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FINAL QUALITY ASSURANCE PROJECT PLAN UNDERGROUND STORAGE TANK 00002 (UST 00002) REVISION 1 NSA CRANE IN
10/17/2012
VRHABILIS-KEMRON, LLC

12

FINAL
QUALITY ASSURANCE PROJECT PLAN
UST SITE 00002

NAVAL FACILITIES ENGINEERING COMMAND
NAVAL SUPPORT ACTIVITY CRANE
CRANE, INDIANA

Contract #: N40083-11-D-0032-0002

PREPARED FOR:

NAVAL SUPPORT ACTIVITY CRANE AND NAVFAC

PREPARED BY:

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REVISION 01

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LIST OF ACRONYMS

AHA	Air Force Base Conversion Agency
APP	Accident Prevention Plan
BFB	4-bromofuorobenzene
BTEX	Benzene, Toluene, Ethylbenzene, and Xylene
CA	Cost Analysis
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CD	Compact Disk
CHMM	Certified Hazardous Materials Manager
CLP	Contract Laboratory Program
COB	Close of Business
COC	Contaminant of Concern
COCs	Contaminants of Concern
COE	Corps of Engineers
COR	Contracting Officer's Representative
CRQL	Contract Required Quantitation Limit
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense
DQI	Data Quality Indicator
DRO	Diesel Range Organics
ECATTS	Environmental Compliance Assessment, Training, and Tracking System
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EMAC	Environmental Multiple Award Contract
EPA	Environmental Protection Agency
ESL	Ecological Screening Levels
FEAD	Facilities Engineering and Acquisition Department
GC	Gas Chromatography
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/FID	Gas Chromatography/Flame Ionization Detector
GC/MS	Gas Chromatography/Mass Spectrometry
GFAA	Graphite Furnace Atomic Absorption
GPS	Global Positioning System
GRO	Gasoline Range Organic
HCl	Hydrochloric Acid
HPLC	High Performance Liquid Chromatography
I/E	Internal/External
IAC	Indiana Administrative Code
ICAL	Initial Calibration
ICB/CCB	Initial Calibration Black/Continuing Calibration Blank
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
ICP-AES	Inductively Coupled Plasma/Atomic Emission Spectrometry
ICP-OES	Inductively Coupled Plasma/Atomic Emission Spectrometry
ICS	Interference Check Sample

ICV	Initial Calibration Verification
ICV/CCV	Initial Calibration Verification/Continuing Calibration Verification
ID	Identification
IDEM	Indiana Department of Environmental Management
IDWM	Indiana Department of Waste Management
II	Initial Investigation
IRIS	Integrated Risk Information System
KEMRON	KEMRON Environmental Services Inc.
LANT	Atlantic
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LLC	Limited Liability Corporation
LOD	Limit of Detection
LOQ	Limit of Quantitation
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicates
MSDS	Material Safety Data Sheets
MTBE	Methyl Ter-Butyl Ether
N/A	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NCR	Non-Conformance Report
NELAP	National Environmental Laboratory Accreditation Program
NFR	No Further Remediation
NIOSH	National Institute for Occupational Safety and Health
NIRIS	Naval Installation Restoration Information Solution
NSA	Naval Support Activity
NTR	Navy Technical Representative
OSHA	Occupational Safety & Health Administration
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PDS	Post Digestion Spike
PE	Professional Engineer
PG	Professional Geologist
PID	Photoionization Detector
PM	Project Manager
PMP	Project Management Professional
POC	Point Of Contact
PT	Proficiency Testing
QA	Quality Assurance
QA/QC	Quality Assurance / Quality Control
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality System Manual
RA	Remedial Action
RAOs	Remedial Action Objectives
RAWP	Remedial Action Work Plan
RCG	Remediation Closure Guide

RFI	RCRA Facility Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRF	Relative Response Factor
RSC	Record of Site Closure
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SIM	Single Ion Monitoring
SOP	Standard Operating Procedure
SPCCs	Spill Prevention, Control and Countermeasure Plans
SRs	Sample Records
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
SVOCs	Semi-Volatile Organic Compounds
SWMU	Solid Waste Management Unit
TAT	Turnaround Time
TBD	To Be Determined
TPH	Total Petroleum Hydrocarbons
TPH-DRO	Total Petroleum Hydrocarbons-Diesel Range Organics
TPH-GRO	Total Petroleum Hydrocarbons-Diesel Range Organics
UFP	Uniform Federal Policy
ug/L	Micrograms per Liter
US	United States
UST	Underground Storage Tank
UXO	Unexploded Ordinance
VOA	Volatile Organic Analyte
VOCs	Volatile Organic Compounds
VRHK	VRHabilis-KEMRON LLC Joint Venture
WAWF	Wide Area Work Flow

QAPP Worksheet #1 Title and Approval Page

Document Title:	FINAL Quality Assurance Project Plan
Lead Organization:	NAVFAC Midwest
Preparer's Name and Organizational Affiliation:	Daniel Robinson VRHabilis-KEMRON, LLC.
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Preparation Date:	17 October 2012

Authorizations:

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Larry Emerson, PE, PMP Project Manager VRHabilis-KEMRON, LLC.	10/19/2012 Date
Jeff Gunn, PMP Construction Manager VRHabilis-KEMRON, LLC.	10/19/2012 Date
Doug Griffin Project Manager IDEM Corrective Action Branch	Date
Thomas J. Brent Navy Environmental Restoration Site Manager Naval Support Activity Crane	Date

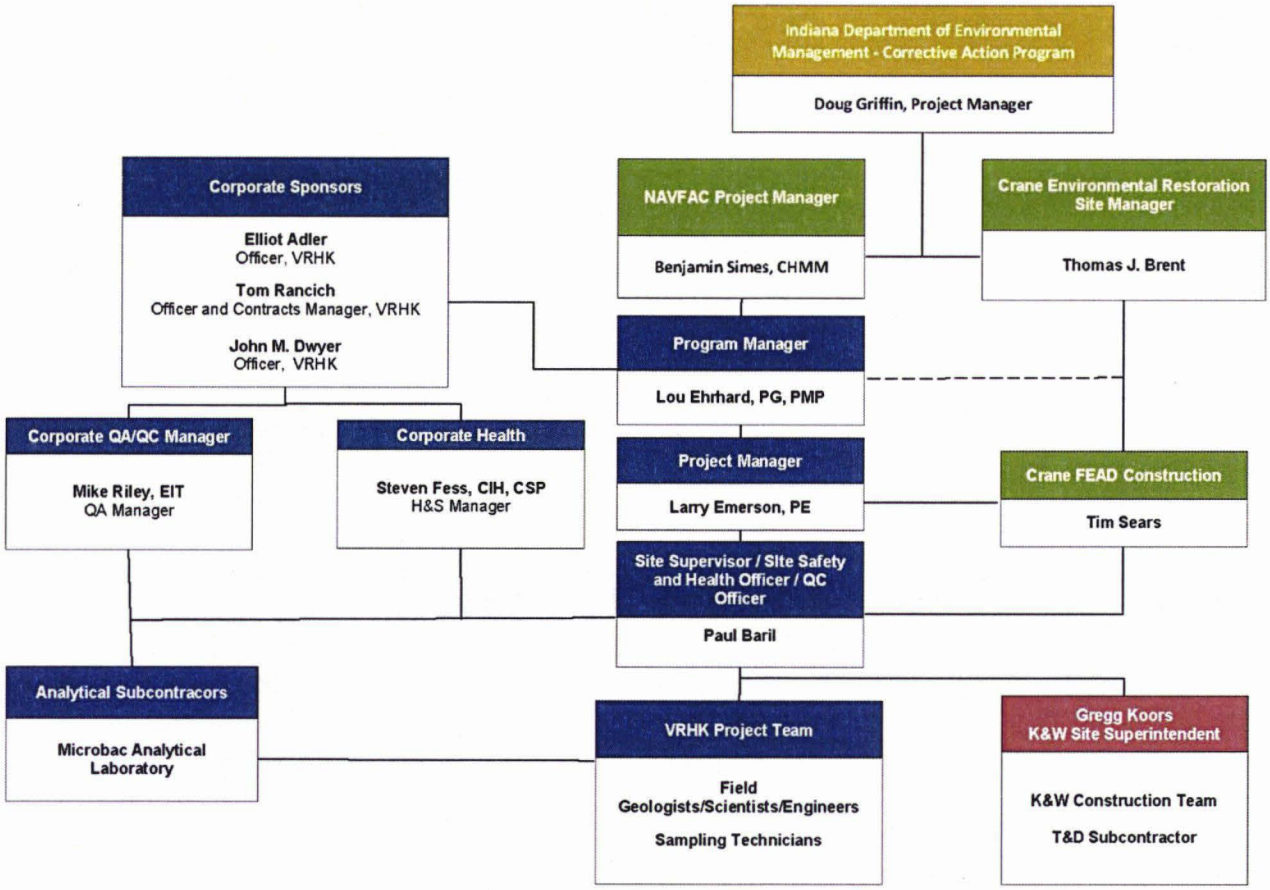
Document Title:	FINAL Quality Assurance Project Plan
Lead Organization:	NAVFAC Midwest
Preparer's Name and Organizational Affiliation:	Daniel Robinson VRHabilis-KEMRON, LLC.
Preparer's Address, Telephone Number, and E-mail Address:	11124 Kingston Pike Suite 119-260 Knoxville, Tennessee, 37934 Tel. 404-636-0928 Email: drobinson@kemron.com
Preparation Date:	17 October 2012

