reagents, a method blank must be processed. The blank concentration found must be less than the analyte reporting limit.

10.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) should contain all the analytes of interest and is analyzed to measure the accuracy of the method. To be acceptable, the LCS recovery must be between 90% and 110% of the expected concentration for each analyte of interest for aqueous samples, and between 85% and 115% of the expected concentration for each analyte of interest for solid sample extracts.

10.4 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE

Matrix spikes (MS) consist of field samples into which known concentrations of target analytes are injected and analyzed as a means of determining the effect of matrix on target analyte detection. The matrix spike duplicate (MSD) serves as a laboratory duplicate analysis. All of the analytes of interest should be spiked. Analyte recovery for matrix spikes is calculated as shown below:

$$\text{MS \% Recovery} = \frac{(C_{found} - C_{native})}{C_{added}} \text{ X } 100$$

where:

C_{found}	=	analyte concentration found in the spiked sample
C_{native}	=	native analyte concentration found in the unspiked sample
C_{added}	=	spike added analyte concentration

The quality control acceptance limits for MS/MSD recovery vary (consult LIMS Program Specifications).

The MSD is analyzed as a measure of the precision of the analytical results generated. The Relative Percent Difference (RPD) between a sample and its duplicate should not be greater than 20% (or as specified by client criteria). RPD is calculated as shown below:

$$\text{RPD} = \frac{| Result_x - Result_{Dup} |}{(Result_x + Result_{Dup}) / 2} \text{ X } 100$$

Acceptance criteria for all spikes and duplicates should be met. If MS/MSD recovery or RPD criteria are not met, results of the laboratory control sample analyses must be carefully considered. If LCS results are acceptable, sample matrix interference is suspected and a notation in the narrative comments is made.

10.5 RETENTION TIME WINDOW (RTW) STUDY

Analyte retention time window studies are conducted periodically, at minimum upon column change out. An analyte RTW study is performed by noting the variations in analyte retention times yielded in the various concentrations of standards run across the course of a day. The analyte RTW study is typically accomplished when initial calibrations are performed. The width of the RTW is established for each analyte based on three times the standard deviation of the analyte retention times noted during the study. Subsequent CCVs are evaluated against these established times and windows, and are acceptable if the RT has not drifted more than 10%. The CCV RT may be used to re-center the window, so long as the 10% drift criterion has been met. If the 10% drift criterion is exceeded, a new ICAL is performed. RTWs are used to identify target analytes,

however, the experience of the analyst must also weigh heavily in the interpretation of chromatograms.

10.6 METHOD DETECTION LIMIT STUDY
The Detection Limit (DL/LOD) is performed as needed, at a minimum, annually, following the guidance of SOP 329.

11. DEVIATIONS FROM METHOD

11.1 Section 7.3.3 of Method SW846 9056 describes instrument calibration by the use of a first order (linear) calibration curve. ALS employs **the type of curve fit that best fits the data generated for each analyte. For example, a linear calibration curve may be used for fluoride, whereas** a second order (quadratic) calibration curve, **consisting of** six points ('zero' point response included; curve not forced through zero), **may be used for other analytes,** based on historical experience that this type of calibration curve (quadratic) provides a better fit of the **analyte** calibration data.

11.2 ALS notes that Method EPA 300.0, Section 7.3, prescribes an eluent concentration of 1.7mM sodium bicarbonate and 1.8mM sodium carbonate. ALS uses an eluent concentration of 0.1M sodium bicarbonate and 0.35M sodium carbonate when using AS-14 columns, and an eluent concentration of 0.1M sodium bicarbonate and 0.8M sodium carbonate when using AS-14A columns, per the application information given with each Dionex column. This eluent profile enables run time to be decreased while maintaining adequate peak resolution throughout the analysis.

12. SAFETY, HAZARDS AND WASTE DISPOSAL

12.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

12.2 WASTE DISPOSAL
All wastes are disposed of in accordance with the Waste Management Plan (WMP)

13. REFERENCES

13.1 U.S. Environmental Protection Agency, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, revision 2.1, 1993. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography".

13.2 U.S. Environmental Protection Agency, SW846, Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, 1996. Method 9056, "Determination of Inorganic Anions by Ion Chromatography".

Analytical Method: E 300.0; SW9056	**Parameter:** Determination of Anions by Ion Chromatography		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Retention Time Window (RTW) Study	Performed initially and as necessary (i.e., redone when required as part of troubleshooting when daily RTWs do not agree within ±10% of expected values)	Analyte RTWs established as three times the standard deviations of the analyte retention times noted in the various concentrations of standards run during the course of the day's study	If analyte retention time not reproducible, identify and correct problem. Perform a new initial calibration. Analyte RTWs must agree within ±10% of expected values.
Initial Calibration; minimum 6-point (includes blank, but curve not forced through zero)	As needed (i.e., at on-set of analyses or when continuing calibration does not meet criteria)	**First or s**econd order **(**quadratic**)** fit calibration curve generated electronically by instrument software. Coefficient of Determination (r^2) of the calibration curve equation must be ≥ 0.99	Evaluate/correct instrument malfunction, prepare a fresh set of calibration standards, and reanalyze initial calibration to obtain an acceptable curve.
Independent Calibration Verification (ICV); second source at or below midpoint	Run immediately following initial calibration	Analyte concentration must agree within ±10% of expected values for both Methods SW9056 and 300.0; analyte retention time must agree within ±10%	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Initial Calibration Blank (ICB)	Run immediately following the ICV	Anion content of ICB must not exceed analyte reporting limit (RL)	Check all calculations. If no computation errors are found, prepare a fresh ICB and analyze. Sample analysis cannot proceed until a successful ICB is analyzed.
Method Blank (MB)	One per each batch of ≤20 field samples of like matrix; one each time a reagent is changed	Anion content of MB must not exceed analyte reporting limit (RL) Exception: Samples with analyte concentrations >10X amount found in blank may be reported and narrated.	Prepare a fresh MB and analyze to confirm whether or not system contamination is present. If contamination in the MB is still present above the RL, perform system maintenance, analyze a CCV/CCB set, and re-analyze all samples associated with the failed MB that were not reportable.
Laboratory Control Sample (LCS)	One per batch of ≤20 field samples.	Results obtained must agree within ±10% of expected (known) analyte concentration for aqueous samples; within ±15% of known analyte concentration for solid sample extracts	Check calculations and preparation for documentable errors. If no errors are found, reanalyze. Associated samples must also be reanalyzed.
Continuing Calibration Verification (CCV) at or below midpoint	Brackets each set of 10 field sample/QC sample analyses	Analyte concentration must agree within ±5% Method SW9056 and within ±10 % Method 300.0; analyte retention time must agree within ±10%	Rerun CCV. If CCV still not compliant, evaluate/correct instrument malfunction and recalibrate. Samples analyzed after a failed CCV must be reanalyzed. If holding times are an issue, complete

Analytical Method: E 300.0; SW9056	**Parameter:** Determination of Anions by Ion Chromatography		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
			a Non Conformance Report (NCR) and notify the PM for sample disposition.
Continuing Calibration Blank (CCB)	Analyzed immediately following each CCV.	Anion content of CCB must not exceed analyte reporting limit (RL) Exception: Samples with analyte concentrations >10X amount found in blank may be reported and narrated.	Prepare a fresh CCB and analyze. If contamination in the CCB is still present above the RL, perform system maintenance, analyze a CCV/CCB set, and re-analyze all samples associated with the failed CCB that were not reportable.
Matrix Spike (MS) and/or Matrix Spike (Laboratory) Duplicate	One MS/MSD set per batch of 20 field samples (this provides an average frequency of one MS per ten samples per Method 300.0 requirements, and one MS/MSD per batch as required by Method SW9056 and EPA Chapter 1).	Recoveries should meet client criteria for the spiked compounds. RPD for the MS DUP should meet advisory limit of $\leq$20%	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, and associated LCS is within control limits, then sample matrix effects are the most likely cause. Note in narrative. For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers
Method Detection Limit (MDL) Study; run at an analyte concentration near to but lower than the reporting limit (RL)	Per SOP 329	Positive result < the analyte reporting limit (RL)	Determine the reason for failure and correct problem. Repeat the MDL study. Consult the Department / Project / QA Managers. The managers may determine that an adjustment to the RL is needed.

APPENDIX A
EXAMPLE

LOGBOOK/PAGE_____

WETCHEM LAB - CONDUCTIVITY SCREENING WORKSHEET

Instrument ID: HACH HQ440d Multi-Parameter meter S/N 13040087275

Workorder ID / Sample No.		Estimated µS	Dilution(s)	Anion(s)	Date	Initials	Comments

***Dilution factor review between conductivity worksheet and instrument run log**

OBTAIN REVIEW SIGNATURE AS EACH PAGE IS COMPLETED. GIVE TO REPORTING GROUP FOR SCANNING/ARCHIVING AS EACH PAGE IS COMPLETED

Reviewed by / Date _____ **Form 1116r8.doc (01/16/2015)**

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF TOTAL SULFIDES IN WATER
REFERENCED METHOD:	EPA 376.1 AND SM4500 S^2 F
SOP ID:	1120
REV. NUMBER:	7
EFFECTIVE DATE:	11/12/2013

ALS
STANDARD OPERATING PROCEDURE 1120 REVISION 7

TITLE: DETERMINATION OF TOTAL SULFIDES IN WATER -- METHODS EPA 376.1 AND SM4500-S^{2-} F

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- Methods EPA 376.1 and SM4500-S^2 F -- describe a procedure to determine the concentration of total sulfides in drinking, surface, and saline waters and domestic and industrial wastes. Acid soluble sulfides are not measurable by these methods (copper sulfide is the only common sulfide in this class).

Total sulfide includes dissolved H_2S and HS^-, as well as acid-soluble metallic sulfides present in suspended matter. Dissolved sulfide is that remaining after suspended solids have been removed by flocculation and settling. This procedure may also be used for the analysis of dissolved sulfides following proper sample pretreatment (see SM.4500-S^2 B).

Information regarding qualitative testing for sulfides is presented in Section 4 of the introduction to Method 4500-S^2 F. This information may be particularly useful when testing for sulfides in industrial wastes.

2. SUMMARY

Samples are preserved with zinc acetate to yield zinc sulfide. Excess standard iodine is quantitatively added to a sample aliquot. Under acidic conditions, the iodine oxidizes the sulfide in the sample to sulfur. The excess unreacted iodine is then back titrated with standard sodium thiosulfate solution in the presence of starch (used as an indicator).

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful analysis of a proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS's standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file indicate that this review for precision, accuracy, completeness and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/ analyst who performed the work and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

The iodometric method suffers from interferences by reducing substances that react with iodine, including thiosulfate, sulfite, and various volatile organic compounds, both solid and dissolved. Per SM4500-S^2 C, these interferences may be eliminated by first precipitating zinc sulfide, and then removing the supernatant and replacing it with deionized water.

5. APPARATUS AND MATERIALS

5.1 specimen cups, polypropylene, 250mL

5.2 transfer pipets, plastic

5.3 stir plate

5.4 magnetic stir bars

5.5 top loading balance, 0.01g sensitivity **verified per SOP 305**

5.6 pH meter (Accumet 50 or equivalent) or pH test strips capable of reading pH <2

5.7 Eppendorf ™ Pippette or equivalent, adjustable, capable of delivering 0.50- 1.0mL **operated per SOP 321**

6. REAGENTS

NOTES: Only ACS grade or better chemicals may be used.
Reagents may be stored in polypropylene containers.

6.1 Deionized (DI) water. Obtained from the laboratory deionized water system.

6.2 Sulfuric Acid (H_2SO_4) solution, 50% (18N): EMD, SX1244-5 or equivalent. Carefully add 100mL concentrated H_2SO_4 to 100mL deionized water, or different volumes as long as same ratio of 1:1. *Shelf Life = 1 year.*

6.3 Standard Iodine (I2) Solution, 0.0250N: JT Baker, 3162-01 or equivalent. Dissolve 25g of Potassium Iodide (KI) in 800mL of DI water. Add 3.2g of iodine crystals and dissolve. Dilute to 1L with DI water. *Shelf Life = 1 year.* **NOTE:** ***Standardize against $Na_2S_2O_3$ solution daily before use.***

6.4 Starch Indicator, 2%: VWR, Cat. No. VW3638-2 or equivalent. *Shelf-life = per expiration date indicated on label by manufacturer.*

6.5 Hydrochloric Acid (HCl), 6N: JT Baker, 9530-33 or equivalent. Carefully add a measured volume of concentrated HCl to an equal volume of DI water (1:1 dilution). *Shelf Life = 1 year.*

6.6 Standard Potassium Biiodate [$KH(IO_3)_2$] Solution, 0.025N: EM Science, PX1351-2 or equivalent. Dissolve 0.4062g of $KH(IO_3)_2$ salt in 500mL of DI water. *Shelf Life = 1 year.*

6.7 Sodium Thiosulfate ($Na_2S_2O_3$) Solution, approximately 0.025N: EM Science #SX0820-1 or equivalent. Dissolve 3.95g of anhydrous $Na_2S_2O_3$ and 0.4g of NaOH in 1L of DI water. *Shelf Life = 1 year.* **NOTE:** ***Standardize against $KH(IO_3)_2$ once before use.***

6.8 Sulfide (Na_2S) Spiking Solution: Dissolve 15g of Sodium Sulfide ($Na_2S \bullet 9H_2O$) in 200mL of deionized water. *Shelf Life = 1 year.* **NOTE:** ***Standardize daily before use.***

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client request. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA):

7.1 All samples must be collected according to an approved sampling plan. Samples of 500mL or larger may be collected in either plastic or glass containers and should be chilled (4±2°C).

7.2 During collection and prior to preservation and precipitation of the sulfide by zinc acetate, the samples should not be agitated. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

7.3 Aqueous samples must be preserved by adding 2.0mL of 2N Zinc Acetate Solution [$Zn(C_2H_3O_2)_2 \bullet 2\ H_2O$] and 1.5 mL of 20% Sodium Hydroxide Solution (NaOH) per 500mL sample.

7.4 To meet holding time requirements, analysis must be completed within 7 days of collection.

Regulation	Holding Time	Reference
SDWA	7 days	EPA-570/9-82-002
CWA	7 days	CFR 40 Part 136.3

8. STANDARDIZATION OF REAGENTS

8.1 STANDARDIZATION OF SODIUM THIOSULFATE ONCE BEFORE USE AS FOLLOWS:

8.1.1 Transfer 10.00mL standard potassium biiodate [$KH(IO_3)_2$] solution into a 200mL beaker.

8.1.2 Dilute to about 100mL with DI water.

8.1.3 Add approximately 2g of potassium iodide (KI) pellets.

8.1.4 Add 4-5 drops of 50% sulfuric acid (H_2SO_4).

8.1.5 Add 2-3 drops of starch indicator.

8.1.6 Tare the beaker + contents on a top loading balance.

8.1.7 On the stir plate, add sodium thiosulfate solution drop wise until the beaker's contents turns from blue to colorless.

8.1.8 Record the amount of $Na_2S_2O_3$ solution used.

8.1.9 Repeat twice more and average the results of the three replicates. Calculate the concentration of the sodium thiosulfate solution as follows:

$$\text{Sodium thiosulfate conc (eq/L)} = (0.025) * (10.00) / (V)$$

where:

0.025 = normality of potassium biiodate solution titrated

10.00 = volume of potassium biiodate solution titrated

V = volume of sodium thiosulfate solution required to reach endpoint

8.2 STANDARDIZATION OF STANDARD IODINE (I_2) SOLUTION DAILY BEFORE EACH USE AS FOLLOWS:

8.2.1 Add approximately 100mL of DI water to a plastic specimen cup containing a stir bar.

8.2.2 Place on stir plate and add approximately 2.0mL of 6N HCl.

8.2.3 Place on top loading balance and tare container and contents. Add approximately 2.5mL of the iodine standard solution and record the weight to the nearest 0.01g.

8.2.4 Add 2-3 drops of starch indicator and tare container and contents on balance.

8.2.5 Place on a magnetic stir plate and add $Na_2S_2O_3$ solution drop wise until a colorless endpoint is reached.

8.2.6 Place the container and contents back on the balance and record the weight of the titrant used.

8.2.7 Repeat twice more. Calculate the concentration of the standard iodine solution as follows (this calculation can be conveniently carried out using the spreadsheet template located at i:\oprtns\wtchm\sulfide\ template\s1.xls).

$$\text{Iodine conc (eq/L)} = (CTh) * (VTh) / (VI)$$

where:

CTh = concentration (eq/L) of sodium thiosulfate solution

VTh = volume of sodium thiosulfate required to reach endpoint

VI = volume of iodine solution titrated

8.3 STANDARDIZATION OF SULFIDE SPIKING SOLUTION DAILY BEFORE EACH USE AS FOLLOWS:

8.3.1 Add approximately 100mL of DI water to a plastic specimen cup containing a stir bar.

8.3.2 Pipet 0.50mL of the prepared Sulfide Spiking Solution into the container.

8.3.3 Place container on stir plate and add approximately 2.0mL of 6N HCl.

8.3.4 Place container on top loading balance and tare the container and contents.

8.3.5 Add standard iodine solution until a brown color persists (8-15mL). Record standard iodine solution weight to the nearest 0.01g.

8.3.6 Add 2-3 drops of starch indicator and tare container and contents on balance.

8.3.7 Place on a magnetic stir plate and add $Na_2S_2O_3$ solution drop wise until a colorless endpoint (color change from dark blue to clear) is reached.

8.3.8 Weigh the container and contents; record the weight, to the nearest 0.01g, of the titrant used.

8.3.9 Repeat twice more and average the result of the three replicates.

8.3.10 Calculation the concentration of the sulfide spiking solution as follows (the spreadsheet template located at i:\oprtns\wtchm\sulfide\template\s1.xls may be used).

$$\text{mg sulfide / L} = [(A*B)-(C*D)]*16000$$

where:

A = volume (mL) of iodine solution

B = normality of iodine solution

C = volume (mL) of sodium thiosulfate solution

D = normality of sodium thiosulfate solution

16000 = constant which converts meq/mL to mg/L

9. FIELD AND QUALITY CONTROL (QC) SAMLE PREPARATION AND ANALYSIS

9.1 Prepare a Laboratory Control Sample (LCS by transferring about 200mL of DI water each to three plastic cups. Pipet the standardized Sulfide Spiking Solution into each container as needed, usually 0.50 mL.

9.2 Prepare a Method Blank (MB) by transferring approximately 200 mL of DI water to a clean plastic cup.

9.3 Prepare each field sample by mixing the sample thoroughly in the original capped container, then transferring 200mL of the well-mixed sample into a labeled plastic cup. Select a sample representative of the batch and prepare an additional aliquot to serve as the laboratory duplicate. One duplicate sample (DUP) must be prepared for every twenty or fewer environmental samples processed together as a unit.

NOTE: If dissolved sulfides is to be determined, suspended solids must first be removed (see SM4500-S^2 B).

9.4 The sulfide determinations should be performed in the following analytical sequence:

1. MB
2 LCS
3 DUP
4– 20 field samples

If a second batch of samples is to be analyzed, the sequence repeats beginning with a MB, LCS, DUP, and 20 field samples.

9.5 Place each cup on a stir plate and add a stir bar. Add approximately 2.0mL of 6N HCl and check the pH of the solution using a pH test strip. **NOTE:** The pH of the solution must be 2 or less. Add additional 6N HCl as necessary.