Authorizations:

Benjamin Simes, CHMM Navy Technical Representative, Project Manger Naval Support Activity Crane	Date
Janice Nielsen LANT QAM (Quality Assurance Manager) Naval Support Activity Crane	Date
Document Control Number:	Crane-PM-ATL

QAPP Worksheet #5 Project Organizational Chart

A project management organizational structure for this project is included to show the reporting structure for the project.



The VRHabilis-KEMRON project team is comprised of a Joint Venture between VRHabilis, LLC and KEMRON Environmental Services, Inc. This NAVFAC program will be led by Mr. Lou Ehrhard, an experienced Project/Program Manager. He will be supported by a staff of senior engineers, scientists, and remediation experts. He has selected Mr. Larry Emerson, a qualified Project Manager and Professional Engineer, to manage the UST Project for the Naval Support Activity Crane, Crane, IN. Mr. Ehrhard has granted Mr. Emerson the authority to work directly with the Navy and the installations with his technical and contractual oversight. As the Project Manager assigned to this task order, Mr. Emerson will be responsible for all day to day activities and will coordinate all activities with Thomas Brent, the Navy Environmental Restoration Site Manager, and Mr. Benjamin Simes, the Navy Project Manager. He will have work stoppage authority or will authorize a field supervisor to have work stoppage authority in his absence from the field. He will ensure that the appropriate resources are coordinated by the Project QA Officer to provide project QC on all documents, and field work.

In addition, VRHK has a highly skilled staff of diverse environmental professionals to provide technical and field support to Mr. Emerson as needed throughout the performance of the project. In addition to the key personnel assigned, we have selected a project team which will include a Project Quality Control Officer, Field Manger, Health & Safety Manager, Site Safety and Health Officer (SSHO), Project Quality

Control Manager, and a Field Data Collection Specialist. All of the key and project personnel have relevant and recent experience with the removal of USTs.

Microbac Laboratories, Inc. (Microbac) in Marietta, Ohio will perform the majority of the chemical analyses for this project. Microbac is a full service environmental laboratory facility for analysis of groundwater, municipal and industrial waste water, soil, and solid waste. A laboratory certification requirement for the State of Indiana is not required by the State. Microbac is a certified by the National Environmental Laboratory Accreditation Program (NELAP). Microbac is a Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) Certified Laboratory. The Laboratory Certification is included in Appendix A.

KEMRON will select additional subcontractors as the project proceeds for supporting activities such as transportation, drilling, disposal, surveying, and other specialty subcontracting needs. These subcontractors will be selected by competitive bidding or by qualifications. Applicable State and local licensure and/or certifications will be included in the assessment of qualifications.

QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number & E-mail	Procedure (Timing, Pathways, etc.)
Point of Contact with IDEM	Navy NTR	Benjamin SImes	847-688-2600 ext.320 Benjamin.simes@navy.mil	All appropriate materials and information about the project will be forwarded to the IDEM representatives by the Navy RPM. Submittals to IDWM will be made by the Crane Environmental Restoration Site Manager.
Manage all Project Phases	Contractor PM	Lou Ehrhard	847-266-1350 lehrhard@kemron.com	Notify Navy NTR and Restoration Manager by phone and email of field related problems by COB of the next business day. All technical questions and scheduled field work will be coordinated through the Navy NTR
Overall QA Manager	VRH-KEMRON's Corporate QA Manager	Mike Riley	404-636-0928 mriley@kemron.com	Overall responsibility for ensuring QAPP compliance
Field and Analytical Corrective Actions	VRH-KEMRON Quality Control Officer	Paul Baril	814-823-5385 Paul.baril@vrhabilis.com	The need for corrective action for field and analytical issues will be determined by the QC Officer and PM.
Reporting Lab Data Quality Issues	Microbac Project Manager	Stephanie Mossburg	740-373-4071 smossburg@microbac.com	All QA/QC issues with project field samples will be reported by Microbac to the task manager and/or field team leader upon identification and no later than 2 business days from identification.
QAPP Amendments	NAVFAC COR, IDEM Project Manager			Any major changes to the QAPP must be approved by NAVFAC and IDEM before the changes can be implemented.[1]

[1]Major changes to the QAPP that would require pre-approval include using alternative SOPs from those provided in the document.

QAPP Worksheet #9 Project Scoping Session Participants Sheet

Project Name:	SWMU 9 UST Project
Site Names:	Former Gas Station Areas: East of Building B-150 • Includes USTs: B7, B8, and Former B9 South of Building B-55 (Former Heavy Equipment Garage) • Includes USTs: B26 and B27.
Site Locations:	Naval Support Activity Crane Crane, Indiana
Projected Date(s) of Sampling:	December 2012
Project Manager:	Larry Emerson
Date of Session:	August 28, 2012
Scoping Session Purpose:	Project Kickoff Meeting - General Discussion, Coordination, and Resolution of Issues

Attendees

Name	Title	Affiliation	Phone Number & E-mail	Project Role
Benjamin Simes	Navy Project Manager and Technical Representative,	Navy	847-688-2600 Benjamin.simes@navy.mil	Lead Organization Contracting Officer's Representative & RPM
Thomas Brent	Navy Environmental Restoration Manager	Navy	812-854-6160 Thomas.brent@navy.mil	POC for NSA Crane. COR Support
Marc McFarland	NAVFAC Safety	Navy	812-854-5015 Marc.mcfarland@navy.mil	Construction and Safety Manager
Jared Cornelius	Storage, NAVFAC	Navy	812-854-2876 Jared.cornelius@navy.mil	UST Specialist NAVFAC
Tim Sears	FEAD	Navy	812-854-2448 Tim.sears@navy.mil	Point of Contact for Crane
Lou Erhard	Program Manager	VRHK	847-266-1350 lehrhard@kemron.com	Program Manager
Larry Emerson	Project Manager	VRHK	847-266-1350 lemerson@kemron.com	Coordinates RA planning, field activities and reporting
Teri Lu	Field Manager	VRHK	920-836-23-24 Teri.lu@vrhabilis.com	Coordinates RA field implementation
Paul Baril	SSHO	VRHK	814-823-5385 Paul.baril@vrhabilis.com	Supervisor of RA implementation
Keith Welch	Certified UST Contractor	K&W Fueling Systems	765-932-4980 kwelch@kwfuelsystems.com	Conducts RA implementation

Comments/Decisions:	**General introductions and comments** 1. Be on time or early. Respect everyone's schedule when making changes to meetings, deadlines, setting up conference calls, etc. 2. Meet all deadlines for reports and milestones.

3. Communication has been a problem with other EMAC contractors. **In all cases, include Ben, Tom and Tim in all correspondence.** At times, Jared may be included in the list.
4. If a meeting is scheduled and someone cannot make it, stick to the schedule as much as possible.
5. Proposals are to be tabbed and presented in a 3 ring binder. No comb binding, please. 1 in advance, 3 copies plus 1
6. AT&T is the only cellphone tower on base. Expect reception problems.
7. RapidGate continues to be a problem. Requirement has been waived. Contractors will get all names to Tim at least a week ahead of time to set up proper access.
8. Any changes in key personnel must be disclosed in writing and sent to Ken Seymour, Ben, Tom and Tim.
9. All contractors and subs need to complete the ECATTS training available on line at navfac.ecatts.com. Password is NAVFAC, click EMS. All working on site must have proof of compliance.

Project Overview, Changes,

1. It's believed that this project documentation will be **Corrective Action** instead of UST. May schedule a phone conference on Monday to discuss
2. **What was in the tanks**? #6 fuel oil (heating oil is not regulated). At the fueling station, gasoline is suspected.
3. **Groundwater** - possibly only 2 wells will be needed - discussion about bedrock depth - may be greater than 30' but a toss-up. Tim said they recently did not hit rock although they may only have been at 15'.
4. Currently, ground is very dry due to the drought. Fall may be wetter.
5. **Any trucks -** weigh in and out. Tim would appreciate a heads up when the traffic is coming and going.
6. **For the closure**, VRHK is welcome to do the research but the submittal will be done by NSA Crane personnel
7. **Tanks are covered** with soil. May have water, old fuel or gas or maybe even sand inside.
8. **MSDS sheets** must be submitted 1-2 weeks before mobilization, and copies kept on site during the field work. Include all materials, even WD-40. Do not need to include sunscreen or insect spray if it is for personal use only.
9. This will be a **Level D job site** - steel toes, hard hats, hiviz clothing.
10. **Fire Permits** can be obtained from Larry Web (812-854-1235). Call and they'll come right out. Permit is good for 30 days. Need to tell him all we are doing. They will designate a smoking space.
11. **Fire extinguishers** required per OSHA regulations on site and in all equipment
12. **Trucks and excavators** - the fewer pieces on site at one time the better. Most fueling and all regular maintenance will be done off site. Only fluids will be truck mounted nurse tanks for refueling. All spill prevention regulations will be observed.
13. **Confined space program** not necessary since there is no reason or plan for anyone to enter the tanks. They will be pumped out and defumed in accordance with OSHA and IDEM regs.
14. **Roll offs** will be brought in for hauling off the tank pieces. Tank will be broken up and cut up in the hole after pumping and defuming then dug out like soil.
15. **Excavated soil** will be placed on visqueen and covered to protect

from wind and rain. May be required to line the excavation with visqueen if rain is predicted.

16. **Trucks** will be placed on visqueen only if contaminated, otherwise dry sweep before leaving the site.
17. **Digging Permit** - Notify Tim at least 48 hours beforehand. Flag site with white flags of the approximate dig zone.
18. We mark work site but NSA Crane personnel will **locate utilities**. Expect gas, electric, water. Field crew should dig with the anticipation of hitting something.
19. **Shoring** - system from McAllister. Sheet piling.
20. **APP** must address trenching, excavation and shoring
21. This is a **HAZWOPER** site. All are required to possess 40 hr and current certificate.
22. **Camera passes -** send Tim an email with camera owner's name, camera make and model/serial number for approval. Must keep it at all times.
23. Tim expects **Daily Reports**. Need to submit to Tim, Tom and Ben. End of the week photos. Paul is familiar with the format that includes the Three Phase Inspection Program.
24. WAWF requires **certified payroll** since it is Davis Bacon.
25. **Yellow warnings** required around the job site, orange **barricades.** There will be base workers needing to access the Boiler House.
26. **Piping** to be removed - at least to the gas island and everything outside the boiler house
27. **Conversion factor** - Tom uses 1.4 tons/CY, we may have used 1.5 but will check.
28. **Excavations** done 100-200 feet away ran into RR rails.
29. If **UXO** is found, work stops immediately. Paul will notify Tim.
30. **Manifests** - Tom signs the tickets and needs to know ahead of time. He will use a 30 day turnaround time for manifests and weight tickets.
31. **All project paperwork** - manifests, weight tickets, daily reports, photos, etc., will be submitted at the end of the project all at one time to Tim, Ben and Tom. Paul will submit end of the week overview by email with photos.
32. **IDEM** will not be on site when we remove the tanks
33. Since the tanks are not registered, a letter is needed that says all fees have been waived, and then file permit for removal at least a month before mobilization.
34. **For anyone driving on the base** - must have proof of insurance and no felony within last 5 years. DUI is counted as a "felony."
35. All vehicles must have valid registration and current insurance.
36. Plan for quick TAT for soil sample analyticals, 2 day TAT if possible, to avoid extended periods of open holes.

Action Items:

Plans and submittals

1. Work Plan submittal planned for early September
2. UFP QAPP, APP, Work Plan - Tom will review the disposal, erosion control plans, Navy chemist will also be reviewing UFP QAPP and we need to upload to website
3. UFP QAPP - +/- 10% samples for approval
4. Tom- prefers to submit work plans with the state signed by his Navy boss
5. APP needs to address sheet piling, trenching and shoring plan

6. Work Plan and UFP QAPP will go to the state. APP will not.
7. APP, MSDS, SSHP and AHAs will go to Marc and Tim. APP and AHAs should be separate
8. All plans should include a transmittal sheet with approval box. Tim will provide an example
9. **NAVFAC Safety Shack** is an excellent resource
10. Documents that will go to Tim
    a. Paul's Qualification Portfolio
    b. Certificates of training for all workers on site - HAZWOPER, ECATTS,
    c. Certificate of Insurance for VRHabilis-KEMRON
    d. Requests for camera passes
    e. Security Pass information for all employees on the project site
11. AHA format - COE format may be ok. Need to send a sample to Marc. Otherwise, use format found on the Safety Shack website.
12. May need to access Safety Shack website for APP with work plan format.
13. May be able to use an abbreviated UFP QAPP (Tier 2) developed by the Navy on their Quality website.