9.6 Place the cup on the top loading balance and tare the container and contents.

9.7 Add standard iodine solution to the sample until a brown color persists (approximately 2.5mL if non-detect).

9.8 Weigh the container and contents on the balance. Record the weight of the iodine solution added.

9.9 Add 2-3 drops of starch indicator and tare container and contents on balance.

9.10 Place on stir plate and titrate to a colorless endpoint by adding standardized sodium thiosulfate solution drop wise.

9.11 Place the container and contents back on the balance and record the weight of the titrant used.

9.12 Calculate the amount of sulfide in the field or quality control sample using the spreadsheet template located at i:\oprtns\wtchm\sulfide\template\s1.xls. The template calculates the sulfide concentration based on the following equation:

$$\text{mg sulfide / L} = \frac{[(A * B) - (C * D)] * 16000}{\text{mL sample}}$$

where:

A = volume (mL) of iodine solution
B = normality of iodine solution
C = volume (mL) of sodium thiosulfate solution
D = normality of sodium thiosulfate solution
16000 = constant that converts meq/mL to mg/L

10. QUALITY CONTROL

10.1 DEFINITION OF ANALYSIS BATCH

For this method, an analysis batch is defined as a group of twenty (20) or fewer field samples that is associated with one unique set of batch QC samples and processed

together as a unit. Batch QC samples are defined as the method blank (MB), laboratory duplicate (DUP) and laboratory control sample (LCS). All quality control samples must be carried through all stages of the sample preparation and measurement steps.

10.2 BLANKS

Blanks are run to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Each time a batch of samples is analyzed or there is a change in reagents, a method blank (MB) must be processed. A blank consists of 200mL DI water. See QC Table for acceptance limits.

10.3 LABORATORY CONTROL SAMPLE

The laboratory control sample (LCS) is analyzed to measure the accuracy of the method. It consists of an aliquot of clean matrix (in this instance, 200mL DI water), into which a known amount of analyte is spiked. One LCS must be analyzed per batch of 20 or fewer field samples processed together as a unit.

Results obtained are compared to results expected. The mathematical evaluation is expressed as percent recovery, %R, calculated as follows:

$$\%R = \frac{\text{Concentration}_{\text{Found}}}{\text{Concentration}_{\text{Target}}} \times 100$$

See QC Table for acceptance limits.

10.4 LABORATORY DUPLICATE

A second aliquot of one sample per batch of twenty (20) or fewer field samples processed together as a unit is prepared and analyzed as a laboratory duplicate (DUP). The laboratory duplicate is analyzed as a measure of the precision of the analytical results generated. The duplicate results are compared mathematically (shown below), with the precision expressed as Relative Percent Difference (RPD):

$$\text{RPD (\%)} = \frac{|\ \text{Result}_x - \text{Result}_{\text{Dup}}\ |}{\frac{(\text{Result}_x + \text{Result}_{\text{Dup}})}{2}} \times 100$$

For this procedure, the RPD must not be greater than 20%.

11. DEVIATIONS FROM METHOD

Section 6.0 of Method EPA 376.1 directs that if the sample has been precipitated (i.e., if Zinc Acetate has been added), the reagents are to be added to the original sample container. ALS processes samples by adding reagents to a well-mixed aliquot of sample.

12. SAFETY HAZARDS AND WASTE

12.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

12.2 WASTE DISPOSAL

All wastes are disposed of in accordance with the Waste Management Plan (WPM).

13. REFERENCES

13.1 U.S. Environmental Protection Agency, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, 1983, Method 376.1, "Sulfide, Titrimetric, Iodine".

13.2 A.P.H.A., A.W.W.A. and W.P.C.F., 1989. Standard Methods for the Examination of Water and Wastewater, 20th edition, Pages 4:167, "Method 4500-S2- F, Iodometric Method".

Analytical Method: EPA 376.1; SM4500-S^{2-} F	**Parameter:** Determination of Total Sulfides in Water		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Calibration: Analyte content determined by direct concentration (obtained by calculation from titration). Titrant is standardized each day of use	Daily; each day of use	Titrant must calculate to be to be approximately 0.025N	If titrant does not meet concentration criteria; prepare fresh and standardize.
Laboratory Control Sample (LCS)	One per batch of ≤20 samples	Concentration results obtained must agree between 80% and 120% of expected value	Check calculations and preparation for documentable errors. If no errors are found, reanalyze. Associated samples must also be reanalyzed.
Method Blank (MB),	The MB may be run initially. One per batch of ≤20 samples.	Sulfide content of any blank must not exceed the analyte reporting limit (typically 2mg/L S^2)	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.
Laboratory Duplicate (DUP)	One per batch of ≤20 samples	RPD must be ≤20%	Check all calculations for errors. If no errors are found, discuss with Department/Project/QA Managers.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION AND ANALYSIS OF HEXAVALENT CHROMIUM IN SOLID MATRICES USING ALKALINE DIGESTION
REFERENCED METHOD:	SW3060A AND 7196A
SOP ID:	1121
REV. NUMBER:	8
EFFECTIVE DATE:	NOVEMBER 12, 2013

ALS
STANDARD OPERATING PROCEDURE 1121 REVISION 8

TITLE: DETERMINATION OF HEXAVALENT CHROMIUM IN SOLID MATRICES USING ALKALINE DIGESTION (METHOD SW3060A) AND ANALYSIS BY METHOD SW7196A

FORMS: NONE

APPROVED BY:

TECHNICAL MANAGER _____ DATE _____

QUALITY ASSURANCE MANAGER _____ DATE _____

LABORATORY MANAGER _____ DATE _____

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) and the methods it references -- SW3060A and SW7196A -- describe the alkaline digestion and determination of hexavalent chromium (Cr^{+6}) in soils, sludges, sediments and similar waste materials.

2. SUMMARY

This method uses an alkaline digestion to solubilize both water-insoluble and water soluble Cr^{+6} compounds in solid and waste samples. An aliquot of sample is digested for sixty minutes at 90-95°C in a solution of sodium hydroxide and sodium carbonate. This solubilizes the Cr^{+6} and stabilizes it against reduction to Cr^{+3}.

The Cr^{+6} in the alkaline extract is determined colorimetrically by reaction with diphenylcarbazide (DPC). This reaction is highly selective for Cr^{+6}. Addition of an excess of DPC yields a red-violet complex whose absorbance is measured photometrically at 540nm.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 It is the responsibility of all personnel who work with samples or data involving this method to consult the applicable LIMS Program Specification for client-specific requirements prior to initiating handling of samples or data.

3.3 Analysts must demonstrate the capability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of supervisory/training review, results of precision and accuracy tests performed, or the successful analysis of a proficiency test sample.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file indicate that this review for precision, accuracy, completeness and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving these methods to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 The stability of Cr^{+6} in a solid sample is governed by the oxidizing/reducing tendency (redox potential) of the sample matrix. The redox potential of a particular sample matrix may be sufficiently low to favor the reduction of Cr^{+6} to Cr^{+3}. Under such circumstances, low matrix spike recovery of Cr^{+6} should not be interpreted as a failure of the analysis. Rather, failure to recover spiked Cr^{+6} in a particular sample matrix should be viewed as evidence that Cr^{+6} is not stable in that matrix and, therefore, native Cr^{+6} should not be expected.

4.2 Various organic constituents may be brought into solution when a soil sample is carried through the alkaline digestion procedure, resulting in a yellow to dark brown colored extract. The color can contribute to the absorbance measured on the spectrophotometer and thereby cause a high bias to the results if not accounted for. Furthermore, dissolved organics can interfere with the colorimetric determination of Cr^{+6} with diphenylcarbazide. It may be necessary to dilute the extract in order to alleviate this interference. Color can be accounted for by taking a sample's initial absorbance reading and subtracting it out from the sample's final absorbance reading after the addition of diphenylcarbazide.

4.3 Although not typically found in the alkaline digestates of soils, certain substances may interfere in the analytical methods for Cr^{+6} if the concentrations of these interfering substances are high and the Cr^{+6} concentration is low. Hexavalent molybdenum and mercury salts react to form color with diphenylcarbazide; however, the red-violet intensities produced are much lower than those for chromium at the specified pH. Concentrations of up to 200mg/L of hexavalent molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to 10 times that of chromium will not cause trouble.

4.4 For waste materials or soils containing soluble Cr^{+3} concentrations greater than four times the laboratory Cr^{+6} reporting limit, Cr^{+6} results obtained using this method may be biased high due to method-induced oxidation. The addition of Magnesium (Mg^{2+}) in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation.

5. APPARATUS AND MATERIALS

5.1 UV/VIS Spectrophotometer, Sequoia Turner™ Model 340 or equivalent. Suitable for measurements at 540nm with a light path of 1.0cm or longer.

5.2 analytical balance, capable of weighing to 0.0001g, verified per SOP 305

5.3 laboratory balance, top loading, capable of weighing to 0.1g, verified per SOP 305

5.4 pH meter with electrode and proper calibration buffer solutions

5.5 centrifuge capable of achieving approximately 3500rpm

5.6 Hot block, or water bath, capable of maintaining a temperature of 90-95°C

5.7 vortex mixer

5.8 magnetic stir plate and stir bars

5.9 repeater pipet, variable Eppendorf™ or equivalent

5.10 cuvettes, optically matched, 1-inch diameter

5.11 centrifuge tubes, polypropylene with screw-cap closures, 50mL

5.12 volumetric flasks, Class A, 500mL and 1L sizes

5.13 beakers, polypropylene with snap tight lid, 220mL

5.14 graduated cylinder, 50mL capacity (delivered volume verified by laboratory)

5.15 syringe, outfitted with a 0.45µm or 0.7µm filter disk

5.16 magnetic wand

5.17 thermometer, for checking hot block or water bath temperature

6. REAGENTS

6.1 Deionized (DI) water obtained from the laboratory DI water system

6.2 Ottawa sand, clean/inert, EMD, SX0075-3 or equivalent

6.3 Lead Chromate ($PbCrO_4$), powdered, JT Baker, 0970-01 or equivalent

6.4 pH paper, basic, capable of reading pH 11–13, EM Science, Cat. #9585 or equivalent

6.5 Sulfuric Acid, (H_2SO_4), 10%: Dilute 20mL of 50 % H_2SO_4 (1:1 ratio with DI water) to a final volume of 100mL with DI water. Alternately, dilute 10mL of conc. H_2SO_4 to a final volume of 100mL with DI water. EMD, SX1247-2 or equivalent. ***Store in a polypropylene bottle.*** *Shelf Life =1 year.*

6.6 Acetone, Burdick & Jackson, Cat. 010-4 or equivalent.

6.7 Nitric Acid, (HNO_3), 2N : Dilute 100mL of concentrated HNO_3, JT Baker, 9598-34 or equivalent, to a final volume of 700mL using DI water. ***Store in a polypropylene bottle.*** *Shelf Life = 1 year*

6.8 Diphenylcarbazide (DPC) Solution: Dissolve 0.25g, EM Science, DX2200-1 or equivalent, 1,5-diphenylcarbazide in 50mL of reagent grade acetone. ***Store in a brown glass bottle in the refrigerator.*** Discard when the solution becomes discolored (approx. 3 months).

6.9 Magnesium Chloride ($MgCl_2$) Solution, 2.5M, 51% (w/v): GFS Chemicals, item no. 724 or equivalent. Dissolve 51.0g $MgCl_2 \bullet 6\ H_2O$ in DI water and diluting to 100mL. ***Store in a polypropylene bottle.*** *Shelf Life = 1 year or as indicated by vendor.*

6.10 Potassium Phosphate Buffer: Mallinckrodt, 7100-02 or equivalent. Dissolve 68.0g reagent grade KH_2PO_4 and 87.1g K_2HPO_4 in DI water and dilute to 1L**.** ***Store in a polypropylene bottle.*** *Shelf Life = 1 year.*

6.11 Sodium Hydroxide (NaOH)/Sodium Carbonate ($NaCO_3$) Digestion Reagent: Dissolve 20.0g reagent grade NaOH and 30.0g anhydrous $NaCO_3$ in 1L of DI water. ***The pH must be 11.5 or greater; if not, discard.*** *Shelf Life = 1 month.*

6.12 Cr^{+6} Stock Standard, 500mg/L (**First Source**): Purchase as a certified stock solution from a commercial vendor or prepare by dissolving 0.1414g dry Potassium Chromate ($K_2Cr_2O_7$) in DI water using a 100mL Class A volumetric flask and diluting to full volume with additional DI water. Mix thoroughly. ***Store in a polypropylene bottle and refrigerate.*** *Shelf Life = 1 year.*

6.13 Cr^{+6} Intermediate Standard, 10mg/L (**First Source**): Dilute 2.0mL of first source 500mg/L Cr^{+6} stock standard to 100mL with DI water. ***Store in a polypropylene bottle and refrigerate.*** *Shelf Life = 3 months.*

6.14 Cr^{+6} Stock Standard, 500mg/L (**Second Source**): Purchase as a certified solution from a commercial vendor or prepare by dissolving 0.1414g dry $K_2Cr_2O_7$ in DI water using a 100mL Class A volumetric flask. Bring to volume using DI water, mix thoroughly. If purchased, the certified solution must be from a different vendor than the first source stock standard. If prepared in-house, the $K_2Cr_2O_7$ salt must be from a different vendor, if possible, or at least a different lot number than

the salt used to prepare the first source stock standard. ***Store in a polypropylene bottle and refrigerate.*** *Shelf Life = 1 year.*

6.15 Cr^{+6} Intermediate Standard, 10mg/L (**Second Source**): Dilute 2.0mL of second source 500mg/L Cr^{+6} stock standard to 100mL with DI water. ***Store in a polypropylene bottle and refrigerate.*** *Shelf Life = 3 months.*

7. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

7.1 All samples must be collected according to an approved sampling plan.

7.2 Samples should be collected in containers that do not contain stainless steel.

7.3 Samples should be stored at 4±2°C until analyzed.

7.4 The holding time for hexavalent chromium in solids is 30 days from collection. "Hexavalent chromium has been shown to be quantitatively stable in field-moist soil samples for 30 days from sample collection. In addition, Cr^{+6} has been also been shown to be stable in the alkaline digestate for up to 168 hours (i.e., 7 days) after extraction from soil." (SW3060A, Section 6.4).

8. PROCEDURE

NOTE: A batch is defined as twenty (20) field samples or less of like matrix. One of each of the laboratory quality control (QC) samples outlined below [i.e., Method Blank, Blank Spike/LCS, MS/MSD set (soluble), MS/MSD set (insoluble), and post-digestion spike], must be prepared for each batch of field samples to be analyzed.

8.1 ALKALINE DIGESTION

8.1.1 For each sample, weigh 2.5g of well-mixed moist sample into a 50mL disposable polypropylene centrifuge tube.

8.1.2 Method Blank. Weigh 2.5g clean silica sand into a 50mL disposable polypropylene centrifuge tube.

8.1.3 Laboratory Control Sample (LCS). Weigh 2.5g clean silica sand into a 50mL disposable polypropylene centrifuge tube. Spike with **0.25mL** of 500mg/L Cr^{+6} stock standard (**second source**). **Be certain to have another person witness the spiking.**

8.1.4 Soluble Matrix Spike/Matrix Spike Duplicate (MSs/MSDs). For one of the samples in the batch, spike two duplicate 2.5g aliquots with **0.25mL** of 500mg/L Cr^{+6} stock standard (**first source**). **Be certain to have another person witness the spiking.**

8.1.5 Insoluble Matrix Spike/Matrix Spike Duplicate (MSn/MSDn). For one of the samples in the batch, spike two duplicate aliquots with 10 - 20mg

of powdered $PbCrO_4$. The amount of $PbCrO_4$ added must be weighed on an analytical balance and recorded in the Aqueous Extractions logbook to the nearest 0.0001g. **Be certain to have another person witness the spiking.**

8.1.6 To all of the above, add the following:

- 50mL of Sodium Hydroxide (NaOH)/Sodium Carbonate ($NaCO_3$) Digestion Reagent using a 50mL volume-verified graduated cylinder
- 0.50mL of Potassium Phosphate Buffer Solution
- 0.75mL of 51% $MgCl_2$ Solution

8.1.7 Cap each tube and mix by shaking for at least five minutes. Place each tube into the hot block or water bath, maintained at 90-95°C (check temperature and record in laboratory logbook) and heat for one hour. During this hour, sequentially remove each sample from the heating apparatus , agitate and replace (approximately every 5 minutes). Continue the sequential agitation of the samples for the entire one-hour heating period.

8.1.8 After one hour of heating and agitating, remove the field and QC samples from the heating apparatus and let cool to room temperature. Manually agitate periodically.

8.1.9 Centrifuge for about 15 minutes at about 3500rpm.

8.1.10 Label a 220mL beaker for each field and laboratory QC sample prepared. Place a labeled 220mL polypropylene beaker on a top loading balance and tare the balance. Transfer the appropriate supernatant liquid (from the digestion tube) into the beaker. **Do not tare the balance at this time; it is critical that the balance is not disturbed.** Remove the beaker with contents from the balance and add a stir bar.

8.1.11 Place a calibrated pH probe (see manual for further instruction) into the digestate solution and, while stirring, add 2N HNO_3 drop wise until the pH = 7.5±0.5. **CAUTION: Carbon dioxide will be evolved, perform in fume hood.**

8.1.12 Using a magnetic wand on the outside of the beaker, remove the stir bar. Rinse the stir bar repeatedly with DI water during removal. Place the beaker with contents back on the previously tared balance. Gravimetrically bring the sample solution to a 100mL final volume with DI water (100.0mL = 102.74g).

8.1.13 Attach the beaker's lid and mix well by swirling (careful, the capped beaker will build up pressure slightly).

8.2 COLORIMETRIC ANALYSIS OF STANDARDS AND SAMPLES

8.2.1 STANDARD CURVE PREPARATION

Prepared fresh on each day of analysis. Use DI water to achieve the final volume. Prepare working standards in optically matching 1" spectrophotometer cuvettes as described below:

Standard Concentration (mg/L Cr^{+6})	Volume of 10mg/L Cr^{+6} Intermediate Std (mL)	Final Volume (mL)
0	0	20.0
0.01	0.02	20.0
0.05	0.10	20.0
0.10	0.20	20.0
0.30 (CCV)**	0.60	20.0
0.50	1.00	20.0
0.10 (ICV)*	0.20 *	20.0

* Independent Calibration Verification (ICV). The ICV must be prepared from a second source that is independent of that which is used to prepare the calibration standards. ***Use the Second Source 10mg/L Intermediate Standard.*** An Initial Calibration Blank (ICB), consisting of a 20.0mL aliquot of DI water, must be analyzed immediately following the ICV.

** This standard is also analyzed repeatedly as the Continuing Calibration Verification (CCV). A Continuing Calibration Blank (CCB), consisting of a 20.0mL aliquot of DI water, must be analyzed immediately following each CCV.

8.2.2 POST-DIGESTION SPIKE RECOVERY STUDY

Alkaline soil digestates are often yellow to dark brown in color due to the presence of dissolved organic matter. Dissolved organic matter can interfere with the colorimetric determination of Cr^{+6}. The interference can often be overcome by diluting the digestates prior to analysis.
Note: All samples (including QC samples) are diluted 5X initially.

The level of further dilution necessary to overcome the interference is determined by conducting a Cr^{+6} post-digestion spike recovery study, at various levels of dilution, as follows:

- The alkaline digestate for the sample that was selected as the MS/MSD sample is usually used for the post-digestion spike.