APP/SSHP	2 copies	Tim Sears, Marc McFarland
AHAs	2 copies	Tim Sears, Marc McFarland
Work Plan		Ben Simes, Tom Brent
UFP-QAPP	2 copies	Ben Simes, Tom Brent
UFP-QAPP	digital	Navy chemist (contact info forthcoming)
Final Plans	3 copies + 1 CD	IDEM Ben Simes

Consensus Decisions:

QAPP Worksheet #10 Problem Definition

10.1 Problem Definition, Site History, and Background:

This QAPP is classified as site specific, based on the UFP-QAPP guidance. Information presented in this generic QAPP will include only general information about the scope of work at the SWMU 9 Project site. Specific details regarding the project planning and problem definition of the site is included in this QAPP and the site specific SAP and/or Remedial Action Work Plan (RAWP). The purpose of this Quality Assurance Project Plan (QAPP) is to define responsibilities and authorities, and to prescribe requirements for assuring that the remedial actions at the SWMU 9 Project are planned and executed in a manner consistent with U.S. Environmental Protection Agency (EPA) Region II and Indiana Department of Environmental Management (IDEM) objectives, thus resulting in data that is usable to meet project objectives

10.1.1 Site History and Background

For the purposes of this QAPP the SWMU 9 UST Project Site consists of four (4) selected USTs (B7, B8, B26, B27) and remaining contamination from one (1) former UST (B9, exhumed) that are located within SWMU 9. These USTs are known to have been installed at the facility as early as 1953 and are located east of building B-150 (B7, B8, and B9) and south of building B-55 (B26 and B27). The USTs were reported to be used in support of the operations at each building respective building.

Based on the Navy's records, the following summarizes what is known about the USTs associated with this RAWP:

- East of Building B-150
    - USTs B7 and B8 are reported to be concrete, oriented along a north-south axis, and have respective 10,473 gallon storage capacities. The two (2) existing tanks reportedly held #6 fuel oil. Former UST (B9) was reported to be oriented along an East-West axis, have a 23,707 storage capacity, with an unknown composition. The contents of the exhumed tank have not been reported. An exploratory excavation conducted in the vicinity of B9 reported the presence of hydrocarbon contaminated soil.

- South of Building B-55 (Former Heavy Equipment Garage)
    - USTs B26 and B27 are reported to be concrete, oriented along an east-west axis, and have respective 25,800 gallon storage capacities. One of the tanks likely held gasoline and the other is assumed to have held #2 Fuel Oil.

During a separate investigation in June 2007 a Draft Resource Conservation and Recovery Act Facility Investigation (RFI) Report Addendum was prepared for SWMU 9 South (Pesticide Control/R-150 Tank Area). During this investigation eight soil borings were completed in the vicinity of the former fuel station (09SB058 - 09SB063, 09SB076 and 09SB077) associated with USTs B-26 and B-27. Both surface soil (0 - 2 feet below ground surface [bgs)) and subsurface soil (>2 feet bgs) samples were collected from each boring. The samples were collected in the vicinity of the former concrete fueling island (09SB058 - 09SB060), and the former pump house and two 25,800 gallon concrete underground storage tanks (09SB061 - 09SB063, 09SB076 and 09SB077). The samples were analyzed for volatile organic Compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides/PCBs, inorganics, and total petroleum Hydrocarbons (TPH).

The only compounds detected with concentrations in excess of the human health screening criteria were petroleum hydrocarbon diesel range organics (DRO) in samples 09SB590608 (280 [estimated] ug/kg) and 09SB61SS0002 (600 ug/kg), and petroleum hydrocarbon gasoline range organic (GRO) in sample 09SS610002 (200 ug/kg). The human health screening criteria for DRO and GRO are 230 mg/kg and 120 mg/kg, respectively.

In addition, the surface soil sample data were screened against Region 5 ecological screening levels (ESLs). Lead was the only compound that exhibited concentrations which exceed its ESL (11 mg/kg). Figure 2 shows the spatial distribution of the lead concentrations in surface soil at the Former Gas Station area which exceeded the ESL. The surface samples with lead concentrations above the ESL are 09SB58SS0002 (13.6 J mg/kg), 09SB59SS0002 (27.9 J mg/kg), 09SB60SS0002 (13.1 J mg/kg), 09SB61SS0002 (24.6 J mg/kg), 09SB62SS0002 (12 J mg/kg), 09SB63SS0002 (23.8 J mg/kg).

10.1.2 Summary of Remedial Action Objectives

The remedial action objectives (RAOs) for this Remedial Action (RA) is to obtain Record of Site Closure (RSC) with no land use controls or site restrictions (No Further Action) for four (4) selected USTs (B7, B8, B26, B27), associated piping, and remaining soil contamination from one former UST (B9). All of these tanks, present and former, are located within SWMU 9. Soil removal activities shall only be employed if the soil contamination is above action levels.

10.1.3 Summary of Remedy

The remedy for the site will be the removal of product, tanks, associated piping, and affected soil associated the UST systems. The RSCs and associated reports will be submitted to the Navy in Draft form for review and comment, then submitted to the IDEM and Navy in Draft Final form for review and comment and finally submitted to the Navy and the IDEM in Final form incorporating all agreed changes. The Remedy will be considered complete when the IDEM approves No Further Remediation (NFR) required for soil contamination associated with the USTs in question. Groundwater at the Site will be assessed during this RA, but no remedy will be performed on groundwater as part of this action. Should groundwater need to be addressed, it will be under a separate RAWP. The new UST Rule (329 IAC 9) will be adhered to along with all applicable portions of the Remediation Closure Guide (RCG). If necessary for RSC completion, soil and groundwater sampling efforts shall be conducted in areas potentially impacted by the former USTs. Groundwater and soil samples shall be analyzed for parameters within each UST as specified in the RCG. Soil and groundwater sampling activities shall be in accordance with any and all applicable, current Navy, State, and Federal laws, regulations, guidance, policy, and NSA Crane Policy. If sampling results indicate contamination in excess of acceptable unrestricted use criteria, VRHK shall conduct soil removal activities in order to complete an RSC for each site.

QAPP Worksheet #11 Project Quality Objectives/ Systematic Planning Process Statements

11.1 Who Will Use the Data?

Soil data gathered from the UST Site 00002 Project will be used by the Navy, in conjunction with IDEM Corrective Action, to assess current conditions at the site, to assess potential risk to potential receptors, to collected data to demonstrate achievement of the selected remedial goal(s), and to evaluate the remedy's continuing protection of human health and the environment.

Groundwater data will be used by the Navy and IDEM to assess potential risk to potential receptors and to evaluate the remedy's continuing protection of human health and the environment.

11.2 What Will the Data be Used for?

The environmental data that will be generated through this site work will be used to confirm the completion of the implemented remedy to accomplish site-specific objectives. During the UST removal, data will be used to determine the nature and extent of contamination. For those areas determined to be impacted by contamination, site data will be generated to determine the extent of excavation for the soils surrounding the USTs.

Any groundwater, or ponded surface water, will be used to evaluate groundwater conditions, and whether contaminant concentrations in groundwater pose a risk to receptors.

The data will also be used to locate, evaluate, and select backfill borrow sources, and to regulate how much of the excavated soils are disposed offsite.

11.3 What Type of Data Are Needed?

Analytical data requirements will vary based results of the initial confirmation sampling activities. Initially, soil samples will be collected as confirmation samples to determine if contaminants are present in soil surrounding the USTs. Confirmation soil samples will be analyzed for volatile organic compounds (VOCs) specifically benzene, toluene, ethylbenzene, xylenes (BTEX) and methyl-tert-butyl ether (MTBE), naphthalene, polycyclic aromatic hydrocarbons (PAHs) and total petroleum hydrocarbons (TPH). If soils are impacted by the contaminants of concern (COCs), and the analytes in soil exceed the screening criteria, then groundwater will be sampled for those analytes that have impacted the soil. In the event that surface water enters the excavation, then samples will be collected for the same analysis that confirmation soil samples are collected in each individual UST area, which is based on the product stored in individual USTs. Backfill borrow sources will be sampled for BTEX, TPH, polychlorinated biphenyls (PCBs), pesticides, selected metals (lead). Waste characterization samples will be collected from the materials excavated from the UST areas. One sample will be collected for every 50 cubic yards removed for BTEX, MTBE, Naphthalene, PAHs and TPH.