- Transfer 16.0mL of DI water into each of two labeled 1-inch spectrophotometer cuvettes Using a pipet, remove 0.50mL of water from one of the cuvettes and replace it with 0.50mL of 10mg/L intermediate Cr^{+6} standard (first source). **Be certain to have another person witness the spiking.**

- Pipet a 4.0mL aliquot of the selected sample digestate into each cuvette. Adjust to a 20mL final volume using DI water. Mix using the vortex mixer. Note that use of a 4.0mL aliquot of digestate brought to a 20.0mL final volume constitutes a 5-fold dilution.

- To both cuvettes, add 0.5mL of 10% H_2SO_4; mix well.

- Measure the absorbance of both the unspiked and spiked post-digestion aliquots (at 540nm using the spectrophotometer. Record these "initial" readings in LIMS. **These absorbance readings are due to the native color of the sample and will be subtracted from the absorbance readings measured after adding DPC color reagent.**

- Add 0.20mL DPC color reagent to both cuvettes, mix. After allowing about 10 minutes for the reddish color to develop and stabilize, measure the absorbance of both aliquots at 540nm. Record the "final" readings in LIMS.

- An acceptable initial calibration must be generated before the Post-Digestion Recovery Study results can be evaluated (see following Sections). Evaluate the Post-Digestion Recovery Study results as discussed in Section 8.2.4 below.

- If the Post-Digestion Recovery Study results do not meet the 85-115% control criteria, the additional actions that may be taken are contingent upon the analyst's judgment, the particular set of circumstances, and client input.

  Typically the study is repeated on a further diluted aliquot of the same digestate, until satisfactory recovery is achieved. All samples are then diluted at the same level of dilution, based on the successful post-digestion spike study results. The analyte reporting limit for the diluted sample analyses is elevated accordingly.

  Overall though, the objective is that any further actions taken are representative of the client's sample matrix. Consequently, for failures affecting samples in a batch that are from several client sources, several actions may be

necessary. Confer with the Project Manager(s). The appropriate course of action will be developed at the time needed, particular to the circumstance.

8.2.3 STANDARD/SAMPLE DEVELOPMENT

NOTE: **Be sure to properly adjust spectrophotometer.** Insert the proper stray light filter into position. Adjust the wavelength to 540nm. Insert the DI water blank cuvette into the cuvette holder. While in TRANS MODE, press and hold the ZERO SET button while adjusting the ZERO knob until the display indicates 0.00. Set MODE switch to ABS and adjust 100%T/A knobs to exactly 0.000. If further information is required see Barnstead/Thermolyne Corp. Operator's Reference Manual, Model 340 Digital Spectrophotometer.

- Transfer a 4.0mL aliquot (or smaller volume if it was determined that a greater dilution is necessary) of each digestate into a 1-inch diameter spectrophotometer cuvette. Bring to a 20.0mL final volume using DI water. Use of a 4.0mL aliquot of digestate brought to a 20.0mL final volume represents a 5-fold dilution.

- Add 0.5mL of 10% H_2SO_4 to each cuvette and mix using the vortex mixer. Measure the absorbance of the samples at 540nm using the spectrophotometer. Record these "initial" readings in LIMS. This absorbance reading is due to the native color of the sample and will be subtracted from the absorbance reading measured after adding DPC color reagent.

- Add 0.4mL DPC solution to the cuvettes and vortex.

- Allow the cuvettes to stand until a stable reddish color has developed (about 10 minutes).

8.2.4 ANALYTICAL SEQUENCE

Use the spectrophotometer (set at 540nm) to read the absorbance of each of the cuvette's contents. Record each reading. The following analytical sequence must be observed:

- Absorbance measurements must begin with the calibration standards, immediately followed by the ICV/ICB.

- Analyze the Post-Digestion Study samples. The recovery quality control limits for these samples is 85-115 %. If the

Post-Digestion Recovery Study results exceed these limits, address as discussed in Section 8.2.2.

- After the ICV and ICB have been analyzed, **up to ten** samples (i.e., Method Blank, BS/LCS, post-digestion spike study, field samples, etc.) can be analyzed. Prepare digestates (or dilutions thereof), develop and analyze as previously discussed. Note: Insoluble MS/MSD's should require 100X dilution.

- ***The continuing calibration verification (CCV) standard, followed by the Method Blank as the Continuing Calibration Blank (CCB), must be analyzed between each set of ten samples, and at the close of the analytical sequence.***

  NOTE: To be acceptable, no blank may yield results greater than the analyte reporting limit.

- Continue to analyze samples in sets of ten or less followed by the analysis of the CCV/CCB pair until all samples have been analyzed.

8.3 EVALUATION OF THE CALIBRATION CURVE

8.3.1 Prepare a standard curve by plotting the observed absorbance versus the standard concentration for each calibration standard. Enter the information into the LIMS system.

8.3.2 Enter the absorbance data obtained for each solution analyzed (calibration standards, QC samples and field samples). Also, enter additional data as necessary (e.g., identification, sample volumes or weights, dilutions, percent solids, etc.) This information is also entered into the LIMS system.

8.3.3 Using LIMS evaluate the linear regression analysis between absorbance and Cr^{+6} concentrations for the calibration standards. The correlation coefficient (r^2) for the regression must be $\geq$0.995.

8.3.4 Evaluate the ICV sample. The ICV result must agree within $\pm$10 % of the expected value.

8.3.5 Evaluate the ICB sample result (result must be less than analyte reporting limit).

8.3.6 Evaluate the CCV results. The CCV result must agree within $\pm$10% of the expected value.

8.3.7 Evaluate the CCB results (results must be less than analyte reporting limit).

NOTE: *Acceptance criteria and corrective actions are summarized in the attached QC Summary Table.*

8.4 CALCULATION OF SAMPLE CONCENTRATION

The Cr^{+6} concentration in the samples is calculated as follows:

$$Cr^{+6}\ \text{Concentration (mg/Kg)} = \frac{(C * 100 * 20)}{(A * 2.5 * S)}$$

where:

C	=	Concentration of Cr^{+6} (mg/L) in cuvette
A	=	Aliquot (mL) of digestate pipetted into the cuvette
20	=	final volume (mL) of solution in cuvette
100	=	final volume (mL) of digestate
2.5	=	moist weight (g) of soil digested
S	=	ratio of solids in sample (% solids/100)

8.5 CALCULATION OF POST-DIGESTION SPIKE RECOVERY

8.5.1 For both aliquots, subtract the "initial" absorbance reading from the "final" absorbance reading to obtain a "corrected" absorbance reading.

8.5.2 Enter the corrected absorbance into the spreadsheet to calculate the solution Cr^{+6} concentration.

8.5.3 Calculate the post-digestion spike recovery as follows:

$$\text{Post-Digestion Spike (\% R)} = \frac{(S - N)}{0.25} * 100$$

where:

S	=	Cr^{+6} concentration (mg/L) in spiked aliquot
N	=	Cr^{+6} concentration (mg/L) in unspiked aliquot
0.25	=	amount of Cr^{+6} spike added (mg/L); 0.25 if this SOP is followed without deviation

8.5.4 The Post-Digestion Spike recovery acceptance limits are 85-115%. If the recovery falls outside of this range, the digestate post-digestion spike recovery study must be repeated at greater levels of dilution until acceptable recovery is achieved. Results of the spike recovery study are extrapolated to determine appropriate levels of dilution for the other samples in the prep batch. In other words, the other samples in the

batch must be diluted such that the "initial" absorbance reading (i.e., the absorbance measured before adding DPC reagent), is not greater than the initial absorbance reading for the post-digestion spike sample at the level of dilution that was necessary for acceptable recovery.

9. QUALITY CONTROL (QC)

NOTE: *Acceptance criteria and corrective actions are detailed in the attached QC Summary Table.*

9.1 BLANKS

Method blanks (MBs) are run to demonstrate that interferences from the analytical system, glassware, and reagents are under control. For this procedure, the MB consists of clean silica sand that has been digested as described previously in this SOP. The blank must not yield positive results greater than the reporting limit (usually 2.0mg/Kg Cr^{+6}).

9.2 SPIKED SAMPLES

The nature and evaluation of Post-Digestion Spike samples are discussed in previous Sections.

9.2.1 Blank/Laboratory Control Spike (LCS) samples consist of clean matrices which are spiked with a known amount of target compound. For this SOP, silica sand is used as the clean matrix. LCS analyses are performed to evaluate the accuracy of the analytical system.

9.2.2 For this SOP, the matrix spiked samples (MSs/MSDs and MSn/MSDn) consist of aliquots of selected field sample into which known concentrations of target analytes are spiked and analyzed as a means of determining the effect of matrix on target analyte detection. The spiked sample analyses are performed to determine the effect of sample matrix interferences.

9.2.3 EVALUATION OF SPIKED SAMPLE RESULTS

Spiked samples are evaluated based on percent recovery (%R), which is a calculation of the amount of target yielded vs. the amount of target anticipated. The following equation is used to calculate spike recovery:

$$\%R = \frac{Concentration_{Found} - Concentration_{Aliquot}}{Concentration_{Anticipated}} \times 100$$

where:

$Conc_{Found}$ = amount of target yielded by the spiked analysis

$Conc_{Aliquot}$ = amount of target found in the unspiked aliquot ("zero" for clean LCS matrix aliquots; sample results for sample matrix aliquots)

$Conc_{Anticipated}$ = amount of target expected to be yielded based on amount spiked

The control limits established for the LCS analysis are 80-120 %.

Advisory limits for the MS/MSD analysis are set at 75-125 % of the anticipated value.

9.2.4 DUPLICATE PRECISION

The matrix spiked samples are prepared and analyzed in duplicate to measure analytical precision. Precision is evaluated in terms of Relative Percent Difference (RPD), and is calculated as follows:

$$RPD\ (\%) = \frac{|Result_x - Result_{Dup}|}{\frac{(Result_x + Result_{Dup})}{2}} \times 100$$

An RPD of $\leq$20 is set as the advisory quality control limit.

9.3 METHOD DETECTION LIMIT STUDY

A method detection limit (MDL) study shall consist of the analysis of a blank and a minimum of seven replicate analyses at a concentration level near to the capabilities of the method and below the analyte reporting limit (RL). The MDL study should be performed as needed and at a minimum, annually.

10. DEVIATIONS FROM METHOD

10.1 According to Section 8.5 of Method SW3060A, "An acceptance range for matrix spike recoveries is 75-125 %. If the matrix spikes are not within these recovery limits, the entire batch must be rehomogenized/redigested/reanalyzed." This SOP does not require redigestion of samples when matrix spike recoveries are outside of the 75-125 % limits. Rather, recoveries outside these limits are attributed to matrix effects and the results are narrated and flagged in the data report accordingly (see also comments in Interferences Section).

10.2 Method SW3060A suggests the need for sample characterization and suggests "additional analytical parameters" such as pH, ferrous iron, sulfides, oxidation-reduction potential (ORP), total organic carbon (TOC), biological oxygen demand (BOD) and chemical oxygen demand (COD). ALS considers these additional analyses to be beyond the scope of this analysis.

10.3 Unlike Method SW3060A, Section 8.4, this SOP does not prescribe that a sample duplicate be prepared and analyzed. In this SOP, a measure of analytical precision is provided by the RPD calculated for the soluble MS/MSD.

10.4 Section 8.6.2 of Method SW3060A prescribes that if the post-digestion spike recovery is outside of 85-115 %, either the Method of Standard Additions (MSA) is used or that additional analytical parameters should be considered in order to

interpret the results. In this SOP, when a post-digestion spike is outside of 85-115% recovery, the spike is repeated on the diluted sample aliquot of the selected sample digestate. The level of dilution must be increased until satisfactory recovery is achieved. Furthermore, the other samples in the batch must be diluted according to the results from the post-digestion spike recovery study. Other corrective measures may be applied contingent upon the particular circumstance and client input.

10.5 Section 5.2 of Method SW7196A describes the creation of the chromium stock solution as dissolving 0.1414g $K_2Cr_2O_7$ in DI water and bringing the solution to a 1L final volume. ALS creates a 10-times stronger chromium stock standard by dissolving 0.1414g $K_2Cr_2O_7$ in DI water and bringing the solution to a 100mL final volume. Furthermore, Section 5.3 of Method SW7196A describes the creation of the standard chromium solution by diluting 10.0mL of chromium stock solution to a 100mL final volume. ALS creates the standard chromium solution by diluting 2.0mL of the chromium stock solution to a 100mL final volume. Therefore, at 10mg/L Cr^{+6}, ALS's standard chromium solution is 2-times stronger than that provided for in the Method.

10.6 Section 7.1 of Method SW7196A prescribes that the colorimetric analysis of calibration standards and samples be performed in 100mL volumetric flasks (standards and samples are brought to 100mL using DI water). ALS uses a 20.0mL final sample volume and conducts the color development directly in the 1-inch diameter spectrophotometer cuvettes.

10.7 In Section 7.2.1, Method SW7196A indicates that the method is suitable for the analysis of hexavalent chromium ranging in concentration from 0.5 to 5mg/L Cr^{+6}. ALS's modifications to the Method (e.g., the use of more concentrated chromium standards), enables a typical calibration range of 0.010 to 0.50mg/L Cr^{+6} (2.0-100mg/Kg Cr^{+6}, correspondingly) to be achieved.

10.8 In Section 7.3.1, Method SW7196A indicates that the spike added concentration for the interference check standard should not be less than 30mg/L Cr^{+6}. This SOP prescribes a spike added concentration of 0.25mg/L Cr^{+6}, which is appropriate for the analytical range of ALS's analysis.

10.9 Based on sample volumes and reagent strengths cited in the Methods, Section 4e of Method SM3500-Cr B and Section 7.1 of Method SW7196A prescribe the addition of 2.0mL diphenylcarbazide to the 100mL final sample volume for color development. In accordance with reagent strengths, the 20.0mL final sample volume employed, and color development carried-out directly in the spectrophotometer cuvettes (all discussed above), ALS uses 0.20mL of diphenylcarbazide to achieve color development.

10.10 Section 8.5 of Method SW7196A calls for the analysis of one matrix spike replicate OR one replicate sample for every ten samples. ALS accomplishes this

by preparing and analyzing one matrix spike/matrix spike duplicate per batch of 20 samples.

10.11 Section 7.1 of Method SW7196A discusses correcting sample absorbance by subtracting out the absorbance of the reagent water blank. ALS does not correct absorbance readings using the reagent water blank because a 0.0 standard (consisting of reagent water) is incorporated as part of the calibration curve.

10.12 Section 7.1 of Method SW3060A suggests the use of a temperature blank to monitor the inferred temperature of the alkaline digestates while housed in the 90-95°C hot block or water bath. ALS does not employ a temperature blank but directly monitors the hot block or water bath with a NIST-traceable verified glass thermometer.

10.13 Section 7.6 of Method SW3060A discusses filtration of the alkaline digestates. ALS spins the digestates in a centrifuge to achieve effective control of solids interferences.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 3rd Edition, Final Update III, Chapter 3, Method 3060A, “Alkaline Digestion for Hexavalent Chromium”, Revision 1, December 1996.

12.2 US EPA SW-846, Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, 3rd Edition, Final Update III, Chapter 4, Method 7196A, “Hexavalent Chromium (Colorimetric)”, Revision 1, July 1992.

Analytical Method: SW3060A & 7196A	**Parameter:** Alkaline Digestion and Analysis of Hexavalent Chromium (Colorimetric) in Solid Matrices		**Summary of Quality Control Procedures and Corrective Actions**
QC Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point (plus blank)	As needed (i.e., at on-set of analyses or when continuing calibration does not meet criteria)	Correlation coefficient (r^2) for linear regression must be $\geq$0.995	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Independent Calibration Verification (ICV); second source; at or below the midpoint	Once after each initial calibration	Response must agree within $\pm$10% of initial calibration	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Continuing Calibration Verification (CCV); at or below the midpoint	Run after every ten samples and to end any run sequence (must be followed by a CCB analysis)	Response must agree within $\pm$10% of initial calibration	Check that calculations and preparation are correct, evaluate/correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.
Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	ICB is run following the ICV; CCB is run after each CCV and to close a run sequence	Cr^{+6} content of the blank must be < RL; RL usually 2.0mg/Kg Cr^{+6}(which corresponds to 0.01mg/L without conversion to solid matrix units)	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.
Method Blank (MB)	One per batch of $\leq$20 field samples	Cr^{+6} content of the blank must be < RL; RL usually 2.0mg/Kg Cr^{+6}	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples must also be reprepared and analyzed.
Blank (laboratory control) Spike (BS, LCS)	One per batch of $\leq$20 field samples	Recoveries must be within $\pm$20% of expected values	Check for documentable errors (e.g., calculations and spike preparation). If no computation errors are found, prepare a fresh spike and analyze. If quality criteria still not met, all field and quality control samples must be reprepared and analyzed.
Matrix Spike (MSs or MS)	One per batch of $\leq$20 field samples	Recoveries should be within $\pm$25 % of expected values	Check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike (laboratory) Duplicate (MSDs or MSDn)	Matrix-specific; one prepared for each sample batch of $\leq$20 field samples	(See MS recovery criteria above). RPD should be $\leq$20.	(See MS recovery criteria above). For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department and QA Managers.
Method Detection Limit (MDL) Study; run at an analyte concentration near to but lower than the reporting limit (RL)	As needed; at a minimum annually.	Positive result < the analyte reporting limit (RL).	Determine the reason for failure and fix problem with the system. Repeat the MDL study. If criteria still not met, discuss with QA Manager (RL may be adjusted if required).

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATIN OF PH BY ELECTROMERIC MEASURESMENT
REFERENCED METHOD:	EPA 150.1, SW9040C, SW9045D, AND SM4500-H+B
SOP ID:	1126
REV. NUMBER:	18
EFFECTIVE DATE:	11/12/2013_____

ALS
STANDARD OPERATING PROCEDURE 1126 REVISION 18

TITLE: **DETERMINATION OF pH BY ELECTROMETRIC MEASUREMENT METHODS EPA 150.1, SW9040C, SW9045D, AND SM4500-H^+ B**

FORMS: **NONE**

APPROVED BY:
TECHNICAL MANAGER _____ DATE _____
QUALITY ASSURANCE MANAGER _____ DATE _11/12/2013_____
LABORATORY MANAGER _____ DATE __11/13/2013___

1. SCOPE AND APPLICATION

This standard operating procedure (SOP) and the methods it references -- EPA Method 150.1, SW9040C (aqueous), SW9045D (soils, sludges and wastes), and SM4500-H^+ B -- describes the electrometric measurement of pH in environmental matrices. Some water content is required to conduct the pH measurement. Method SW9040C is used to measure the pH of single or multiphase matrices whose aqueous phase constitutes at least 20% of the total volume of the sample matrix. Method SW9045D is used to measure the pH of solid, sludge, or non-aqueous liquid matrices, whose water content, if present at all, constitutes less than 20% of the total volume of the sample matrix. The corrosivity of concentrated acids and bases, or concentrated acids and bases mixed with inert substance, cannot be measured using this SOP.

2. SUMMARY

The basic principle of electrometric pH measurement is to determine of the activity of the hydrogen ion [H+] by potentiometric measurement using standard buffers and a reference electrode. ALS uses a pH meter equipped with a combination pH electrode to measure pH. The pH is measured directly in water samples (while stirring). The pH of soils and wastes is measured by mixing with deionized water (1:1 ratio) and measuring the pH of the aqueous suspension. The pH-unit measuring device is calibrated with a series of standard buffer solutions of known pH at a temperature of 25±2°C.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the capability to generate and interpret results acceptably to utilize this method. Demonstration of performance may include Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of an unknown proficiency test sample.

3.3 ALS's LIMS program specification system and associated project analyte nicknames are the means by which client-specific requirements for sample preparation, analysis, data evaluation and reporting are communicated to the laboratory. This system includes automated electronic controls where possible. The criteria defined in the program specification supercede ALS standard criteria. It is the responsibility of all personnel who work with samples or data involving this method, to consult the applicable LIMS program specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file documentation indicates that this review for precision, accuracy, completeness, and reasonableness is complete and satisfactory. Any errors that are found require corrective action, which includes notifying the technician/analyst who performed the work of the errors and documentation of the measures taken to correct those errors.

3.5 It is the responsibility of all personnel who work with samples involving this method to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 In general, the glass electrode is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or moderate salinity (<0.1 molar salinity).

4.2 The sodium ion is the most likely interference for the pH electrode. The electrode used in this SOP is constructed from a special low sodium error glass. Error due to sodium is negligible when measuring pH values less than 12.

4.3 pH measurements are affected by temperature in two ways (1) mechanical effects that are caused by changes in the properties of the electrode, and (2) chemical effects caused by equilibrium changes. Temperature difference between calibration buffers and samples could cause errors. If the sample temperature differs by more than ±2°C from the buffers, it is necessary to make corrections for temperature. In order to overcome these temperature differences, all standards (buffers) and samples are placed in a water bath maintained at 25±2°C until equilibrated (approximately 1 hour).

4.4 Errors will occur if the electrode becomes coated. If the electrode becomes coated with an oily material, rinse the electrode with mild detergent or methanol. If inorganic deposits are coating the electrode, soak the electrode in 0.1M HCl for one-half hour, followed by soaking in the electrode storage solution for at least one hour. Refer to the electrode's instruction manual for additional information on cleaning the electrode.

4.5 The electrode junction should be flushed by holding the probe and pressing down on the top after use in especially dirty or viscous samples or when the electrode response becomes sluggish. After flushing the junction, the probe should be filled with internal filling solution.

5. APPARATUS AND MATERIALS

5.1 pH meter, Fisher Accumet 50 or equivalent capable of reading two decimal places

5.2 Combination pH electrode, Orion ROSS-SURE FLOW or equivalent with internal filling solution

5.3 Centrifuge tubes, 50mL, plastic, disposable

5.4 Magnetic stir plate and Teflon™-coated stir bars

5.5 Laboratory balance, capable of weighing ±0.01g

5.6 Water bath maintained at 25±2°C

6. REAGENTS AND STANDARDS

NOTE: Only reagent grade chemicals and deionized (DI) water shall be used in all pH tests.

6.1 Standard buffer solutions: Certified; purchased from vendors. These commercially available solutions must be validated by comparison with NIST standards. These buffer solutions may be purchased at various pH units (e.g., 4, 5, 7, 9, and 10). For the procedures described in this SOP, solutions at 2.00, 4.00, 7.00, 10.00, 12.45 pH units are used. A second source (at a pH of 7.00) buffer is also required. *Shelf Life* = per manufacturer's expiration date as long as condition is good.

6.2 Electrode internal filling solution: Use only Orion ROSS Reference Electrode Filling Solution 3M KCl Cat. No. 810007 with the ROSS SURE-FLOW pH electrode. *Shelf Life* = one year after date of opening.

6.3 Electrode storage solution: Add 0.25g of potassium chloride (KCl) to 50mL of pH 7 buffer solution. *Shelf Life* = replenish as needed to maintain sufficient volume.

6.4 Hydrochloric Acid (HCl), 0.1M: Dilute 0.83mL concentrated HCl to 100mL using deionized water. *Shelf Life* = one year.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

7.1 All samples should be collected according to an approved sampling plan.

7.2 The measurement of pH is intended to be a field parameter, as the buffering capacity of a sample may change the pH of the sample. EPA Methods 150.1 and SW-846 9040C and 9045D state that "samples should be analyzed as soon as possible." ALS strives to analyze all pH samples as soon as possible after receipt; prior notification

and/or special arrangements for Saturday sample receipt and analysis are encouraged between the client and the laboratory. However, the intention of this 4 day window is to accommodate the scenario of Friday sample collection, followed by login and release of the sample to the laboratory for analysis the following Monday, with the laboratory performing the analysis within 4 days of internal receipt.

7.3 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere. Therefore, the sample containers should be filled completely and kept sealed prior to analysis.

7.4 Samples must be refrigerated at 4±2°C until analysis.

8. PROCEDURE

8.1 pH METER STANDARDIZATION, GENERAL OPERATION AND ELECTRODE CARE

Over time, a pH electrode's slope and its zero potential will change. ***Therefore, the pH electrode must be standardized each day before use.*** Standardization requires the use of at least three certified buffer solutions. ALS uses 4.00, 7.00 and 10.00 buffer solutions to standardize the pH meter. For corrosivity characterization, and if the sample pH is <4 or >10 pH units, the calibration of the pH meter should include a buffer of pH 2.00 for acidic wastes and pH 12.45 for caustic wastes. The procedure for standardizing the pH meter follows.

8.1.1 If not already prepared, turn on the water bath and set to maintain a temperature of 25±2°C.

8.1.2 To clear the existing values from the pH meter's previous standardization, press "channel" on the main screen until the display indicates the current pH channel (channel B). Then press "pH" to select pH mode. Next press "standardize". A menu of standardization options will appear. Press "2" to clear the existing standards. The pH meter will return to the main screen (all pH standardization points have been cleared from the memory).

8.1.3 Check the pH electrode's internal filling solution level. The level of the filling solution must cover the coil and be at least one inch above the sample level. Uncover the filling hole and add more internal filling solution if needed. The filling hole must remain uncovered during analysis.

8.1.4 Place fresh aliquots (approximately 20mL) of the pH 4.00, 7.00 and 10.00 buffers in 50mL centrifuge tubes and cap the tubes. Record the IDs of the buffers on the bench sheet. Place all buffers into the water bath and allow to equilibrate for at least 1 hour.(Always 3 points)

NOTE: If the sample pH is <4 or >10 pH units, the meter must be re-calibrated using the 2.00and/or 12.45 pH buffer solutions on either the high or low end.

NOTE: The pH buffer aliquots dispensed into the centrifuge tubes are useable only for that day's calibration. These buffer aliquots must be disposed of daily.

8.1.5 pH meter standardization can begin once the pH buffers have come to temperature in the water bath. Press "channel" on the pH meter's screen until the display indicates the current pH channel (channel B). Then press "pH" to select pH measurement. Make sure the filling hole of the pH electrode is uncovered.

8.1.6 Remove the centrifuge tube containing the first source pH 7.00 buffer from the water bath and record the temperature of the water bath on the bench sheet. Uncap the centrifuge tube, add a stir bar, and stir gently on the stir plate.

NOTE: All aqueous samples and buffer standards must be stirred while taking pH measurements. Place an insulation pad on top of the stir plate to prevent error from the transfer of heat to the solution being measured.

8.1.7 Place the pH electrode in the pH 7.00 buffer solution. Allow the pH meter's display to stabilize (i.e., no change in pH reading for at least ten seconds).

8.1.8 Press "standardize" (a menu of standardization options is displayed). Press "1" to select "Update or Add a Standard". Enter the value of the pH buffer being used for standardization, then press "enter". The screen will display on-screen instructions, press "enter". The pH meter will return to the main screen with the added buffer point shown. Record the buffer point on the bench sheet. If the buffer tube is to be re-read later in the run as a continuing calibration verification (CCV), it must be placed back into the water bath immediately to maintain the proper temperature.

NOTE: The pH electrode must be rinsed thoroughly between measured solutions using deionized water.

8.1.9 With the pH meter in measurement mode, repeat Steps 8.1.6 through 8.1.8 for the pH 4.00 and pH 10.00 buffers to complete the three-point standardization. **If the sample pH is <4 or >10 pH units, the meter must be re-calibrated using the 2.00, and/or 12.45 pH buffer solutions, on either the high or low end.**

8.1.10 Additional pH electrode considerations:

- Rinse the pH electrode with DI water between each measured solution. Avoid rubbing or vigorous wiping of the glass electrode to reduce the chance of error due to polarization.

- After analyzing dirty or viscous samples, or when electrode response becomes sluggish, it may be necessary to empty the electrode completely and hold the junction open under running deionized water. Empty any water from the electrode and refill with fresh ROSS filling solution. Flush the electrode by pushing down on the top briefly, check the internal filling solution level again and add more if necessary.
- Alternately, the general cleaning procedure can be performed. Soak the electrode in 0.1M HCl for thirty minutes, followed by soaking in the pH storage solution for at least one hour.
- Store the pH electrode by immersing in the storage solution. The filling hole is covered during storage and the meter is placed in standby mode.

8.2 FIELD AND QC SAMPLE PREPARATION AND ANALYSIS

NOTE: All samples and buffer solutions, including the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV), must be placed in the water bath for at least 1 hour prior to pH measurement.

8.2.1 Prepare the ICV by transferring an aliquot (approximately 20mL) of second-source pH 7.00 buffer to a 50mL centrifuge tube. Place the centrifuge tube in the water bath and allow to equilibrate for at least one hour.

8.2.2 The previously prepared first-source pH 7.00 buffer aliquot can be used as the CCV. Keep the centrifuge tube in the water bath to maintain the proper temperature.

8.2.3 Label a series of 50mL centrifuge tubes with the identities of the samples to be analyzed (or mark otherwise).

8.2.4 For each aqueous sample, transfer a 20mL aliquot into a designated centrifuge tube, cap and place the tubes in the water bath. Prepare one out of every ten field samples in duplicate.

8.2.5 For each solid matrix sample to be analyzed, place a 20g aliquot of sample into a 50mL centrifuge tube and add 20mL of DI water. Record the sample weight and DI water volume on the bench sheet. Cap the tubes and shake the mixtures manually for five minutes. Additional DI water may be added if the sample readily absorbs water and no free aqueous phase is present. Place the prepared tubes in the water bath. Prepare one out of every ten field samples in duplicate.

NOTE: Analysis can proceed once the prepared solutions have been allowed to equilibrate in the water bath for at least one hour.

This time period also allows the solids to settle for the solid matrix samples being analyzed.

8.2.6 When analysis is ready to proceed, press the "pH" and "meas/monitor" keys to place the pH meter in continuous pH mode. **Make sure the filling hole of the pH electrode is uncovered.**

8.2.7 **The ICV must be analyzed following standardization of the pH meter before any samples can be analyzed.** Remove the ICV from the water bath and record the temperature of the water bath on the bench sheet. Uncap the tube and add a stir bar. Stir gently on the stir plate. Use an insulation pad to prevent the transfer of heat to the measured solution. Immerse the pH electrode into the ICV.

8.2.8 Wait until a stable reading is achieved (i.e., no change in reading for at least ten seconds). Record the ICV's pH value on the bench sheet.

The ICV is run to verify the pH meter's initial standardization. The ICV result must agree within ±0.05 pH units of the known value (i.e., pH 7.00) before sample analyses can proceed. If the ICV fails, check the integrity of the second source buffer. Replace the ICV and reanalyze. If the ICV still fails, the pH meter must be restandardized.

8.2.9 Following an acceptable ICV analysis, up to 10 samples (including a Duplicate) can be analyzed, followed by a CCV. The CCV must be analyzed to bracket the first ten sample analyses between the ICV and CCV. Following the CCV, up to 10 more samples can be analyzed, followed by the CCV again. The sequence of up to 10 sample analyses followed by the CCV can be continued until all of the samples have been analyzed. Put the CCV back into the water bath after each reading to maintain it at the proper temperature.

If any of the CCV results fails to agree within ±0.1 pH units from the expected value, the pH meter must be restandardized and all samples run after the last acceptable CCV analysis must be reanalyzed.

8.2.10 For aqueous samples, uncap the tube and add a stir bar. Stir gently on the stir plate. Use an insulation pad to prevent the transfer of heat to the measured solution. Immerse the pH electrode into the sample solution. Wait until a stable reading is achieved then record the sample's pH on the bench sheet. Report the results as "pH in water at 25°C."

8.2.11 Do not add a stir bar to the centrifuge tubes containing the prepared solid matrix samples. For solid sample analysis, immerse the pH electrode just deep enough into the aqueous phase to make electrical contact; do not push the electrode into the settled solids. Wait until a stable reading is

achieved, then record the sample's pH on the bench sheet. Report solid results as "solid pH in water at 25°C."

8.2.12 A laboratory duplicate is run for every ten environmental samples analyzed. The pH results for aqueous sample duplicates should agree within ±0.2 pH units of each other. The pH results for solid sample duplicates should agree within ±0.5 pH units of each other. If these criteria are exceeded, consideration will be given to the CCV standard performance. If no problems occurred with the CCV analyses, the laboratory will remark in the case narrative that the duplicate precision criteria have been exceeded, and will report the analysis results as is.

9. QUALITY CONTROL

Calibration checks and duplicate analyses are discussed in PROCEDURE above.

10. DEVIATIONS FROM METHOD

10.1 ALS does not employ a temperature sensor to provide temperature compensation control at the pH meter. Instead, temperature compensation is facilitated by the use of a water bath maintained at 25±2°C. This approach allows for a consistent temperature between calibration buffers and samples without the need for additional calculations due to temperature differences.

10.2 Method SW9040C, Section 7.4 and Method EPA 150.1, Section 8.4 states that successive aliquots be measured (usually 3) until the difference is less than 0.1 pH units. ALS does not employ this practice, but runs a duplicate sample per 10 environmental samples.

10.3 Method SW9040C, Section 7.1.2 states that for corrosivity characterization, the sample must be measured at 25±1°C if the pH is above 12.0. ALS maintains the water bath at 25±2°C.

10.4 Method SW9040C, Section 7.1.2 states that for corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes. ALS uses a pH 12.45 buffer for caustic wastes.

11. SAFETY, HAZARDS AND WASTE DISPOSAL

11.1 SAFETY AND HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

11.2

11.3 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

12. REFERENCES

12.1 U.S. Environmental Protection Agency, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, 1971. Method 150.1, "pH (Electrometric)".

12.2 U.S. Environmental Protection Agency, SW846, Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, Revision 2, January 1995. Method 9040C, "pH Electrometric Measurement".

12.3 U.S. Environmental Protection Agency, SW846, Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, Revision 3, January 1995. Method 9045D, "Soil and Waste pH".

12.4 A.P.H.A., A.W.W.A. and W.P.C.F., 1989. Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998. Method 4500-H^+ B, "(pH) Electrometric Method", pp 4-87 to 4-91.

12.5 Orion ROSS SURE-FLOW pH electrode instruction manual (227355-001 Rev.C).

12.6 Fisher Model 50 Accumet pH meter operating instructions (Rev. C 2/92).

Analytical Method: EPA 150.1, SW9040C, SW9045D, SM4500-H^+ B	**Parameter:** pH		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
pH Meter Standardization	Must be performed each day pH meter is used.	N/A	The pH meter must be successfully standardized before sample analyses can proceed.
Initial Calibration Verification (ICV)	Run following the initial standardization, before any samples are analyzed.	ICV results must agree within ±0.05 pH units of the known value.	If ICV fails, check the integrity of the second-source buffer solution. Replace and reanalyze. If ICV still fails, problem must be identified and corrected and pH meter restandardized before analyses can proceed.
Continuing Calibration Verification (CCV)	Run following the analyses of every ten sample set and to close-out the run sequence.	CCV results must agree within ±0.1 pH units of the expected value.	If CCV fails, pH meter must be restandardized and all samples run since the last acceptable CCV must be reanalyzed.
Laboratory Duplicate (DUP)	One per batch of ≤10 field samples.	Aqueous duplicate values should agree within ±0.2 pH units of each other; solid matrix duplicate values should agree within ±0.5 pH units of each other.	If criteria are not met and CCV analyses are within QC limits, narrate client data package report.

ALS
STANDARD OPERATING PROCEDURE 1127 REVISION 9

TITLE: DETERMINATION OF NITROGEN AS NITRATE PLUS NITRITE (NO_3^--N + NO_2^--N), NITRITE (NO_2^--N), AND NITRATE (NO_3^--N) IN ENVIRONMENTAL WATER AND SOIL SAMPLES USING A COLORIMETRIC, AUTOMATED, CADMIUM REDUCTION PROCEDURE -- METHODS EPA 353.2, SM 4500-NO_3^- I, AND QUIKCHEM 10-107-04-1-C

FORMS: NONE

APPROVED BY:

PRIMARY AUTHOR _____ DATE 4/11/13

QUALITY ASSURANCE MANAGER _____ DATE 4/11/13

LABORATORY MANAGER _____ DATE 4/11/13

1. SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) references EPA Method 353.2, Standard Method (SM) 4500-NO3- I, and Quikchem Method 10-107-04-1-C for the determination of nitrate + nitrite (NO_3^--N + NO_2^--N) and nitrite only (NO_2^--N) in environmental water and soil samples. Nitrate (NO_3^--N) is determined by calculating the difference of [(NO_3^--N + NO_2^--N) - NO_2^--N] results. A Lachat Auto Flow Injection Analyzer, using a copperized cadmium reduction column procedure, is used to analyze the prepared samples. Values are reported as nitrogen, in the forms of nitrate + nitrite (NO_3^--N + NO_2^--N), nitrite (NO2--N), and nitrate (NO_3^--).

2. SUMMARY

Solid samples are first extracted with deionized water (at approximately a 1:10 ratio) by shaking using a rotary tumbler. The total nitrate + nitrite (NO_3^--N + NO_2^--N) content of the aqueous leachates or samples is determined quantitatively by first reducing nitrate (NO_3^--N) to nitrite (NO_2^--N) by passing the sample through a copperized cadmium column. The resultant nitrite (NO_2^--N), which consists of the original nitrite plus the reduced nitrate, is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylendiamine dihydrochloride to form a highly colored azo dye. This azo dye is then measured colorimetrically at 520nm. Aqueous samples are quantitated using a calibration curve created from standards made in deionized water. Native nitrite (NO_2^--N) *only* content is determined by conducting the analysis in a manner that bypasses the cadmium reduction column. The NO_2^--N is diazotized and coupled to yield an azo dye that is measured colorimetrically and quantitated using calibration curves. Finally, native nitrate (NO_3^--N) content is determined by subtracting the native nitrite ***only*** results from the combined nitrate + nitrite results.

3. RESPONSIBILITIES

3.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for review.

3.2 Analysts must demonstrate the ability to generate and interpret acceptable results utilizing these methods. This demonstration may come in the form of Supervisory/training review, results of precision and accuracy tests performed, or the successful completion of a proficiency evaluation test.

3.3 It is the responsibility of all personnel who work with samples or data involving this method to consult the applicable LIMS Program Specification for client-specific requirements prior to initiating handling of samples or data.

3.4 The Department Supervisor or designee performs final review and sign-off of the data. Initialing and dating the file indicate that this review for precision, accuracy, and completeness and is complete and satisfactory. Any errors that are found require corrective action, which includes notification to the technician/analyst who performed the work and documentation of measures taken to remediate the data.

3.5 It is the responsibility of all personnel who work with samples involving these methods to note any anomalies or out-of-control events associated with the analysis of the samples. Any discrepancies must be noted and corrective action taken and documented.

4. INTERFERENCES

4.1 Nitrate is very common in the laboratory environment because nitric acid is used extensively throughout the laboratory. Nitrate contamination of samples and blanks can be very troublesome. It is extremely important to use clean laboratory technique at all times. Use clean, disposable containers whenever possible and minimize the use of glassware.