Confirmation soil samples and waste characterization samples will be analyzed using EPA Methods 8260B for VOCs, specifically BTEX, MTBE, and naphthalene, 8270-SIM for PAHs and naphthalene, and 8015 for TPH.

Groundwater or surface water samples may be analyzed using EPA Methods 8260B for VOCs, specifically BTEX, MTBE, and naphthalene, EPA Method 8270-SIM for PAHs and naphthalene, and EPA Method 8015 for TPH.

Backfill borrow source samples will be analyzed using EPA Methods 8260B for VOCs, specifically BTEX, EPA Method 8015 for TPH, EPA Method 8082 for PCBs, EPA Method 8081 for pesticides, and by EPA Method 6010 for Lead.

11.4 How "good" does the information need to be in order to support the environmental decision?

The data must support applicable requirements for the SWMU 9 Project. All data must meet the data quality objectives as specified in this QAPP, including the specific matrices to be sampled, location for sampling, analytical methods and the analytes to be included by matrix and concentration, screening and/or action levels by analyte, and acceptance criteria. The data quality will be reviewed to document the description of samples collected/shipped, sampling equipment utilized, and deviations from the work plan, including problems encountered and corrective actions taken. The applicable screening criteria for each COC is:

- Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.
- Backfill and Restoration Guidelines, provided by NAVFAC

The laboratory QC limits for each COC are included in Appendix B.

11.5 How much information is needed?

The amount of data needed will depend on the extent and type of COCs encountered at each UST removal area; therefore, the exact total number of samples to be collected cannot be defined at this point. Worksheet 18 provides a description of proposed sampling locations, an estimated number of potential samples collected at each UST removal area based on the size of each UST and the proposed sampling plan.

11.6 Where, when and how should the data be collected/generated?

The data will be collected as described in the Sampling and Analysis Plan (SAP) and the associated sampling standard operating procedures (SOPs). A portion of the sample locations will be pre-determined based on the size and shape of the USTs. However, a majority of the sampling locations will be based on the size and shape of the excavation well as the results of the initial confirmation samples. Groundwater sampling locations will be determined in the event that COCs are detected in soil.

11.7 Who will collect and generate data?

The VRHK environmental sampling team will collect and generate data under the management of the Field Team Leader, the QA Officer, and the Project Manager. Laboratory analyses of environmental samples collected by VRHK will be performed by Microbac Ohio Valley Division.

11.8 How will the data be reported?

11.8.1 Field Data Deliverables

Raw data from field measurements and sample collection will be reviewed by VRHK and retained in project files. Data relevant to the site-specific objectives will be tabulated and included in reports submitted for regulatory review. Such data may include but is not necessarily limited to PID readings, elevations of sample points, groundwater depths, and GPS locations.

Field data from groundwater monitoring events will be included in reports submitted for regulatory review. These data will be recorded on field data sheets that will be submitted with the sample chain of custody. The field data will be entered into the laboratory information management system and included in the electronic data deliverable (EDD).

Field sample records (SRs) will be completed by field personnel and copies of these SRs will be included in the project reports as applicable. Typical categories of information in the SRs include, but are not necessarily limited to: sampling location, date, time, interval/depth sampled, and groundwater field measurements. Any significant problems encountered in the field, resolution of those problems, deviations from planned procedures, or other matters that could impact field sampling execution and analytical results will be discussed in project reports.

11.8.2 Off-Site Laboratory Data Package Deliverables

Microbac provides four levels of laboratory reports in order to match the documentation level required by specific projects. The laboratory will provide Level 2 CLP-like data packages for all decision making data for the NSA Crane SWMU 9 Project. The data packages scanned to CD and delivered as hard copy will include the normal components of a CLP-like deliverable, including case narrative, sample summary report (Form 1), QA/QC summary forms (CLP-like forms), laboratory chronicle, chain-of-custody forms, data qualifiers, raw data; and, run logs. The following list of specific elements defines the items that will be included in all data packages prepared for project sample analyses:

- Case Narrative, including: laboratory analytical batch number; matrix and number of samples included; analyses performed and analytical methods used; description of any problems or exceedence of QC criteria and corrective action taken. The laboratory manager or his/her designee will sign the case narrative.
- Copy of chain of custody forms for all samples included in the analytical batch;
- Tabulated sample analytical results with units, data tags/qualifiers, percent solids, sample weight or volume, dilution factor, laboratory batch and sample number, VRHK field sample number, and dates sampled, received, extracted and analyzed all clearly specified. Surrogate percent recoveries will be included for organic analyses.
- Surrogate spike recoveries reported in all organic reports where appropriate. The reports will specify the control limits for surrogate spike results, as well as the spiking concentration. Any out-of-control recoveries will be reported immediately to the Project QA Manager. Any out-of-control recoveries (as defined by the applicable method) will result in the sample being rerun (with both sets of data to be reported).
- All calibration, quality control, run logs and sample raw data including chromatograms, quantitation reports and other instrument output data.
- Blank summary results indicating samples associated with each blank.

- Matrix spike/matrix spike duplicate result summaries with calculated percent recovery and relative percent differences.
- Laboratory control sample results, when applicable, with calculated percent recovery.
- Electronically formatted data deliverable results, in a format specified by the Project Manager.

If necessary, manual peak integration will be conducted for applicable analyses. Any manual integration will be completely and fully documented in the data package. The case narrative will give a brief explanation of the need for manual integration. The resulting data will be tagged, and photographs of chromatograms before and after manual integration will be provided in the raw data. Appropriate department supervisors of Microbac will review and sign-off on all data generated by manual integration. In the event that manual integration is necessary, manually integrated peaks will also be evaluated during the data validation process.

Data package deliverables will be consistent with the DoD QSM, version 4.2. The complete Level 2 analytical package, will be provided electronically (scanned onto a CD) as a pdf file. The Microbac Information Management System (LIMS) will use the data qualifiers established by the DoD QSM, version 4.2, as well as the project specific data qualifiers. Reporting level requirements are logged into the LIMS system with the sample set. Level 1 and 2 data packages are assembled upon printout of the final report by the data entry staff. Copies of the raw analytical data for level 3 and 4 data packages are turned in by the analysts upon completion of analysis. Data summary forms are printed from specialized software packages by the analysts in each laboratory. The forms are reviewed by the department supervisor before insertion into the final data package. Table 11.8.1 summarizes the content of a Level 2 data package.