4.2 This colorimetric procedure requires an optically clear sample. Furthermore, turbid samples or samples with suspended solids can cause a build up of suspended matter in the reduction column and restrict flow. Because nitrate and nitrite are found in a soluble form, if necessary, the sample should be pre-filtered through a 0.45µm pore diameter membrane filter. Sample color may also be corrected for by dilution to overcome the color interference, or by running the samples through the manifold with the cadmium column in the off-line position and without the addition of color reagent (the absorbance reading can then be calculated by subtracting the final from the initial absorbance reading, to obtain a corrected absorbance reading).

4.3 Concentrations of iron (Fe), copper (Cu) or other metals above several µg/mL lower reduction efficiency. EDTA is added to the buffer solution to eliminate this interference.

4.4 Residual chlorine can interfere by oxidizing the cadmium (Cd) column, thereby reducing efficiency. Removal of residual chlorine can be achieved by adding sodium thiosulfate ($Na_2S_2O_3$) solution.

4.5 Samples that contain large concentrations of oil and grease will coat the Cd granules surface. This interference can be removed by pre-extracting the sample with an organic solvent.

4.6 Sample color that absorbs at about 520nm interferes with the colorimetric determination. This may be corrected by diluting the sample or by running the sample through the manifold with the cadmium column in the off-line position and without the addition of color reagent for color formation, and calculating the corrected absorbance reading.

4.7 Samples that are over acidified with sulfuric acid preservative will cause a negative response within the peak area during analysis. Standard procedures recommend acidifying samples for nitrate + nitrite (NO_3^--N + NO_2^-- N) to a pH of less than 2 for preservation. All samples will have the pH adjusted to between 2 - 9 prior to analysis.

4.8 If the absorbance reading is too high on the laboratory blank, try flushing the column with DI water. If the buffer appears to be contaminated, prepare a fresh batch of solution using clean laboratory techniques. If high blanks continue to be a problem, remove the Cu-Cd granules from the column, treat with 6N HCl and 2% $CuSO_4$ and repack the column (see Section 12 of this SOP).

4.9 If the nitrite (NO_2^-- N) check sample's percent recovery becomes too high (>120%), treat reduction column granules as described in Section 12 of this SOP or repack the column in order to improve the column's efficiency.

5. APPARATUS AND MATERIALS

5.1 Flow injection analysis equipment designed to deliver and mix/react sample and reagents in the required order and ratios. Lachat QuikChem 8000, or equivalent, equipped with the following:

5.1.1 Autosampler, with sample tubes (12x75)

5.1.2 Peristaltic multichannel pump

5.1.3 Reaction unit or analytical manifold with Cadmium

5.1.4 Absorbance detector with 520nm filter

5.1.5 Data system (Windows 3.1 and Omnion software Version 1.4, or equivalents)

5.2 Volumetric dispensers, Eppendorf™ or equivalent, capable of dispensing 0.01-5.0mL

5.3 TCLP-type mechanical tumbler, capable of 1 hour soil extraction by agitation

5.4 Corning pH meter, Model 320 or equivalent, and appropriate buffer solutions for calibration. Capable of a two-point calibration.

5.5 Centrifuge, capable of sustaining approximately 3500rpm

5.6 Centrifuge tubes with caps, disposable, 50mL

5.7 Volumetric flasks, various sizes, Class A

5.8 Magnetic stir bars, Teflon coated

5.9 Magnetic stir plate, capable of variable speed control

5.10 Analytical balance, capable of weighing ± 0.0001g, verified per SOP 305

5.11 Syringe, with 0.45µm filter disk

6. REAGENTS

NOTES: Only ACS grade or better chemicals and reagents may be used. Reagents and solutions may be stored in either plastic or glass containers.

To prevent bubble formation, degas all solutions, except the standards or otherwise noted reagents, with helium. Use He at 140KPa (20lb/in^2) through a helium degassing tube. Bubble the He through the solution for one minute.

6.1 Ammonium Chloride/EDTA Buffer: Make in-house by dissolving 85g of ammonium chloride (NH_4Cl) and 1.0g Disodium Ethylenediamine Tetraacetate EDTA ($C_{10}H_{14}N_2O_8 \bullet 2H_2O$) salt in 800mL degassed DI water. Adjust to pH 8.5 with 10N Sodium Hydroxide (NaOH). Dilute to 1000mL with degassed DI water. *Shelf life = 1 year.*

6.2 Sodium Hydroxide Solution, 10N: Dissolve 160.0g Sodium Hydroxide (NaOH) to a final volume of 400mL using DI water. Not degassed. *Shelf life = 1 year.*

6.3 Color reagent: To 250mL degassed DI water, add 50mL concentrated Phosphoric Acid (H_3PO_4) and 20.0g Sulfanilamide. After dissolving the sulfanilamide completely, add 1.0g N-(1-naphthyl)-ethylenediamine dihydrochloride. Mix to dissolve, then dilute to 500mL with degassed deionized water. *Store refrigerated in an amber container. Shelf life = 6 months.*

6.4 Copper Sulfate ($CuSO_4$) Solution, 2%: Dissolve 4g Copper Sulfate ($CuSO_4 \bullet 5H_2O$) in 100mL DI water. Use a magnetic stirring bar and stir plate to speed dissolution. Not degassed. *Shelf life = Make fresh daily.*

6.5 Copper Sulfate ($CuSO_4$) Solution, 0.2%: Dilute 2% copper sulfate solution (above) ten-fold. *Shelf life = Make fresh daily.*

6.6 Hydrochloric Acid (HCl), 6N: Cautiously add 1 part concentrated HCl to 1 part DI water. Not degassed. *Shelf life = 1 year.*

6.7 Cu-Cd granules: Wash 10-20g new or used 0.3-1.5mm diameter (40 - 60-mesh) Cadmium (Cd) granules with 6N HCl and rinse with DI water. Swirl the Cd granules in portions of 100mL of 2% $CuSO_4$ until a dark colloidal precipitate begins to develop. Gently flush with DI water to remove all precipitated Cu. *Self life = Indefinite, good as long as darkened color persists; regenerate as needed as described in Section 12 of this SOP.*

6.8 Ottawa sand, EMD, SX0075-3 or equivalent. *Keep container sealed tightly. No special storage or shelf life considerations.*

6.9 Ammonium Hydroxide (NH4OH), conc., 15N: Purchased from vendor. Not degassed. *Shelf life = 1 year.*

7. NITRATE CALIBRATION STANDARDS (USED IN NITRATE + NITRITE TEST)

7.1 Nitrate (NO_3^--N) Stock Solution, 10000mg/L: Purchased from a commercial vendor or created from KNO_3 reagent grade salt. Dissolve 30.341g of $NaNO_3$ salt in 500mL deionized water. *Store in refrigerator. Shelf life = 1 year from purchase or creation.*

7.2 Nitrate (NO_3^--N) Stock Solution, 1000mg/L, second-source: Purchased from a commercial vendor or created from KNO_3 reagent grade salt. Dissolve 3.0341g of $NaNO_3$ salt in 500mL deionized water. KNO_3 salt must be from a different vendor or lot number than that used for the first source standard. *Store in refrigerator. Shelf life = 1 year from purchase or creation.*

7.3 Nitrate (NO_3^--N) Intermediate Standards, 100mg/L (first and second source): First-source solution is made by diluting 5.0mL of the 10000mg/L "First Source" NO_3^--N Stock Solution with deionized water, to a final volume of 500mL. This "First Source" Intermediate Standard is used to prepare the daily calibration standards and the continuing calibration verification (CCV) standards. *Store in refrigerator. Shelf Life = 1 year from purchase or creation (note: cannot exceed expiration of parent standard).*

Second-source solution is made by diluting 25.0mL of the "Second Source" 1000mg/L NO_3^--N Stock Solution with deionized water, to a final volume of 250mL. The "Second Source" Intermediate Standard is used to prepare the

Laboratory Control sample (LCS) and initial calibration verification (ICV) standard. *Store in refrigerator. Shelf Life = 1 year from purchase or creation.*

7.4 NO_3^--N Calibration Standards (See ExampleTable below): Made in-house on *each day of calibration* by dilution of the Intermediate Standard (100mg/L, first source).

Note that DI water is used as the diluent to prepare calibration standards for aqueous sample analyses. The final volume of all calibration standards is 5.0mL.

CONCENTRATION OF INITIAL SOLUTION (mg/L)	VOLUME OF INITIAL STANDARD USED (mL)	CONCENTRATION OF FINAL SOLUTION (mg/L)
100*	0.10*	2.0*
100*	0.05*	1.0*
100	0.025	0.50
2.0	0.50	0.20
1.0	0.50	0.10
0.5	0.50	0.05
0.1	0.50	0.01
0	0.00	0.0
*Optional		

7.5 Initial Calibration Verification Standard, (ICV), 0.50mg/L: Dilute 0.025mL of the 100mg/L"second source" NO_3^--N Intermediate Standard into 5.0mL, using deionized water.

7.6 Continuing Calibration Verification Standard, (CCV), 1.0mg/L: The "first source" intermediate standard is used to prepare the continuing calibration verification (CCV) standard. Dilute 0.050mL of the 100mg/L "first source" NO_3^--N intermediate standard into 5.0mL deionized water.

7.7 NO_2^--N Cadmium Column Check Standard, 1.0mg/L: Dilute 0.1mL of the 50mg/L NO_2^--N Intermediate Standard (from section 8.0) into 5.0mL deionized water.

NOTE: The Calibration Standards with concentrations of 10mg/L or less are made daily upon use.

8. NITRITE CALIBRATION STANDARDS (USED IN NATIVE NITRITE *ONLY* TEST)

8.1 Nitrite (NO_2^--N) Stock Solutions, 1000mg/L: Created from $NaNO_2$ reagent grade salts, first and second sources. Second source is second vendor, if available. Used to create the first and second source NO_2^--N 200mg/L Intermediate Standards. Dissolve 0.4925g of $NaNO_2$ salt into 100mL deionized water. *Store in refrigerator. Shelf life = 1 month.*

8.2 Nitrite (NO_2^--N) Intermediate Standards, 200mg/L (first source): First-source solution is made by diluting 20.0mL of the 1000mg/L NO_2^--N Stock Solution with deionized water, to a final volume of 100mL. This "First Source" Intermediate Standard is used to prepare the daily calibration standards and the continuing calibration verification (CCV) standards. *Shelf Life = 1 month (note: cannot exceed expiration of parent standard).*

Note that the Laboratory Control sample (LCS) and Initial Calibration Verification (ICV) standard are made from the "second source" 1000mg/L stock solution or 200mg/L solution, described below.

8.3 Nitrite (NO_2^--N) Intermediate Standards, 200mg/L (second source): Second source solution is made by diluting 20.0mL of the 1000mg/L NO_2^--N Stock Solution with deionized water, to a final volume of 100mL. This "second Source" Intermediate Standard is used to prepare the initial calibration verification (ICV) standard and the Laboratory Control Sample (LCS) standard, which are identical. *Shelf Life = 1 month (note: cannot exceed expiration of parent standard).*

Note that the Laboratory Control sample (LCS) and Initial Calibration Verification (ICV) standard are made from the "second source" 1000mg/L stock solution or 200mg/L solution.

8.4 NO_2^--N Calibration Standards (See Example Table below): Made in-house *each day of calibration* by dilution of the first source 100mg/L NO_2^--N Intermediate Standard.

Note that DI water is used as the diluent to prepare calibration standards.

CONCENTRATION OF INITIAL SOLUTION (mg/L)	VOLUME OF INITIAL STANDARD USED (mL)	CONCENTRATION OF FINAL SOLUTION (mg/L)	FINAL VOLUME OF SOLUTION (mL)
200*	0.050*	0.50*	20*
200	0.020	0.20	20
200	0.010	0.10	20
0.50	0.50	0.05	5
0.20	0.50	0.02	5
0.10	0.50	0.01	5
0	0.0	0.0	5
* Optional			

8.5 Initial Calibration Verification Standard, (ICV), 0.1mg/L: Dilute 0.01mL of the 1000mg/L"second source" NO_2^--N Stock Standard into 100mL, using deionized water or 0.01mL of 200mg/L second source standard into 20mL of DI water.

8.6 Continuing Calibration Verification Standard, (CCV), 0.2mg/L: The "first source" intermediate standard is used to prepare the continuing calibration verification

(CCV) standard. Dilute 0.02mL of the 200mg/L "first source" NO_2^--N intermediate standard into 20.0mL deionized water.

NOTE: The Calibration Standards with concentrations of 10mg/L or less are made daily upon use.

9. SAMPLE COLLECTION, PRESERVATION, HANDLING AND HOLDING TIMES

9.1 All samples must be collected according to an approved sampling plan.

9.2 Sampling and storage of samples in glass bottles or in plastic bottles are permissible. Samples should be kept cool at 4±2°C.

9.3 For analysis of NO_2^--N only, aqueous samples must be analyzed within 48 hours after collection. There is no promulgated preservation or holding time for soils.

9.4 For NO_2^--N + NO_3^--N analysis using the Cd Column, aqueous samples must be acidified to a pH≤2 with Sulfuric Acid (H_2SO_4), with analysis occurring within 28 days after collection.

10. AQUEOUS EXTRACTION OF SOLID SAMPLES AND ASSOCIATED QUALITY CONTROL (QC) SAMPLE PREPARATION

10.1 Weigh a representative 4.0g aliquot of moist sample into a labeled 50mL disposable polypropylene centrifuge tube.

10.2 Method Blank: Weigh 4.0g clean Ottawa sand into a 50mL disposable polypropylene centrifuge tube.

10.3 As applicable, prepare both NO_2^--N + NO_3^--N and NO_2^--N Blank Spike, Laboratory Control Samples (LCSs) as follows:

10.3.1 Weigh 4.0g clean silica sand into a 50mL disposable polypropylene centrifuge tube. Spike with 0.40mL of 100mg/L "second source" NO_3^--N Intermediate Standard (expected concentration = 1.0mg/L NO_3^--N).

10.3.2 Weigh 4.0g clean silica sand into a 50mL disposable polypropylene centrifuge tube. Spike with -0.050mL of 200mg/L second source standard (expected concentration = 0.25mg/L NO_2^--N).

10.4 As applicable, prepare both NO_2^--N + NO_3^--N and NO_2^--N Matrix Spike (MS), Matrix Spike Duplicate (MSD) samples as follows:

10.4.1 Select a representative sample of the batch. Spike two duplicate 4.0g representative aliquots with 0.16mL of 100mg/L "first source" NO_3^--N Intermediate Standard (expected concentration = 0.4mg/L NO_3^--N).

10.4.2 Select a representative sample of the batch. Spike two duplicate 4.0g representative aliquots with 0.02mL of 200mg/L "first source" NO_2^--N intermediate standard (expected concentration = 0.10mg/L NO_2^--N).

10.5 To all of the above add 40.0mL of deionized (DI) water.

10.6 Shake samples for 1 hour on the TCLP tumbler.

10.7 Centrifuge for approximately 15min @ 3500rpm.

11. AQUEOUS FIELD SAMPLE, AQUEOUS EXTRACT AND ASSOCIATED QUALITY CONTROL (QC) SAMPLE PREPARATION

11.1 pH ADJUSTMENT OF AQUEOUS SAMPLES
If the samples were preserved with sulfuric acid, as is the case with nitrate + nitrite samples, adjust the pH to greater than 2 but no more than 9 as follows (see comment Section 4.7):

11.1.1 Place approximately 20mL of aqueous sample into a disposable centrifuge tube containing a stir bar.

11.1.2 Place a previously calibrated pH probe into the sample aliquot and adjust the pH using 10N NaOH until the pH is between 2 - 9. If the pH exceeds 9, add the original preserved sample drop wise to bring the pH back down into range.

NOTE: If samples are drinking water samples analyzed for compliance monitoring, then use concentrated ammonium hydroxide (NH_4OH) as the pH-adjusting reagent.

NOTE: After the pH of the sample is adjusted above pH 2, a 48-hour hold time begins. Samples are normally analyzed immediately after pH adjustment. Note also that for nitrite (only) analysis, samples are unpreserved.

11.2 Aliquot 5mL of each aqueous sample or previously prepared solid sample extract (including associated QC samples) into a designated auto sampler tube. If the aqueous sample or extract contains suspended solids, filter through a 0.45µm pore-diameter membrane filter.

11.3 Prepare the aqueous Method Blank (MB) by aliquotting 5mL of DI water into an auto sampler tube.

11.4 As applicable, prepare both the NO_2^--N + NO_3^--N and NO_2^--N aqueous Blank Spike, Laboratory Control Samples (LCS) as follows:

11.4.1 Aliquot 0.025mL of 100mg/L "second source" NO_3^--N Intermediate Standard into 5mL DI water (expected concentration = 0.5mg/L NO_3^--N).

11.4.2 Aliquot 0.010mL of 1000mg/L second source NO_2^--N into 100mL DI water or 0.010mL of 200mg/L second source solution into 20mL of DI water and mix. Transfer 5mL into a designated auto sampler tube (expected concentration = 0.10mg/L NO_2^--N).

11.5 As applicable, prepare both the NO_2^--N + NO_3^--N and NO_2^--N aqueous Matrix Spike (MS), Matrix Spike Duplicate (MSD) as follows:

11.5.1 Select a representative sample for the batch. Spike two duplicate 5mL aliquots with 0.020mL of 100mg/L "first source" NO_3^--N Intermediate Standard (expected concentration = 0.40mg/L NO_3^--N).

11.5.2 Select a representative sample for the batch. Spike two duplicate 5mL aliquots with 0.01mL of 200mg/L NO_2^--N. Mix well. Transfer 5mL into a designated auto sampler tube (expected concentration = 0.10mg/L NO_2^--N).

12. PREPARATION, USE AND MAINTENANCE OF CADMIUM REDUCTION COLUMN

12.1 CADMIUM PREPARATION

Place 10-20g of coarse cadmium granules (0.3 - 1.5mm diameter) in a 150mL beaker. Wash with two 25mL portions of 6N hydrochloric acid. Rinse several times with DI water.

CAUTION: Collect and store all waste cadmium. Cadmium is toxic and carcinogenic. Wear gloves and follow the precautions described on the Material Safety Data Sheet (MSDS).

12.2 COPPERIZATION

Repeatedly mix the Cd granules in portions of 100mL 2% Copper Sulfate Solution ($CuSO_4$). Swirl for about 5 minutes, then decant the liquid (discard in appropriate waste stream) and repeat with a fresh portion of 2% copper sulfate solution. Continue this process until the blue aqueous copper color persists. Decant and wash the copperized Cd granules at least five times with DI water to remove colloidal copper. The cadmium should be black or dark gray. The copperized cadmium granules may be stored in a stoppered bottle in ammonium chloride buffer.

12.3 PACKING THE CADMIUM COLUMN

The empty cadmium column is available as Lachat Part #50230. Wear gloves and do all cadmium transfers over a special tray or beaker dedicated to this purpose. Clamp the empty column upright so that your hands are free. Unscrew one of the colored fittings from an end of the column, and pull out and save the foam plug (if necessary, glass wool fiber and be used in lieu of the foam plug). When replacing the cap, it

may be necessary to apply Teflon thread tape in order to prevent leakage. The column and threads are glass, so be careful not to break or chip them.

Scoop up prepared copperized cadmium granules with a spatula and pour them into the top of the column so that they sink down to the bottom of the column. Continue pouring the copperized cadmium in while gently tapping the column with a screwdriver handle or similar device to dislodge any air bubbles and also to prevent gaps in the cadmium filling. When the cadmium granules reach about 5 mm from the open end of the column, push in the foam plug and screw on the top fitting. Rinse the outside of the column with DI water.