	ICP/6010/6020 Deliverables	Level 1	Level 2
1	Cover Page	x	x
2	Summary data sheet	x	x
3	Initial Calibration		
4	ICV/CCV form		
5	CRQL Check Standard		
6	Blanks - ICB/CCB		
7	Prep Blank		x
8	ICP Interference check		
9	ICP/MS Interference check		
10	Matrix Spike Recovery		x
11	Post Digestion Spike		
12	Duplicates		x
13	Laboratory Control Sample		x
14	Holding Times		
15	ICP Serial Dilutions		
16	Method Detection Limits		
17	ICP-OES Interelement Corr. Fac.		
18	ICP-OES Interelement Corr. Fac.		
19	ICP Linear Ranges		
20	Preparation Log		
21	Analysis Run Log		
22	ICP-MS Tune		

23	ICP-MS Internal Standards		
24	Raw Data Package		
	GC/MS Deliverables	**Level 1**	**Level 2**
	8260,8270		
1	Cover Page	x	x
2	Analysis Data Sheet - Form 1	x	x
3	Tentatively Identified Compounds	x[1]	x[1]
4	Surrogate Recovery		
5	Internal Standard Areas		
6	Method Blank Summary		x
7	Method Blank Results		x
8	Laboratory Control sample		x
9	MS/MSD Summary		x
10	Instrument Tune Summary		
11	Initial Calibration Data		
12	Second Source Calibration		
13	Continuing Calibration Data		
14	Holding Times		
15	Instrument Run Log		
16	Raw Data Package		
	GC - With Data Download	**Level 1**	**Level 2**
	8021, 8081, etc.		
1	Cover Page	x	x
2	Analysis Data Sheet - Form 1	x	x
3	Surrogate Recovery		
4	Method Blank Summary		x
5	Method Blank Results		x
6	Laboratory Control sample		x
7	MS/MSD Summary		x
8	Initial Calibration Data		
9	Second Source Calibration		
10	Continuing Calibration Data		
11	Holding Times		
12	Instrument Run Log		
13	Raw Data Package		
	No Data Download	**Level 1**	**Level 2**
	RSK, HPLC, IC, etc.		
1	Cover Page	x	x
2	Analysis Data Sheet - Form 1	x	x
3	Surrogate Recovery		
4	Method Blank Summary		x
5	Method Blank Results		x
6	Laboratory Control sample		x
7	MS/MSD Summary		x
8	Initial Calibration Data		
	GC/MS Deliverables	**Level 1**	**Level 2**
9	Second Source Calibration		
10	Continuing Calibration Data		

11	Holding Times		
12	Instrument Run Log		
13	Raw Data Package		

X item provided at that laboratory report level.
1 Item provided only when specified in remedial action or other site-specific work plan.

11.9 How will the data be archived?

Documents will be maintained as they relate to support the final decision making process and regulatory closure. This information will be kept in hard copy and electronic formats. VRHK will provide deliverables in both electronic and hard copies of the Level 2 reports generated as part of the SWMU 9 UST Project will be submitted within the final reports to the Navy.

For the Laboratory, each sample delivery group is assigned a unique login number when initially logged into the LIMS system. The report master files are stored in order of the login number and can be easily retrieved using this system. Computer records from the Microbac LIMS are maintained on the system for one year and are then archived on magnetic tape. Two backup copies of the system are maintained on magnetic tape at all times. The backup and archive tapes are stored in a fireproof safe at the laboratory indefinitely. Extraction lab logbooks and bench sheets are maintained chronologically and stored in the laboratory for five years. Inorganics raw data, including analyst notebooks, bench sheets, computer printouts and instrument logs, are stored by parameter in the individual laboratories or in the archive area for a period of five years. Gas Chromatography (GC) data is stored as hardcopy in the individual master files. GC/MS data is stored as hardcopy for three years in the workorder files, and on magnetic tape for at least one year. GC/MS data stored on magnetic tape can be retrieved by the file numbers assigned to them at the time of analysis.

At the conclusion of the RA, KEMRON will upload all data to the Navy Installation Restoration Information Solution (NIRIS) database. NIRIS is a web-based system that manages the Navy's environmental data, documents and records related to cleanup of hazardous waste sites. NIRIS provides the Navy's remedial project managers (RPMs) and other environmental professionals with tools to effectively analyze, visualize, and present analytical and spatial data. KEMRON will upload the NIRIS Electronic Data Deliverable (NEDD) to NIRIS at the conclusion of the RA.

QAPP Worksheet #12 Measurement Performance Criteria Table (1)

Matrix	Soil
Analytical Group[1]	VOCs
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	8260B/MSV01 Standard Operating Procedure for Analysis of Volatile Organic Analytes by Methods 8260A & 8260B	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤30	Field Duplicate	S&A
		Accuracy/Bias /Contamination	< ½ LOQ (See Worksheet 28)	Method Blank	A
		Accuracy/Bias /Contamination	< LOD	Calibration Blanks	A
		Accuracy/Bias	80-120%	LCS	A
		Accuracy/Bias	RPD ≤30	MS/MSD	A
		Precision	RPD ≤20	Laboratory Duplicate	A
		Precision	Standards (% Rec) 4-Bromofluorobenzene (74-121) Toluene-d_8 (81-177) 1,2 Dichloroethane-d_4 (80-120) Dibromofluoromethane (80-120)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23

QAPP Worksheet #12
Measurement Performance Criteria Table (2)

Matrix	Water
Analytical Group[1]	VOCs
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-GWS-01 12-SWS-01 SOP for Groundwater Sampling and Surface Water Sampling	8260B/MSV01 Standard Operating Procedure for Analysis of Volatile Organic Analytes by Methods 8260A & 8260B	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤30	Field Duplicate	S&A
		Accuracy/Bias /Contamination	< ½ LOQ (See Worksheet 28)	Method Blank	A
		Accuracy/Bias /Contamination	< LOD	Calibration Blanks	A
		Accuracy/Bias	80-120%	LCS	A
		Accuracy/Bias	RPD ≤30	MS/MSD	A
		Precision	RPD ≤20	Laboratory Duplicate	A
		Precision	Standards (% Rec) 4-Bromofluorobenzene (74-121) Toluene-d_8 (81-177) 1,2 Dichloroethane-d_4 (80-120) Dibromofluoromethane (80-120)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23

QAPP Worksheet #12
Measurement Performance Criteria Table (3)

Matrix	Soil
Analytical Group[1]	PAHs
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	8270/MSS01 Standard Operating Procedure for Organic Analytes Methods 8270C	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤50	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias /Contamination	< LOD	Calibration Blanks	A
		Accuracy/Bias	Specified QC Limits (See list in Appendix B)	LCS	A
		Precision	RPD ≤40	MS/MSD	A
		Precision	Standards (% Rec) Terphenyl-d14 (18-137) 2-Fluorobiphenyl (30-115) Nitrobenzene-D5 (23-120)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23

QAPP Worksheet #12
Measurement Performance Criteria Table (4)

Matrix	Water
Analytical Group[1]	PAHs
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-GWS-01 12-SWS-01 SOP for Groundwater Sampling and Surface Water Sampling	8270/MSS03 Standard Operating Procedure for Organic Analytes Methods 8270C	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤50	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias	Specified QC Limits (See list in Appendix B)	LCS	A
		Precision	RPD ≤40	MS/MSD	A
		Precision	Standards (% Rec) Terphenyl-d14 (33-141) 2-Fluorobiphenyl (43-116) Nitrobenzene-D5 (35-114)	Surrogates	A
		Precision	Retention time ±30 seconds of last CC Area changes by a factor of two (-50% to +100%)	Internal Standards	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23

QAPP Worksheet #12
Measurement Performance Criteria Table (5)

Matrix	Soil
Analytical Group[1]	TPH-GRO[4] TPH-DRO[4]
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	8015/GCV05 8015/GCS02	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤30	Field Duplicate	S&A
		Accuracy/Bias /Contamination	<LOD	Calibration Blanks	A
		Accuracy/Bias	85-120% 53-127%	LCS	A
		Accuracy/Bias	RPD ≤20 RPD ≤26	MS/MSD	A
		Precision	RPD ≤20	Laboratory Duplicate	A
		Precision	Standards (% Rec) Chlorobenzene(s)-(64-148) o-Terphenyl (43-136) Octacosane (25-162)	Surrogates	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23
[4]Analytical Test for Backfill Requirements

QAPP Worksheet #12
Measurement Performance Criteria Table (6)

Matrix	Soil
Analytical Group[1]	PCBs[4]
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	8082/GCS10 SOP Organic Analysis of PCBs Method 8082/8082A	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤50	Field Duplicate or Field Triplicate	S&A
		Accuracy/Bias /Contamination	<LOQ	Method Blank	A
		Accuracy/Bias	Specified QC Limits (See List C in Appendix B)	LCS	A
		Accuracy/Bias	Analytes (%Rec) PCB-1016 (40-140) PCB-1260 (60-130)	MS/MSD	A
		Precision	RPD ≤40	MS/MSD	A
		Precision	Standards (% Rec) 2,4,5,6-Tetrachloro-m-Xylene (29-133) Decachlorobiphenyl (PCB-209) (30-173)	Surrogates	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23
[4]Analytical Test for Backfill Requirements

QAPP Worksheet #12
Measurement Performance Criteria Table (7)

Matrix	Soil
Analytical Group[1]	Pesticides[4]
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	8081A/8081B SOP for Organic Analysis of Pesticides	Accuracy/Bias /Contamination	<LOQ	Field Blanks	S&A
		Precision	RPD ≤50	Field Triplicate	S&A
		Accuracy/Bias /Contamination	< LOQ	Method Blank	A
		Accuracy/Bias /Contamination	Specified QC Limits (See List in Appendix B)	LCS	A
		Accuracy/Bias	See List K	MS/MSD	A
		Precision	RPD ≤ 30%	MS/MSD	A
		Precision	Standards (% Rec) 2,4,5,6-tetrachloro-m-xylene (39-130) Decachlorobiphenyl (33-143)	Surrogates	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23
[4]Analytical Test for Backfill Requirements

QAPP Worksheet #12
Measurement Performance Criteria Table (8)

Matrix	Soil/Sediment
Analytical Group[1]	Metals[4]
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
12-SSP-01 SOP for Soil Sampling in the Field Or FP-4 Soil Sampling	6010B/BR-ME-005 SOP for Metals by ICP-OES SW-846 Method 6010B	Precision	< 50% RPD	Field Duplicate	S&A
		Accuracy/Bias /Contamination	< ½ LOQ (See Worksheet 28)	Method Blank	A
		Accuracy/Bias /Contamination	< LOD	Calibration Blanks	A
		Accuracy/Bias	80-120%	LCS	A
		Accuracy/Bias	80-120%	MS	A
		Precision	RPD ≤20	Laboratory Duplicate	A
		Accuracy/Bias	For samples >50X LOQ, 5X dilution within ± 10% of original result	Serial Dilution	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet 21
[3]Reference number from QAPP Worksheet 23
[4]Analytical Test for Backfill Requirements

QAPP Worksheet #15
Reference Limits and Evaluation Table (1)

Matrix	Soil
Analytical Group	VOCs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (mg/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (mg/kg)	LOQs (mg/kg)
Benzene	71-43-2	15	5	8260B	0.5	5
Ethylbenzene	100-41-4	76	5	8260B	0.5	5
Toluene	108-88-3	820	5	8260B	0.5	5
m-,p-Xylene	179601-23-1	390	5	8260B	0.5	5
o-Xylene	95-47-6	430	5	8260B	0.5	5
Methyl-tert-butyl-ether	1634-04-4	600	5	8260B	0.5	5
Naphthalene	91-20-3	50	10	8260B	0.5	10

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.

QAPP Worksheet #15
Reference Limits and Evaluation Table (2)

Matrix	Water
Analytical Group	VOCs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (ug/L)	Project Quantitation Limit (ug/L)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/L)	LOQs (ug/L)
Benzene	71-43-2	5	1	8260B	0.125	1
Ethylbenzene	100-41-4	700	1	8260B	0.25	1
Toluene	108-88-3	1000	1	8260B	0.25	1
m-,p-Xylene	179601-23-1	190	1	8260B	0.5	1
o-Xylene	95-47-6	190	1	8260B	0.25	1
Methyl-tert-butyl-ether	1634-04-4	120	1	8260B	0.25	1
Naphthalene	91-20-3	1.4	1	8260B	0.2	1

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.

QAPP Worksheet #15
Reference Limits and Evaluation Table (3)

Matrix	Soil
Analytical Group	PAHs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (mg/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (mg/kg)	LOQs (mg/kg)
Phenanthrene	85-01-8	NE	2.5	8270 SIM	1.25	2.5
Indeno(1,2,3-cd)pyrene	193-39-5	2.1	2.5*	8270 SIM	1.25	2.5
Naphthalene	91-20-3	50	2.5	8270 SIM	1.25	2.5
Acenaphthene	83-32-9	4800	2.5	8270 SIM	1.25	2.5
1-Methylnaphthalene	90-12-0	310	2.5	8270 SIM	1.25	2.5
Acenaphthylene	208-96-8	NE	2.5	8270 SIM	1.25	2.5
Anthracene	120-12-7	24000	2.5	8270 SIM	1.25	2.5
Benzo(a)anthracene	56-55-3	2.14	2.5*	8270 SIM	1.25	2.5
Benzo(a)pyrene	50-32-8	0.21	2.5*	8270 SIM	1.25	2.5
Benzo(b)fluoranthene	205-99-2	2.1	2.5*	8270 SIM	1.25	2.5
Benzo(g,h,i)perylene	191-24-2	NE	2.5	8270 SIM	1.25	2.5
Benzo(k)fluoranthene	207-08-9	21	2.5	8270 SIM	1.25	2.5

Matrix	Soil
Analytical Group	PAHs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (mg/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (mg/kg)	LOQs (mg/kg)
Chrysene	218-01-9	210	2.5	8270 SIM	1.25	2.5
Pyrene	129-00-0	2400	2.5	8270 SIM	1.25	2.5
Dibenzo(a,h)anthracene	53-70-3	0.21	2.5*	8270 SIM	1.25	2.5
Fluoranthene	206-44-0	3200	2.5	8270 SIM	1.25	2.5
Fluorene	86-73-7	3200	2.5	8270 SIM	1.25	2.5
2-Methylnaphthalene	91-57-6	370	2.5	8270 SIM	1.25	2.5

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.
* The Project Quantitation Limit is greater than the Project Action Limit.
NE No Established Project Action Limit

QAPP Worksheet #15
Reference Limits and Evaluation Table (4)

Matrix	Water
Analytical Group	PAHs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (ug/L)	Project Quantitation Limit (ug/L)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/L)	LOQs (ug/L)
Phenanthrene	85-01-8	NE	0.05	8270 SIM	0.025	0.05
Indeno(1,2,3-cd)pyrene	193-39-5	0.29	0.05	8270 SIM	0.025	0.05
Naphthalene	91-20-3	1.4	0.05	8270 SIM	0.025	0.05
Acenaphthene	83-32-9	400	0.05	8270 SIM	0.025	0.05
1-Methylnaphthalene	90-12-0	9.7	0.05	8270 SIM	0.025	0.05
Acenaphthylene	208-96-8	NE	0.05	8270 SIM	0.025	0.05
Anthracene	120-12-7	1300	0.05	8270 SIM	0.025	0.05
Benzo(a)anthracene	56-55-3	0.29	0.05	8270 SIM	0.025	0.05
Benzo(a)pyrene	50-32-8	0.2	0.05	8270 SIM	0.025	0.05
Benzo(b)fluoranthene	205-99-2	0.29	0.05	8270 SIM	0.025	0.05
Benzo(g,h,i)perylene	191-24-2	NE	0.05	8270 SIM	0.025	0.05
Benzo(k)fluoranthene	207-08-9	2.9	0.05	8270 SIM	0.025	0.05