If air remains in the column or is introduced accidentally, a leur-lock syringe can be used to flush the column with ammonium chloride solution and push the air out.

Carefully cap both column ends for storage.

12.4 CADMIUM COLUMN INSERTION PROCEDURE

12.4.1 Before inserting the column, pump all reagents into the manifold making sure that there are no leaks.

12.4.2 Turn the pump to the lowest setting.

12.4.3 On the column, connect one of the union connectors to the outlet tubing of the buffer mixing coil.

12.4.4 Connect the open tubing on the column to the tee fitting where the color reagent is added. **BE CAREFUL TO NOT LET AIR ENTER THE COLUMN!!**

12.4.5 Turn column valve to open position. Return the pump to normal speed.

NOTE: The direction of reagent flow through the column is not relevant.

12.5 MONITORING COLUMN EFFICIENCY

12.5.1 Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column packing, or any change in the cadmium surface characteristics (cadmium granules should be dark gray). If column treatment is necessary, see Note below.

12.5.2 Condition the column by running a 5mg/L NO_3^--N standard through it three times followed by reagent washing for approximately 1min.

12.5.3 Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect the effluent in a graduated cylinder. The flow rate with the column thusly connected should be

greater than 4.0mL/min.

NOTE: The presence of air in the column will decrease efficiency. Usually the problem can be corrected and the granules re-activated by using a leur-lock syringe to flush the column with solution (50mL ammonium chloride solution to push out the air, or 50mL of 0.2% copper sulfate solution followed by 50mL of ammonium chloride solution to re-activate the cadmium granules). However, if the problem cannot be corrected, the column should be repacked per Section 12.3 of this SOP.

12.5.4 If the nitrite (NO_2^-N) check sample's percent recovery becomes too high (i.e., >120%), empty the column and retreat the cadmium granules as described in the Note above. Repacking and reinstalling the column should improve the column's efficiency.

13. GENERAL LACHAT OPERATION AND MAINTENANCE PROCEDURES

13.1 LACHAT INSTRUMENT SET UP

13.1.1 Turn power switches on for auto sampler, pump and the sample-processing module. Turn on the computer.

NOTE: The auto sampler will automatically perform an operation check by lifting the probe out of the rinse reservoir and advancing the sample cartridges by one position. The sample-processing module will open and shut each of the two valves, the LED of the heater control will display the current temperature of the block, and the lamp will come on.

13.1.2 Install the proper manifold for the analysis to be conducted (e.g., nitrate + nitrite analysis).

13.1.3 Place the appropriate interference filter into the upper slot of the detector.

13.1.4 Connect the "sample loop" to ports one and four of the injection valve.

13.1.5 Connect the "carrier" line to port two of the injection valve.

13.1.6 Place the manifold on to the sample processing module and connect the "to manifold" line to port three of the injection valve.

13.1.7 Connect the heating unit tubing to the manifold (if necessary).

NOTE: No heat is necessary for NO_2^--N + NO_3^-N and NO_2^--N analysis.

13.1.8 Insert each pump tube into a pump tube cartridge and place on the pump. Adjust the tension levers to maximum tension. ***Be sure that the pump tubes are seated correctly in the cartridges. This will prevent pinching of the tubes, which may restrict flow.***

NOTE: When preparing for any analysis that requires a column, make sure the column is in the ***off-line*** position at this time.

13.1.9 If you are running a heated procedure, set the heat controller temperature to the required setting by pressing the "on" button. To adjust the temperature, press the "up" and "down" buttons. Press the "on" button again to lock in the setting.

NOTE: ***Be sure to turn the heat controller off at the end of the run!***

13.1.10 Prepare the appropriate working standards and place them in the sampler in order of decreasing concentrations. Complete filling of the sampler tray using the prepared samples to be analyzed.

13.1.11 After flushing the lines with deionized water, place the reagent lines into the proper reagent solutions and begin pumping reagent through the system.

NOTE: ***Some chemistries require a certain sequence in which reagents are introduced. Check the Lachat's operator's manual.***

13.1.12 If running a chemistry that requires a column, place the column ***on-line*** at this time by first turning the pump speed to the lowest setting, then placing the column switch to the "on-line" position. Turn the pump speed back to the required setting of 35rpm slowly, avoiding the introduction of any air bubbles.

13.1.13 Allow the reagents to be pumped through the entire system for at least 3-5 minutes.

13.1.14 Set up the computer as described in Section 13.2. Once the computer is set up, the system is ready to be started to begin calibration and data collection. Refer to the specific analyte procedure Sections of this SOP.

13.1.15 System shut down procedures are addressed in SOP Section 18.0.

13.2 COMPUTER SET UP PROCEDURE

13.2.1 From the Program Manager in Windows, double click on the "Lachat Instruments" icon. Then double click on the "Omnion FIA" icon. Click "OK" in the Omnion data system window.

NOTE: The sample probe will return to the wash reservoir and the injection valves will open and close.

13.2.2 Type your user name and password then click "OK". Click on the "Flow Injection Analysis" icon. If a username and password has not been assigned, have the System Manager assign one, or refer to the installation section of the *QuickChem 8000 Continuum Series Automated Ion Analyzer Omnion FIA Software Installation and Tutorial* manual.

13.2.3 Load the method for the analyte(s) you want to run. From the menu bar, click on "File", then "Open Method". The path for Channel 1 is c:\Omnion\methods\method_temp\noxtemp.met. A new "Method" is created for each analysis each day of use. Each day's data are stored in an analyte subdirectory named for the year, using a mmdd format (e.g., c:\Omnion\methods\nox\2004\0412nox.met). This method pathway refers to NO_3^--N + NO_2^--N run on 04/12/2004.

NOTE: For native nitrite (only) analysis, substitute NO2 for NOX in all pathways. Example: c:\Omnion\methods\no2\2004\ 0412no2.met).

13.2.4 Load the master trays for the analyte(s) you want to run. From the "File" option on the menu bar, click on "Open Tray". Select the appropriate tray. The path for the default tray is c:\Omnion\trays\traytemp\noxtemp.tra. Each day's analysis data are stored in a subdirectory named for the year using a mmdd format (e.g., c:\Omnion\trays\2004\0412nox.tra). Fill in the sample identifications and dilution factors and save the tray by clicking "File", then "Save Tray As".

13.2.5 Check the calibration curve information by clicking on the "Review" icon, or pull down the "Method" menu and click on "Review Analyte Calibration Curve". If calibration curve data appears, clear the previous data by pulling down the "Method" menu and clicking on "Calibration Clear".

NOTE: You must clear the previous calibration curve before analyzing calibration standards.

13.3 MANIFOLD CLEANING PROCEDURE

If the baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

13.3.1 Place all reagent lines in deionized water and pump 2-5 minutes to clear the lines of all residual reagents.

13.3.2 Place the reagent lines and carrier in 1M hydrochloric acid (i.e., 82.6mL concentrated HCl brought to a final volume of liter using DI water). Allow the pump to run for several minutes to flush and clean the lines.

13.3.3 Place all the flushed lines in deionized water and pump until the HCl is thoroughly washed out.

13.4 Reattach all the reagent lines and resume pumping the reagents.

Note that the manifold cleaning procedure should be done with the column off-line or not attached.

14. PROCEDURE FOR DETERMINING NITRATE PLUS NITRITE

NOTE: When particulate matter is present, the sample must be filtered prior to the analyses. The sample may be centrifuged in place of filtration.

14.1 Prepare the calibration standards as described in Sections 7.0.

It should be noted that only the NO_3^--N standards and an NO_2^--N column check standard are used in the determination of nitrate + nitrite (see sequence below). This is done so that the standards are processed in the same manner as the samples (i.e., NO_3^--N content is reduced to NO_2^--N via the cadmium reduction column).

14.2 Prepare the samples as described in Sections 10.0 (aqueous leachates) or 11.0 (aqueous samples).

14.3 Fill the autosampler tubes with the appropriate standards and samples (including QC); load the auto sampler as described in Step 13.1.10 and per the run sequence depicted below:

Stage the autosampler per the following run sequence:

- 2.00mg/L NO_3^--N calibration standard*
- 1.00mg/L NO_3^--N calibration standard*
- 0.50mg/L NO_3^--N calibration standard*
- 0.20mg/L NO_3^--N calibration standard
- 0.10mg/L NO_3^--N calibration standard
- 0.05mg/L NO_3^--N calibration standard
- 0.01mg/L NO_3^--N calibration standard
- 0.00mg/L NO_3^--N calibration standard

- (ICV (second source). Results must agree within ±10% of known value.
- ICB (Initial Calibration Blank, aqueous matrix Method Blank). *See also note below.* Any analyte concentration found must be less than the analyte reporting limit.
- RVS Sample – LCS prepared at reporting limit
- Preparation (Method) Blank. Any analyte concentration found must be less than the analyte reporting limit.
- Laboratory Control Sample (LCS)
- maximum of 10 Field Samples
- CCV (prepared like the 1.0mg/L calibration standard). Results must agree within ±10% of known value.
- CCB (DI water). Any analyte concentration found must be less than the analyte reporting limit.
- maximum of 10 Field Samples
- CCV
- CCB

*Optional

NOTES: *It is often helpful to refer to your tray as a guide to ensure proper placement of standards and samples in the auto sampler.*

No more than ten samples may be analyzed between the ICV/ICB and first CCV/CCB.

No more than ten samples may be analyzed between CCV/CCB sets.

A CCV/CCB set must be analyzed to close-out the run sequence.

14.4 Complete the proper instrument set up as described in Steps 13.1.11 through 13.1.14.

14.5 Make sure the method and tray pathways are correctly set up and that the previous calibration information has been cleared as described in Section 13.2.

14.6 Start the run by clicking on the "Run Tray" icon. Click "Catalogue". Save the run data catalogue in the proper path using a mmdd format (i.e., c:\Omnion\data\ nox\2004\0412nox.fdt). Click "Run" to begin the analysis process.

NOTE: The analysis may be paused or stopped in the middle of the run by clicking on the "Stop" icon. You can add samples or sample dilutions to your run by clicking on the "Tray" window and typing them in at the end of your saved tray. Save the updated information by first clicking "File", then "Save Tray". Restart the run by clicking "Resume". If you edit your tray by deleting standards or samples, you must "Save Tray

As" by using the original file name with a "b" ("c", "d", etc.) added; example: 0412noxb.tra. Restart the analysis by renaming the data file in the same manner; example: 0412noxb.fdt.

15. PROCEDURE FOR DETERMINING NITRITE (ONLY) CONTENT

Prepare samples as described previous in Sections 10.0 or 11.0. To analyze for nitrite (only) content, use the same determination steps described in Section 14.0 above, with the following exceptions:

- Only the nitrite calibration standards (Section 8.0), and only the NO_2^--N QC samples (i.e., LCS and MS/MSD; Sections 10.0 and 11.0) need to be loaded into the autosampler (Step 13.1.10). *See run sequence below.*

- Aqueous field samples and the aqueous extracts of solid field samples analyzed for nitrite (only) do not require pH adjustment.

- The analyzer's cadmium column switch is placed in the "off-line" position for this analysis. The cadmium column is not used in nitrite determination.

- The pathway and naming convention used for the method is c:\Omnion\ methods\no2\2004\0412no2.met (substitute proper date, mmdd, for 0412).

  The pathway and naming convention used for the tray is c:\Omnion\trays\ 2004\0412no2.tra (substitute proper date, mmdd, for 0412).

  The pathway and naming convention used for the run data file is c:\Omnion\ data\no2\2004\0412no2.fdt (substitute proper date, mmdd, for 0412).

- <u>Stage the autosampler per the following run sequence:</u>

  (1) 0.50mg/L NO_2^--N calibration standard*
  (2) 0.20mg/L NO_2^--N calibration standard
  (3) 0.10mg/L NO_2^--N calibration standard
  (4) 0.05mg/L NO_2^--N calibration standard
  (5) 0.02mg/L NO_2^--N calibration standard
  (6) 0.01mg/L NO_2^--N calibration standard
  (7) 0.00mg/L NO_2^--N calibration standard
  (8) ICV (second source). Results must agree within ±10% of known value.
  (9) ICB (Initial Calibration Blank, aqueous matrix Method Blank). *See also note below.* Any analyte concentration found must be less than the analyte reporting limit.

  - RVS Sample – LCS prepared at reporting limit
  - Preparation (Method) Blank. Any analyte concentration found must be less than the analyte reporting limit.
  - Laboratory Control Sample (LCS)

- maximum of 10 Field Samples
- CCV (prepared like the 1.0mg/L calibration standard). Results must agree within ±10% of known value.
- CCB (DI water). Any analyte concentration found must be less than the analyte reporting limit.
- maximum of 10 Field Samples
- CCV
- CCB

*Optional

NOTES: *It is often helpful to refer to your tray as a guide to ensure proper placement of standards and samples in the auto sampler.*

No more than ten samples may be analyzed between the ICV/ICB and first CCV/CCB.

No more than ten samples may be analyzed between CCV/CCB sets.

A CCV/CCB set must be analyzed to close-out the run sequence.

16. PROCEDURE FOR DETERMINING NITRATE (ONLY) CONTENT

Nitrate (only) is determined by subtracting the nitrite (only) sample results from the combined Nitrate + Nitrite sample results. An Excel spreadsheet is used to conduct all nitrate sample calculations. Refer to SOP Section 19.0.

17. REPORTING RESULTS

After the analysis is complete, print a copy of the following analyzer reports (access proper menu, "Open", "Print") and copy data to a data disk (access proper menu, "Import" and save to drive A:\, to be transferred to the LIMS:

- Tray Table(s) with any written comments and handwritten date and initials of analyst.
- Run Time Report(s); must be hand dated and initialed by the analyst.
- Import data (as txt. File) to be transferred i:\oprtns\wtchm\limsdata.

Results are calculated as described in SOP Section 19.

18. SYSTEM SHUT DOWN PROCEDURE

NOTE: ***Some methods require certain reagents to be removed last. Check the Lachat's operator's manual.***

18.1 If applicable, turn the pump setting down then switch the column to the "off-line" position.

18.2 Turn the heat down.

18.3 Remove the reagent lines from each reagent and place into deionized water. Turn the pump setting up so that the DI water is pumped through. Continue to run the system and allow the transmission lines to rinse for 5-10 minutes. ***This is a critical step in preventive maintenance of the manifolds.*** Then, allow the lines to suck air briefly to remove any liquid.

18.4 Release the cartridge tension on the transmission line to the sipper bath and remove the line from the deionized water. Turn the heat controller off (if required).

18.5 Make sure the appropriate files are saved, then exit and log off from the current Omnion session. Exit Windows and shut down the computer.

18.6 Remove the reagent transmission lines from the deionized water and allow all liquid to be pumped out of the manifold by pumping air through the lines.

18.7 Turn off the pump, auto sampler and manifold unit by switching off the power strip.

18.8 Release the pump tube cartridges tension completely to avoid the tubing from becoming crushed.

18.9 To remove the manifold, follow the Lachat set up procedure (Section 13.1) ***in reverse order***, starting with Step 13.1.8.

19. CALCULATIONS

19.1 Excel spreadsheets are used to verify spike information and calculate nitrate results by difference. The pathways for each Excel spreadsheet template are as follows:

Nitrate + nitrite, Nitrite, (aqueous and soil samples), spike verification information: i:\oprtns\wtchm\nox\template\noxautowver.xls.

Nitrate, calculation by difference:
i:\oprtns\wtchm\no3\template\no3temp.xls.

Call up each Excel spreadsheet template you need to use and rename it in the year's subdirectory using the identity of the mmdd (without the year reference) in the batch as a naming convention.

Example: i:\oprtns\wtchm\nox\2002\1211nox.xls.

19.2 The standard curve is plotted by the Lachat and is imported, along with all run data, to LIMS. Linear regression analysis of the standard curve yields a coefficient of determination (r^2) value. For the standard curve to be accepted, the r^2 value must be ≥ 0.995.

19.3 Applicable sample adjustments (i.e., dilutions, % solids) are made in LIMS and final sample analyte concentrations are quantitated. LIMS also calculates QC sample % recoveries and RPD values.

The representative sample calculation for NO_3^--N + NO_2^--N in soil samples is as follows:

$$\text{Concentration (mg/L)} = \frac{A \bullet D \bullet V}{W_{dry}}$$

where:

A = mg/L NO_3^--N + NO_2^--N (calculated from the sample peak area using the standard curve equation)

D = Dilution factor (if applicable)

V = Extract volume (mL); (40mL if this SOP is followed without deviation)

W_{dry} = Dry weight (g) of soil extracted (see equation below)

$$W_{dry} = \frac{W_{moist} \bullet \% \text{ solids}}{100}$$

where:

W_{moist} = moist weight of soil extracted

20. QUALITY CONTROL

20.1 DEFINITION OF ANALYSIS BATCH

For this method, an analysis batch is defined as a group of twenty (20) or fewer field samples that is associated with one unique set of batch QC samples and are processed together as a unit. Batch QC samples are defined in LQAP Section 9.2. All quality control samples must be carried through all stages of the sample preparation and measurement steps.

20.2 METHOD BLANKS

For this procedure, the MB consists of 5mL DI water or 4.0g clean sand. Any analyte concentration found in the blank must be less than the analyte reporting limit.

20.3 LABORATORY CONTROL SAMPLE

For this method, 5mL DI water (representative of aqueous samples) and 4.0g clean sand (representative of solid samples) were used as the clean matrices.

To be acceptable, LCS recovery must be between 90% and 110% (aqueous matrix) and 85% and 115% (soil matrix) of the expected concentration.

20.4 MATRIX SPIKE AND MATRIX SPIKE DUPLICATE

The advisory quality control limits for MS/MSD recovery are set at 75-125%.

The RPD should be ≤20 to be accepted.

20.5 METHOD DETECTION LIMIT STUDY

21. A method detection limit is determined in accordance with ALS SOP 329.

DEVIATIONS FROM METHOD

21.1 Section 6.5 of Method 353.2 provides for treated distilled water for the preparation of samples, standards and reagents. ALS uses deionized (DI) water generated by the in-house laboratory deionization system. It has been demonstrated, through the analysis of MDL studies and PT samples that ALS's DI water is free of NO_3^--N and NO_2^--N and meets ASTM Type I and II standards. Therefore, no additional treatment is applied.

21.2 SM 4500-NO_3^- I reagent citations, Section 6 of Method 353.2, and QuickChem Method 10-107-04-1-C all provide for the preservation of NO_3^--N and NO_2^--N stock standards with chloroform. ALS closely monitors the performance of and assigned shelf lives of the 1000 mg/L nitrate (NO_3^--N) and nitrite (NO_2^--N) stock standards and does not preserve these standards with chloroform.

21.3 Section 6.9 of Method 353.2 discusses the creation and use of a wash solution. Because the type of automated flow injection analyzer used by ALS provides a constant flow of reagents through the system, the use of a separate wash solution is not required.

21.4 The amount of EDTA used in the creation of the ammonium chloride/EDTA buffer solution presented in Section 6.10 of Method 353.2 (0.1g) differs from that presented in the reagent citations of SM4500-$NO3^-$ I (1.0g). ALS uses an approximate median amount of 1.0g, because this amount is cited in the manufacturer's method, QuickChem Method 10-107-04-1-C.

The recipe for the ammonium chloride/EDTA buffer solution presented in Section 6.10 of Method 353.2 mentions the addition of 1/2mL "Brij-35" (available from Technicon Corporation) to the ammonium chloride/EDTA buffer solution. The addition of this material is not provided for in SM4500-$NO3^-$ I or in the manufacturer's method. ALS does not add this material the ammonium chloride/EDTA buffer solution.

21.5 Section 6.0 of Method 353.2 provides for the use of KNO_2 in the creation of the nitrite standards. ALS uses $NaNO_2$ to make the nitrite standards as provided for in Section 3g of SM4500-NO3- I.

21.6 ALS uses a 200mg/L intermediate nitrite solution rather than the 100mg/L intermediate nitrite solution described in Method 353.2 (Section 6.0).

21.7 Section 10 of this SOP describes a solid preparation procedure. ALS notes that Method EPA 353.2 is applicable to the measurement of nitrate/nitrite in surface and saline waters, and domestic and industrial wastes (Section 1.1).

21.8 Section 7.1 of Method 353.2, and QuickChem Method 10-107-04-1-C all provide for the adjustment of sample pH with Ammonium Hydroxide and Hydrochloric Acid. ALS uses 10N Sodium Hydroxide and the actual preserved sample to adjust the pH. If samples are drinking water samples analyzed for compliance monitoring, then concentrated ammonium hydroxide (NH_4OH) will be used as the pH-adjusting reagent.

22. SAFETY, HAZARDS AND WASTE DISPOSAL

22.1 SAFETY HAZARDS

All Safety and Hazards are managed in accordance with the current facility plans:

- Chemical Hygiene Plan (CHP)
- Radiation Protection Plan (RPP).
- Emergency and Contingency Plan (ECP)
- Respiratory Protection Plan (RESPP)

22.2 WASTE DISPOSAL

All Wastes are disposed of in accordance with the Waste Management Plan (WMP)

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23. REFERENCES

23.1 A.P.H.A., A.W.W.A. and W.P.C.F., 1989. Standard Methods for the Examination of Water and Wastewater, 20th edition, Revised 1998, Method 4500-NO_3 I.

23.2 Keeny, D.R. and D.W. Nelson, 1982. Methods of Soil Analysis, Part 2, Agronomy 9:649, American Society of Agronomists, Madison, WI. "Nitrogen - Inorganic Forms".

23.3 U.S. Environmental Protection Agency, EPA-600/4-79-020, Methods for the Chemical Analysis of Water and Wastes, Revision 2.1, 1993, Method 353.2, "*Determination of Nitrate and Nitrate plus Nitrite by Colorimetric, Automated, Cadmium Reduction Procedure*".

23.4 Lachat Automated Ion Analyzer Methods Manual, Method Number 10-107-04-1C, Determination of Nitrate/Nitrite, Nitrite in Surface Water, Wastewater.

Analytical Method: EPA 353.2, SM 4500-NO_3 I	**Parameter:** Nitrate, Nitrate plus Nitrite, and Nitrite in water and soil		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration; minimum 5-point	As needed (i.e., at on-set of analyses or repeated when continuing calibration does not meet criteria)	Coefficient of determination (r^2) for linear regression must be $\geq$0.995	Check that the calibration standards were prepared properly. Evaluate/ correct instrument malfunction and reanalyze initial calibration to obtain acceptable curve.
Independent Calibration Verification (ICV); second source; at or below midpoint	Once after each initial calibration	Response must agree within $\pm$10% of initial calibration	Prepare another ICV and analyze. If ICV still fails, system must be recalibrated.
Blanks: Method Blank (MB), Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)	The MB may be run initially as part of the calibration curve, the ICB is run following the calibration curve. CCBs are run following the CCV (after every ten samples), and to close an analytical run sequence	NO_2^--N / NO_3^--N content of the blank must be < RL	Check all calculations. If no computation errors are found, prepare a fresh blank and analyze. All samples run since the last acceptable blank must also be reanalyzed.
Laboratory Control Sample (LCS)	One LCS must be run per 20 environmental samples of similar matrix	Results must agree within $\pm$10% of expected values for aqueous sample analyses; within ±15% for solid matrix extract analyses	Check all calculations. If no computation errors are found, prepare a fresh LCS and analyze. If criteria are still not met, system must be recalibrated and all samples run since the last acceptable LCS must be reanalyzed.
Continuing Calibration Verification (CCV); at or below midpoint; first source	Run after every ten samples and to end any run sequence (must be followed by a CCB analysis)	Response must agree within $\pm$10% of initial calibration	Check that calculations and preparation are correct, evaluate/ correct instrument malfunction; reanalyze. If CCV still fails, recalibrate system. All samples analyzed after the last acceptable CCV must be reanalyzed.
Matrix Spike (MS)	One per batch of $\leq$20 field samples of similar matrix	Recoveries should meet advisory limits of ±25% of the expected values; client-specified criteria may apply	Compare with LCS results and check for documentable errors (e.g., calculations and spike preparation). If no errors are found, then sample matrix effects are the most likely cause. Note in narrative.
Matrix Spike (laboratory) Duplicate	One per batch of $\leq$ 20 field samples of similar matrix	(See MS recovery criteria above). RPD advisory limit is $\leq$20; client-specified criteria may apply	(See MS recovery criteria above). For RPDs outside of QC limits, check all calculations for errors. If no errors are found, discuss with Department/ Project/QA Managers.

Analytical Method: EPA 353.2, SM 4500-NO_3 I	**Parameter:** Nitrate, Nitrate plus Nitrite, and Nitrite in water and soil		**Summary of Internal Quality Control (QC) Procedures and Corrective Actions**
Quality Control Check	Frequency	Acceptance Criteria	Corrective Action
Method Detection Limit (MDL) Study; run at an analyte concentration near to but lower than the reporting limit (RL)	As needed; at minimum every 6 months.	Positive result < the analyte reporting limit (RL).	Determine the reason for failure and fix problem with the system. Repeat the MDL study. If criteria still not met, discuss with QA Manager (RL may be adjusted if required).
LCR Study	As needed, at minimum every 6 months.	All verification data must agree within ±10% of expected values.	Determine the reason for failure and fix problem with the system. Repeat the LCR study. If criteria still not met, discuss with QA Manager.

9222 B. Standard Total Coliform Membrane Filter Procedure

1. Laboratory Apparatus

For MF analyses use glassware and other apparatus composed of material free from agents that may affect bacterial growth.

a. Sample bottles: See Section 9030B.18.

b. Dilution bottles: See Section 9030B.13.

c. Pipets and graduated cylinders: See Section 9030B.9. Before sterilization, loosely cover opening of graduated cylinders with metal foil or a suitable heavy wrapping-paper substitute. Immediately after sterilization secure cover to prevent contamination.

d. Containers for culture medium: Use clean borosilicate glass flasks. Any size or shape of flask may be used, but erlenmeyer flasks with metal caps, metal foil covers, or screw caps provide for adequate mixing of the medium contained and are convenient for storage.

e. Culture dishes: Use sterile borosilicate glass or disposable, presterilized plastic petri dishes, 60 × 15 mm, 50 × 9 mm, or other appropriate size. Wrap convenient numbers of clean, glass culture dishes in metal foil if sterilized by dry heat, or suitable heavy wrapping paper when autoclaved. Incubate loose-lidded glass and disposable plastic culture dishes in tightly closed containers with wet paper or cloth to prevent moisture evaporation with resultant drying of medium and to maintain a humid environment for optimum colony development.

Presterilized disposable plastic dishes with tight-fitting lids that meet the specifications above are available commercially and are used widely. Reseal opened packages of disposable dish supplies for storage.

f. Filtration units: The filter-holding assembly (constructed of glass, autoclavable plastic, porcelain, or stainless steel) consists of a seamless funnel fastened to a base by a locking device or by magnetic force. The design should permit the membrane filter to be held securely on the porous plate of the receptacle without mechanical damage and allow all fluid to pass through the membrane during filtration. Discard plastic funnels with deep scratches on inner surface or glass funnels with chipped surfaces.

Wrap the assembly (as a whole or separate parts) in heavy wrapping paper or aluminum foil, sterilize by autoclaving, and store until use. Alternatively expose all surfaces of the previously cleaned assembly to ultraviolet radiation (2 min exposure) for the initial sanitization before use in the test procedure, or before reusing units between successive filtration series. Field units may be sanitized by dipping or spraying with alcohol and then igniting or immersing in boiling water for 2 min. After submerging unit in boiling water, cool it to room temperature before reuse. Do not ignite plastic parts. Sterile, disposable field units may be used.

For filtration, mount receptacle of filter-holding assembly on a 1-L filtering flask with a side tube or other suitable device (manifold to hold three to six filter assemblies) such that a pressure

differential (34 to 51 kPa) can be exerted on the filter membrane. Connect flask to a vacuum line, an electric vacuum pump, a filter pump operating on water pressure, a hand aspirator, or other means of securing a pressure differential (138 to 207 kPa). Connect a flask of approximately the same capacity between filtering flask and vacuum source to trap carry-over water.

g. Membrane filter: Use membrane filters (for additional specifications, see Section 9020) with a rated pore diameter such that there is complete retention of coliform bacteria. Use only those filter membranes that have been found, through adequate quality control testing and *certification by the manufacturer*, to exhibit: full retention of the organisms to be cultivated, stability in use, freedom from chemical extractables that may inhibit bacterial growth and development, a satisfactory speed of filtration (within 5 min), no significant influence on medium pH (beyond ± 0.2 units), and no increase in number of confluent colonies or spreaders compared to control membrane filters. Use membranes grid-marked in such a manner that bacterial growth is neither inhibited nor stimulated along the grid lines when the membranes with entrapped bacteria are incubated on a suitable medium. Preferably use fresh stocks of membrane filters and if necessary store them in an environment without extremes of temperature and humidity. Obtain no more than a year's supply at any one time.

Preferably use presterilized membrane filters for which the manufacturer has certified that the sterilization technique has neither induced toxicity nor altered the chemical or physical properties of the membrane. If membranes are sterilized in the laboratory, autoclave for 10 min at 121°C. At the end of the sterilization period, let the steam escape rapidly to minimize accumulation of water of condensation on filters.

h. Absorbent pads consist of disks of filter paper or other material certified for each lot by the manufacturer to be of high quality and free of sulfites or other substances of a concentration that could inhibit bacterial growth. Use pads approximately 48 mm in diameter and of sufficient thickness to absorb 1.8 to 2.2 mL of medium. Presterilized absorbent pads or pads subsequently sterilized in the laboratory should release less than 1 mg total acidity (calculated as $CaCO_3$) when titrated to the phenolphthalein end point, pH 8.3, using 0.02*N* NaOH and produce pH levels of 7 ± 0.2. Sterilize pads simultaneously with membrane filters available in resealable kraft envelopes, or separately in other suitable containers. Dry pads so they are free of visible moisture before use. See sterilization procedure described for membrane filters above and Section 9020 for additional specifications on absorbent pads.

i. Forceps: Smooth flat forceps, without corrugations on the inner sides of the tips. Sterilize before use by dipping in 95% ethyl or absolute methyl alcohol and flaming.

j. Incubators: Use incubators to provide a temperature of 35 ± 0.5°C and to maintain a humid environment (60% relative humidity).

k. Microscope and light source: To determine colony counts on membrane filters, use a magnification of 10 to 15 diameters and a cool white fluorescent light source adjusted to give maximum sheen discernment. Optimally use a binocular wide-field dissecting microscope. Do not use a microscope illuminator with optical system for light concentration from an

incandescent light source for discerning coliform colonies on Endo-type media.

2. Materials and Culture Media

The need for uniformity dictates the use of commercial dehydrated media. Never prepare media from basic ingredients when suitable dehydrated media are available. Follow manufacturer's directions for rehydration. Store opened supplies of dehydrated media in a desiccator. Commercially prepared media in liquid form (sterile ampule or other) may be used if known to give equivalent results. See Section 9020 for media quality control specifications.

Test each new medium lot against a previously acceptable lot for satisfactory performance as described in Section 9020B. With each new lot of Endo-type medium, verify a minimum 10% of coliform colonies, obtained from natural samples or samples with known additions, to establish the comparative recovery of the medium lot.

Before use, test each batch of laboratory-prepared MF medium for performance with positive and negative culture controls. Check for coliform contamination at the beginning and end of each filtration series by filtering 20 to 30 mL of dilution or rinse water through the filter. If controls indicate contamination, reject all data from affected samples and request resample.

a. LES Endo agar:#(1)

Yeast extract	1.2	g
Casitone or trypticase	3.7	g
Thiopeptone or thiotone	3.7	g
Tryptose	7.5	g
Lactose	9.4	g
Dipotassium hydrogen phosphate, K_2HPO_4	3.3	g
Potassium dihydrogen phosphate, KH_2PO_4	1.0	g
Sodium chloride, NaCl	3.7	g
Sodium desoxycholate	0.1	g
Sodium lauryl sulfate	0.05	g
Sodium sulfite, Na_2SO_3	1.6	g
Basic fuchsin	0.8	g
Agar	15.0	g
Reagent-grade water	1	L

Rehydrate product in 1 L water containing 20 mL 95% ethanol. Do not use denatured ethanol, which reduces background growth and coliform colony size. Bring to a near boil to dissolve agar, then promptly remove from heat and cool to 45 to 50°C. Do not sterilize by

autoclaving. Final pH 7.2 ± 0.2. Dispense in 5- to 7-mL quantities into lower section of 60-mm glass or plastic petri dishes. If dishes of any other size are used, adjust quantity to give an equivalent depth of 4 to 5 m. Do not expose poured plates to direct sunlight; refrigerate in the dark, preferably in sealed plastic bags or other containers to reduce moisture loss. Discard unused medium after 2 weeks or sooner if there is evidence of moisture loss, medium contamination, or medium deterioration (darkening of the medium).

b. M-Endo medium:†#(2)

Tryptose or polypeptone	10.0	g
Thiopeptone or thiotone	5.0	g
Casitone or trypticase	5.0	g
Yeast extract	1.5	g
Lactose	12.5	g
Sodium chloride, NaCl	5.0	g
Dipotassium hydrogen phosphate, K_2HPO_4	4.375	g
Potassium dihydrogen phosphate, KH_2PO_4	1.375	g
Sodium lauryl sulfate	0.05	g
Sodium desoxycholate	0.10	g
Sodium sulfite, Na_2SO_3	2.10	g
Basic fuchsin	1.05	g
Agar (optional)	15.0	g
Reagent-grade water	1	L

1) Agar preparation—Rehydrate product in 1 L water containing 20 mL 95% ethanol. Heat to near boiling to dissolve agar, then promptly remove from heat and cool to between 45 and 50°C. Dispense 5- to 7-mL quantities into 60-mm sterile glass or plastic petri dishes. If dishes of any other size are used, adjust quantity to give an equivalent depth. Do not sterilize by autoclaving. Final pH should be 7.2 ± 0.2. A precipitate is normal in Endo-type media.

Refrigerate finished medium in the dark and discard unused agar after 2 weeks.

2) Broth preparation—Prepare as above, omitting agar. Dispense liquid medium (at least 2.0 mL per plate) onto absorbent pads (see absorbent pad specifications, Section 9222B.1) and carefully remove excess medium by decanting the plate. The broth may have a precipitate but this does not interfere with medium performance if pads are certified free of sulfite or other toxic agents at a concentration that could inhibit bacterial growth. Refrigerated broth may be stored for up to 4 d.

c. Buffered dilution rinse water: See Section 9050C.1.

3. Samples

Collect samples as directed in Section 9060A and Section 9060B.

4. Coliform Definition

Bacteria that produce a red colony with a metallic (golden) sheen within 24 h incubation at 35°C on an Endo-type medium are considered members of the coliform group. The sheen may cover the entire colony or may appear only in a central area or on the periphery. The coliform group thus defined is based on the production of aldehydes from fermentation of lactose. While this biochemical characteristic is part of the metabolic pathway of gas production in the multiple-tube test, some variations in degree of metallic sheen development may be observed among coliform strains. However, this slight difference in indicator definition is not considered critical to change its public health significance, particularly if suitable studies have been conducted to establish the relationship between results obtained by the MF and those obtained by the standard multiple-tube fermentation procedure.

5. Procedures

a. Selection of sample size: Size of sample will be governed by expected bacterial density. In drinking water analyses, sample size will be limited only by the degree of turbidity or by the noncoliform growth on the medium (Table 9222:I). For regulation purposes, 100 mL is the official sample size.

An ideal sample volume will yield 20 to 80 coliform colonies and not more than 200 colonies of all types on a membrane-filter surface. Analyze drinking waters by filtering 100 to 1000 mL, or by filtering replicate smaller sample volumes such as duplicate 50-mL or four replicates of 25-mL portions. Analyze other waters by filtering three different volumes (diluted or undiluted), depending on the expected bacterial density. See Section 9215B.2 for preparation of dilutions. When less than 10 mL of sample (diluted or undiluted) is to be filtered, add approximately 10 mL sterile dilution water to the funnel before filtration or pipet the sample volume into a sterile dilution bottle, then filter the entire dilution. This increase in water volume aids in uniform dispersion of the bacterial suspension over the entire effective filtering surface.

b. Sterile filtration units: Use sterile filtration units at the beginning of each filtration series as a minimum precaution to avoid accidental contamination. A filtration series is considered to be interrupted when an interval of 30 min or longer elapses between sample filtrations. After such interruption, treat any further sample filtration as a new filtration series and sterilize all membrane filter holders in use. See Section 9222B.1 *f* for sterilization procedures and Section 9020B.3*m* and *n* for UV cleaning and safety guidelines.

c. Filtration of sample: Using sterile forceps, place a sterile membrane filter (grid side up) over porous plate of receptacle. Carefully place matched funnel unit over receptacle and lock it in place. Filter sample under partial vacuum. With filter still in place, rinse the interior surface of the funnel by filtering three 20- to 30-mL portions of sterile dilution water. Alternatively, rinse funnel by a flow of sterile dilution water from a squeeze bottle. This is satisfactory only if the

squeeze bottle and its contents do not become contaminated during use. Rinsing between samples prevents carryover contamination. Upon completion of final rinse and the filtration process disengage vacuum, unlock and remove funnel, immediately remove membrane filter with sterile forceps, and place it on selected medium with a rolling motion to avoid entrapment of air. If the agar-based medium is used, place prepared filter directly on agar, invert dish, and incubate for 22 to 24 h at 35 ± 0.5°C.

If liquid medium is used, place a pad in the culture dish and saturate with at least 2.0 mL M-Endo medium and carefully remove excess medium by decanting the plate. Place prepared filter directly on pad, invert dish, and incubate for 22 to 24 h at 35 ± 0.5°C.

Differentiation of some colonies from either agar or liquid medium substrates may be lost if cultures are incubated beyond 24 h.

Insert a sterile rinse water sample (100 mL) after filtration of a series of 10 samples to check for possible cross-contamination or contaminated rinse water. Incubate the rinse water control membrane culture under the same conditions as the sample.

For nonpotable water samples, preferably decontaminate filter unit after each sample (as described above) because of the high number of coliform bacteria present in these samples. Alternatively, use an additional buffer rinse of the filter unit after the filter is removed to prevent carryover between samples.

d. Alternative enrichment technique: Place a sterile absorbent pad in the lid of a sterile culture dish and pipet at least 2.0 mL lauryl tryptose broth, prepared as directed in Section 9221B.1.*a*1), to saturate pad. Carefully remove any excess liquid from absorbent pad by decanting plate. Aseptically place filter through which the sample has been passed on pad. Incubate filter, without inverting dish, for 1.5 to 2 h at 35 ± 0.5°C in an atmosphere of at least 60% relative humidity.

If the agar-based Endo-type medium is used, remove enrichment culture from incubator, lift filter from enrichment pad, and roll it onto the agar surface, which has been allowed to equilibrate to room temperature. Incorrect filter placement is at once obvious, because patches of unstained membrane indicate entrapment of air. Where such patches occur, carefully reseat filter on agar surface. If the liquid medium is used, prepare final culture by removing enrichment culture from incubator and separating the dish halves. Place a fresh sterile pad in bottom half of dish and saturate with at least 2.0 mL of M-Endo medium and carefully remove excess liquid from absorbent pad by decanting plate. Transfer filter, with same precautions as above, to new pad. Discard used enrichment pad.

With either the agar or the liquid medium, invert dish and incubate for 20 to 22 h at 35 ± 0.5°C. Proceed to ¶ e below.

e. Counting: To determine colony counts on membrane filters, use a low-power (10 to 15 magnifications) binocular wide-field dissecting microscope or other optical device, with a cool white fluorescent light source directed to provide optimal viewing of sheen. The typical coliform colony has a pink to dark-red color with a metallic surface sheen. Count both typical and

atypical coliform colonies. The sheen area may vary in size from a small pinhead to complete coverage of the colony surface. Atypical coliform colonies can be dark red, mucoid, or nucleated without sheen. Generally pink, blue, white, or colorless colonies lacking sheen are considered noncoliforms. The total count of colonies (coliform and noncoliform) on Endo-type medium has no consistent relationship to the total number of bacteria present in the original sample. A high count of noncoliform colonies may interfere with the maximum development of coliforms. Refrigerating cultures (after 22 h incubation) with high densities of noncoliform colonies for 0.5 to 1 h before counting may deter spread of confluence while aiding sheen discernment.

Samples of disinfected water or wastewater effluent may include stressed organisms that grow relatively slowly and produce maximum sheen in 22 to 24 h. Organisms from undisinfected sources may produce sheen at 16 to 18 h, and the sheen subsequently may fade after 24 to 30 h.

f. Coliform verification: Occasionally, typical sheen colonies may be produced by noncoliform organisms and atypical colonies (dark red or nucleated colonies without sheen) may be coliforms. Preferably verify all typical and atypical colony types. For drinking water, verify all suspect colonies by swabbing the entire membrane or pick at least five typical colonies and five atypical colonies from a given membrane filter culture. For waters other than drinking water, at a minimum, verify at least 10 sheen colonies (and representative atypical colonies of different morphological types) from a positive water sample monthly. See Section 9020B.8. Based on need and sample type, laboratories may incorporate more stringent quality control measures (e.g., verify at least one colony from each typical or atypical colony type from a given membrane filter culture, verify 10% of the positive samples). Adjust counts on the basis of verification results. Verification tests are listed below.

1) Lactose fermentation—Transfer growth from each colony or swab the entire membrane with a sterile cotton swab (for presence-absence results in drinking water samples) and place in lauryl tryptose broth; incubate the lauryl tryptose broth at 35 ± 0.5°C for 48 h. Gas formed in lauryl tryptose broth and confirmed in brilliant green lactose broth (Section 9221B.2 for medium preparation) within 48 h verifies the colony as a coliform. Simultaneous inoculation of both media for gas production is acceptable. Inclusion of EC broth inoculation for 44.5 ± 0.2°C incubation will provide information on the presence of fecal coliforms. Use of EC-MUG with incubation at 44.5 ± 0.2°C for 24 h will provide information on presence of *E. coli*. See Section 9222G for MF partition procedures.

2) Alternative coliform verifications—Apply this alternative coliform verification procedure to isolated colonies on the membrane filter culture. If a mixed culture is suspected or if colony separation is less than 2 mm, streak the growth to M-Endo medium or MacConkey agar to assure culture purity or submit the mixed growth to the fermentation tube method.

a) Rapid test—A rapid verification of colonies utilizes test reactions for cytochrome oxidase (CO) and β-galactosidase. Coliform reactions are CO negative and β-galactosidase positive within 4 h incubation of tube culture or micro (spot) test procedure.

b) Commercial multi-test systems—Verify the colony by streaking it for purification,

selecting a well-isolated colony, and inoculating into a multi-test identification system for Enterobacteriaceae that includes lactose fermentation and/or β-galactosidase and CO test reactions.

6. Calculation of Coliform Density

Compute the count, using membrane filters with 20 to 80 coliform colonies and not more than 200 colonies of all types per membrane, by the following equation:

$$\text{(Total) coliforms/100 mL} = \frac{\text{coliform colonies counted} \times 100}{\text{mL sample filtered}}$$

If no coliform colonies are observed, report the coliform colonies counted as ''<1 coliform/100 mL.''

For verified coliform counts, adjust the initial count based upon the positive verification percentage and report as ''verified coliform count/100 mL.''

$$= \frac{\text{number of verified colonies}}{\text{total number of coliform colonies subjected to verification}} \times 100$$

a. Water of drinking water quality: While the EPA Total Coliform Rule for public water supply samples requires only a record of coliform presence or absence in 100-mL samples, it may be advisable to determine coliform densities in repeat sampling situations. This is of particular importance when a coliform biofilm problem is suspected in the distribution system. Quantitative information may provide an indication of the magnitude of a contaminating event.

With water of good quality, the occurrence of coliforms generally will be minimal. Therefore, count all coliform colonies (disregarding the lower limit of 20 cited above) and use the formula given above to obtain coliform density.

If confluent growth occurs, covering either the entire filtration area of the membrane or a portion thereof, and colonies are not discrete, report results as ''confluent growth with (or without) coliforms.'' If the total number of bacterial colonies, coliforms plus noncoliforms, exceeds 200 per membrane, or if the colonies are not distinct enough for accurate counting, report results as ''too numerous to count'' (TNTC) or ''confluent,'' respectively. For drinking water, the presence of coliforms in such cultures showing no sheen may be confirmed by either transferring a few colonies or placing the entire membrane filter culture into a sterile tube of brilliant green lactose bile broth. As an alternative, brush the entire filter surface with a sterile loop, applicator stick, or cotton swab and inoculate this growth to the tube of brilliant green lactose bile broth. If gas is produced from the brilliant green bile broth tube within 48 h at 35 ± 0.5°C, coliforms are present. For compliance with the EPA Total Coliform Rule, report confluent

growth or TNTC with at least one detectable coliform colony (which is verified) as a total coliform positive sample. Report confluent growth or TNTC without detectable coliforms as invalid. For invalid samples, request a new sample from the same location within 24 h and select more appropriate volumes to be filtered per membrane, observing the requirement that the standard drinking water portion is 100 mL, or choose another coliform method that is less subject to heterotrophic bacterial interferences. Thus, to reduce interference from overcrowding, instead of filtering 100 mL per membrane, filter 50-mL portions through two separate membranes, 25-mL portions through each of four membranes, etc. Total the coliform counts observed on all membranes and report as number per 100 mL.

b. Water of other than drinking water quality: As with potable water samples, if no filter has a coliform count falling in the ideal range, total the coliform counts on all filters and report as number per 100 mL. For example, if duplicate 50-mL portions were examined and the two membranes had five and three coliform colonies, respectively, report the count as eight coliform colonies per 100 mL, i.e.,

$$\frac{[(5 + 3) \times 100]}{(50 + 50)} = 8 \text{ coliforms/100 mL}$$

Similarly, if 50-, 25-, and 10-mL portions were examined and the counts were 15, 6, and <1 coliform colonies, respectively, report the count as 25/100 mL, i.e.,

$$\frac{[(15 + 6 + 0) \times 100]}{(50 + 25 + 10)} = 25 \text{ coliforms/100 mL}$$

On the other hand, if 10-, 1.0-, and 0.1-mL portions were examined with counts of 40, 9, and <1 coliform colonies, respectively, select the 10-mL portion only for calculating the coliform density because this filter had a coliform count falling in the ideal range. The result is 400/100 mL, i.e.,

$$\frac{(40 \times 100)}{10} = 400 \text{ coliforms/100 mL}$$

In this last example, if the membrane with 40 coliform colonies also had a total bacterial colony count greater than 200, report the coliform count as ≥400/100 mL.

Report confluent growth or membranes with colonies too numerous to count as described in *a* above. Request a new sample and select more appropriate volumes for filtration or utilize the

multiple-tube fermentation technique.

c. Statistical reliability of membrane filter results: Although the precision of the MF technique is greater than that of the MPN procedure, membrane counts may underestimate the number of viable coliform bacteria. Table 9222:II illustrates some 95% confidence limits. These values are based on the assumption that bacteria are distributed randomly and follow a Poisson distribution. For results with counts, *c*, greater than 20 organisms, calculate the approximate 95% confidence limits using the following normal distribution equations:

$$\text{Upper limit} = c + 2\sqrt{c} \quad \text{Lower limit} = c - 2\sqrt{c}$$

7. Bibliography

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Endnotes

1 (Popup - Footnote)

* Dehydrated Difco M-Endo Agar LES (No. 0736), dehydrated BBL M-Endo Agar LES (No. 11203), or equivalent.

2 (Popup - Footnote)

† Dehydrated Difco M-Endo Broth MF (No. 0749), dehydrated BBL *m*-Coliform Broth (No. 11119), or equivalent may be used if absorbent pads are used.

Total Coliform Detection in Potable Water
Method: 9131
Reagents: Colilert-24 (IDEXX)
Collier- 18 (18 hour test) (IDEXX)
Coli tag-(CPI Intl.)

Scope: Rapid detection of Total coliform and simultaneous E.Coli detection in drinking water samples

Procedure:

Samples received to be on ice representing 4 degrees C cooling. Chain of custody for samples to be filled completely, with date time location, billing and reporting agencies. Chlorine <1.0 mg/L (PPM) Sodium Thiosulfate used to remove chlorine is in the whirlpac bags. Hold Time for samples are 30 Hours

RDM- rapid detection media will be used in this procedure. All Medias listed above use the same technology for detection.

Add contents of one snap pack RDM to 100 ml of sample. Whirl pack bag and securely fasten. Shake to dissolve media.

Incubate samples for 24 hours (18 Hours for Colilert 18) at 35 Degrees C +/- .5 Degrees

After incubation, compare sample vial to comparator vial for color change. If the sample vial is less yellow than the comparator vial, the sample is considered negative for total coliform. Any increase in color intensity from the comparator vial, indicates a positive total coliform sample.

To check for the inclusion of E. coli in the positive samples. Verification will be done using a 6 watt Ultraviolet radiation lamp, along with a positive control comparator. Samples that are positive for Total Coliform are compared under UV light. A blue luminescence indicates a Positive E.Coli in the positive Total coliform sample.

Positive Total Coliform and E. coli samples must be submitted to the proper authorities when testing for Public Water Systems.

Control Samples

No control samples are needed to be run on RDM Media. Controls have been run by the manufacturer and are available for review by lot number on their respective websites.

Detection Limit- 1 CFU/ 100 ml

Colilert/ Colilert 18
www.idexx.om
Colitag
www.cpiinternational.com

New Lots of media must be documented on the media chart with
Name
Manufacturer
Date Received
LOT number
Certificate of acceptability (yes or no)
Expiration Date

This data can ensure that a back log audit can be performed in the event of an Out-of –Control data situation.

Reviewed by

_____ Date _____

Heterotrophic Plate Count
Method 9124

Reagents: HPC Media (prepared, 10 ml) (BBL)
Petri Plates (sterile)

Scope: Estimation of total number of live heterotrophic bacteria present in drinking water.

Procedure

Samples received to be on ice representing 4 degrees C cooling. Chain of custody for samples to be filled completely, with date time location, billing and reporting agencies. Chlorine <1.0 mg/L(PPM) Sodium Thiosulfate used to remove chlorine is in the whirlpac bags. Hold Time for samples are 8 Hours

Select the dilution of sample so that the total number of colonies on a plate will be between 30 and 300 colonies. Most plates for potable water will be using a 1 ml sample volume.

Prepare media by melting media at 45-55 degrees C in water bath. Temper media to 45 degrees. Pipette sample onto Petri dish using sterile techniques and appropriate dilutions. Prepare all Petri dished in replicates. Pour tempered HPC agar onto plate and mix gently using a cross hatch pattern and swirl pattern. Let solidify on a level surface. Place in incubator inverted Incubate plates for 48 hours at 35 degrees C. Count plates immediately using a light box and colony counter.

To compute the HPC for the sample, count both plates and record each count. Add the counts together and divide by 2. Record the sample count to the bench worksheet.

Quality control samples for each sample run will be done using a negative control method. A blank will be run with each sample batch and incubated. A negative plate for the control ensures that no contamination of the media and dilution buffer was present at the time of the testing sequence. In the event of an out of control data, samples will be recollected and retested.

Detection Limit: <1 CFU/ ml to 300 CFU/ 0.1 ml

Media BBL Prepared HPC Media
www.weberscientific.com

New Lots of media must be documented on the media chart with
- Name
- Manufacturer
- Date Received
- LOT number
- Certificate of acceptability (yes or no)
- Expiration Date

This data can ensure that a back log audit can be performed in the event of an Out-of –Control data situation.

Reviewed by

_____ Date _____

Fecal Coliform in Wastewater/ Non Potable Water
Method- 1003
Reagents: Hach MFC with rosalic acid (HACH)
PALL Filter funnel kit with membrane filter (PALL)

Scope: Detection of Fecal coliform bacteria in wastewater and

non potable water

Procedure:

Samples received to be on ice representing 4 degrees C cooling. Chain of custody for samples to be filled completely, with date time location, billing and reporting agencies. Chlorine <1.0 mg/L (PPM) Sodium Thiosulfate used to remove chlorine is in the whirlpac bags. Hold Time for samples are 6 Hours

Select the dilution for the samples that will be run. Serial dilutions can be performed for unknown samples. Plates should have less than 200 colonies of growth to be accurately acceptable. Carefully unwrap a sterile PALL filter funnel and place it on the manifold for vacuum. Filter the appropriate size sample through to reveal an acceptable count. With the filter still in place flush the sides of the filter funnel with 20-30 ml portions of dilution buffer starting from top to bottom of filter while vacuum is still on the vessel. After all the water has been eliminated from the funnel, remove funnel and place red cap on bottom of funnel where it connected to the manifold. Remove filter sleeve and discard retaining the cap of the funnel for placement on top.

Open 2 ml ampoule of MFC reagent and inoculate the absorbent pad under the filter media. (Sterile tweezers may be used to lift the filter up to gain access. Control blanks at the beginning and end of each run are not necessary due to disposable filter funnels and membrane kits.

Place cover and snap into place on top of funnel plate. Be sure all areas are sealed. Invert plate and place it into the water bath @ 44.5 for 24 hours.

After incubation remove plates and review them on a lighted colony counter. Typical fecal coliform will be various shades of blue on the membrane. All other colonies observed are not counted as they are not fecal coliform.

Compute the count by the equation:
Fecal Coliform Colonies/100 ml= FC counted X 100 / ml sample

Detection Limit: <1 CFU/ ml to 300 CFU/ 0.1 ml

Media HACH Prepared MFC Media 2 ml ampoule
www.weberscientific.com
www.hach.com

New Lots of media must be documented on the media chart with
- Name
- Manufacturer
- Date Received
- LOT number
- Certificate of acceptability (yes or no)
- Expiration Date

This data can ensure that a back log audit can be performed in the event of an Out-of –Control data situation.

Reviewed by

_____ Date _____

M-Coliblue-24
Methods 8074, 8367*, and 10029** * USEPA accepted ** USEPA approved
Membrane Filtration Method m-ColiBlue24®, and Pseudomonas Broth
Scope and Application: potable water, no potable water, recreation water, and wastewater
Bacteria, Coliform

Introduction the Membrane Filtration (MF) method is a fast, simple way to estimate bacterial populations in water. The MF method is especially useful when evaluating large sample volumes or performing many coliform tests daily. Method In the initial step, an appropriate sample volume passes through a membrane filter with a pore size small enough (0.45 micron) to retain the bacteria present. The filter is placed on an absorbent pad (in a Petri dish) saturated with a culture medium that is selective for coliform growth. The Petri dish containing the filter and pad is incubated, upside down, for 24 hours at the appropriate temperature. After incubation, the colonies that have grown are identified and counted using a low-power microscope. Convenient Packaging Hatch's Pour Rite™ Ampoules contain prepared selective media. This eliminates the measuring, mixing, and autoclaving needed when preparing dehydrated media. The ampoules are designed with a large, unrestrictive opening that allows media to pour out easily. Simply break off the top of the ampoule and pour the medium onto an absorbent pad in a Petri dish. Each ampoule contains enough medium for one test. Medium packaged in Pour Rite Ampoules has a shelf-life of one year. Ampoules are shipped with a Certificate of Analysis and have an expiration date printed on the label. • When the sample is less than 20 mol (diluted or undiluted), add 10 mol of sterile dilution water to the filter funnel before applying the vacuum. This aids in distributing the bacteria evenly across the entire filter surface. • The volume of sample to be filtered will vary with the sample type. Select a maximum sample size to give 20 to 200 colony-forming units (CFU) per filter. The ideal sample volume of no potable water or wastewater for coliform testing yields 20–80 coliform colonies per filter. Generally, for finished, potable water, the volume to be filtered will be 100 Mr.

Bacteria, Coliform Methods 8074, 8367*, and 10029** * USEPA accepted ** USEPA approved Membrane Filtration Method m-ColiBlue24®, and Pseudomonas Broth Scope and Application: potable water, no potable water, recreation water, and wastewater

Tips and Techniques

Bacteria, Coliform Bacteria, Coliform Page 2 of 28 Bacterium Coliform.fm

Potable Water Procedures to test potable water with the MF Method, examine a 100-mL sample for total coliform by incubating a filter at 35 ±0.5 °C for 22–24 hours on m-Endo Broth. Coliform ferment lactose in the medium and produce an acid-aldehyde complex. This complex combines with Schiff's Reagent (also in the medium) to form an

iridescent green coating over the colonies. When magnified 10 to 15 times, coliform appear as dark red colonies with a greenish-gold sheen. 1. Place a sterile absorbent pad in a sterile Petri dish using sterilized forceps. Replace the lid. *Note: Do not touch the pad or the inside of the Petri dish. Note: To sterilize forceps, dip forceps in alcohol and flame in an alcohol or Bunsen burner. Let forceps cool before use. Note: For ease of use, Petri dishes with pads are available.* 2. Invert an m-Endo Broth Pour Rite Ampoule 2 to 3 times to mix the broth. Use the ampoule breaker (Cat. No. 24846-00) to break open the ampoule. Carefully pour the contents evenly over the absorbent pad. Replace the Petri dish lid. Repeat *step 1* and *step 2* for each Petri dish being prepared. 3. Set up the Membrane Filter Assembly. Use sterilized forceps to place a membrane filter, grid side up, into the assembly. 4. Shake the sample vigorously to mix. Pour 100 mol of sample into the funnel. Apply vacuum and filter the sample. Rinse the funnel walls three times with 20 to 30 mol of sterile buffered dilution water. 1)

Detection Limit: <1 CFU/ ml to 300 CFU/ 0.1 ml

Media HACH Prepared MFC Media 2 ml ampoule
www.weberscientific.com
www.hach.com

New Lots of media must be documented on the media chart with
- Name
- Manufacturer
- Date Received
- LOT number
- Certificate of acceptability (yes or no)
- Expiration Date

This data can ensure that a back log audit can be performed in the event of an Out-of –Control data situation.

Reviewed by

_____ Date _____