Matrix	Water
Analytical Group	PAHs
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (ug/L)	Project Quantitation Limit (ug/L)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/L)	LOQs (ug/L)
Chrysene	218-01-9	29	0.05	8270 SIM	0.025	0.05
Pyrene	129-00-0	87	0.05	8270 SIM	0.025	0.05
Dibenzo(a,h)anthracene	53-70-3	0.029	0.05*	8270 SIM	0.025	0.05
Fluoranthene	206-44-0	630	0.05	8270 SIM	0.025	0.05
Fluorene	86-73-7	220	0.05	8270 SIM	0.025	0.05
2-Methylnaphthalene	91-57-6	27	0.05	8270 SIM	0.025	0.05

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.
* The Project Quantitation Limit is greater than the Project Action Limit.
NE No Established Project Action Limit

QAPP Worksheet #15
Reference Limits and Evaluation Table (5)

Matrix	Soil
Analytical Group	TPH[3]
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (ug/kg)	Project Quantitation Limit (ug/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/kg)	LOQs (ug/kg)
TPH-GRO	8006-61-9	1000	100	8015D/8015M	45	100
TPH-DRO	68334-30-5	1000	10000	8015D/8015M	2500	10000

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Backfill and Restoration Guidelines, Appendix B.
[3] Analytical Test for Backfill Requirements
* The Project Quantitation Limit is greater than the Project Action Limit.
NE Not Established

QAPP Worksheet #15
Reference Limits and Evaluation Table (6)

Matrix	Soil
Analytical Group	PCBs[3]
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (mg/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/kg)	LOQs (ug/kg)
Aroclor-1254	11097-69-1	1	0.0165	8082	8.25	16.5
Aroclor-1260	11096-82-5	1	0.0165	8082	8.25	16.5
Aroclor-1016	12674-11-2	1	0.0165	8082	8.25	16.5
Aroclor-1242	53469-21-9	1	0.0165	8082	8.25	16.5
Aroclor-1248	12672-29-6	1	0.0165	8082	8.25	16.5
Aroclor-1221	11104-28-2	1	0.0165	8082	8.25	16.5
Aroclor-1232	11141-16-5	1	0.0165	8082	8.25	16.5

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method.
[2] The project action limits are based on the backfill and restoration requirements.
[3] Analytical Test for Backfill Requirements

QAPP Worksheet #15
Reference Limits and Evaluation Table (7)

Matrix	Soil
Analytical Group	Pesticides[3]
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (ug/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (ug/kg)	LOQs (ug/kg)
Alpha-Chlrodane	5103-71-9	1.5	0.00165	8081B	0.33	1.65
Gamma-Chlordane	57-74-9	1.5	0.00165	8081B	0.33	1.65
4,4'-DDD	72-54-8	3.2	0.00033	8081B	0.33	1.65
4,4'-DDE	72-55-9	3.2	0.00165	8081B	0.33	1.65
4,4'-DDT	50-29-3	3.2	0.00165	8081B	0.33	1.65
Dieldrin	60-57-1	3.2	0.00033	8081B	0.33	1.65
Heptachlor	76-44-8	1.5	0.00165	8081B	0.33	1.65

[1] Achievable LODs and LOQs are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to Microbac Laboratories, Inc. and are based on dry weight for soils.
[2] The project action limits are based on the backfill and restoration requirements.
[3] Analytical Test for Backfill Requirements
NE Not Established

QAPP Worksheet #15
Reference Limits and Evaluation Table (8)

Matrix	Soil
Analytical Group	Metals[3]
Concentration Level	Low

Analyte	CAS Number	Project Action Limit[2] (mg/kg)	Project Quantitation Limit (mg/kg)	Analytical Method	Achievable Laboratory Limits[1]	
					LODs (mg/kg)	LOQs (mg/kg)
Lead	7439-92-1	400	5	6010B	0.5	5

[1] Achievable limits of detection (LODs) and limits of quantitation (LOQs) are limits that an individual laboratory can achieve when performing a specific analytical method. The LODs and LOQs documented in the above table are specific to TestAmerica and are based on dry weight for soils.
[2] Project Action Limit source: Indiana Department of Environmental Management Office of Land Quality, Remediation Closure Guide, March 22, 2012 with corrections through July 9, 2012.
[3] Analytical Test for Backfill Requirements

QAPP Worksheet #17
Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g. grid system, biased statistical approach):

Following removal of the USTs, associated subsurface confirmation soil samples will be collected based on a modified grid system (based on the size of the USTs and the linear footage on the exposed side walls). The objectives for the confirmation sampling:

- Analytical sampling will be conducted with the intent to confirm and/or delineate COC concentrations to below the project screening criteria
- If a potential COC release is suspected, by field screening techniques, then soil samples will be collected in the suspected release areas. Soil and samples will be collected using discrete sampling methods.
- If the soil sampling results indicate that COC concentrations exceed the soil screening criteria, then second-phase (or step-out) soil samples will be collected from locations to horizontally or vertically delineate the impacted area to determine the extent of excavation.
- If the soil sampling results indicate that COC concentrations exceed the soil screening criteria, then NAVFAC and IDEM will be consulted in such an event to determine if groundwater sampling is warranted. Additionally, if water enters the open excavation, it will be sampled for the constituents associated with the individual UST.

In additional to confirmation soil sampling, the following samples will be collected for the project.

- Backfill borrow source samples. The backfill borrow material must be certified as "clean" through analytical sampling
- Waste Characteristic Sampling: As materials are excavated from the UST areas, one sample every 50 cubic yards of material will be sampled for the constituents associated with the individual UST.
- Air monitoring and sampling will be conducted at the discretion of the SSHO to monitor workers exposure.

Excavation Confirmation Soil Samples

Post-excavation confirmation sampling refers to the sampling of the bottom surface area and along the sidewalls of the excavation areas from which the USTs and impacted soil has been removed. After excavating the USTs and impacted soil, samples will be collected to document that the remedial goals have been met.

Confirmation samples will be collected on from the soil below both ends and the middle of the USTs and along the sidewalls of the excavation at a point half the distance from the surface to the bottom of the UST excavation at a rate of one (1) sample per 20 linear feet. Samples will also be collected from below the dispenser islands (if any) and from the piping at a rate of one (1) sample per 20 linear feet.

Offsite Backfill Source Samples

The Excavations will be backfilled after the USTs and any contaminated soils are removed from the site. The areas will be backfilled to match existing grade. The backfill soil obtained from an off-site borrow source will have properties similar to the native site soils. The backfill soil will be obtained from a source where due diligence shows no evidence of a release of a regulated substance (i.e., clean fill) and will include a certification regarding the origin of the clean fill, including a statement that, to the best of the provider's knowledge, the backfill soil has not been contaminated with the release of regulated substances. The backfill soil will also be subject to analytical testing to assure that it is not contaminated and meets the requirements outlined below.

One representative composite sample of the each virgin fill source will be collected with a decontaminated hand auger, stainless steel trowel, or disposable sampling equipment at random locations throughout the stockpile or in situ, as appropriate.

Waste Characterization Samples

Waste characterization sampling will be conducted for the soil that was been removed from the excavation and temporarily stockpiled in the constructed staging area. In accordance with IDEM 329 IAC 9, a grab soil sample will be collected directly from excavated material for every fifty (50) cubic yards of soil that is excavated regardless of whether it will be returned to the excavation or disposed of. During soil removal activities a portable PID will be utilized to screen soils for contamination. If field screening and laboratory analysis indicate that soil will require disposal, then soil will be disposed offsite at a disposal facility that is approved for use by the Navy.

Waste Characterization samples will be collected directly from the soil stockpile at a rate of one (1) sample per 50 cubic yards of soil. The analysis for each sample will be based on the contents of the UST and/or impacted soil.

Perimeter and Remedial Action Exposure Monitoring

The purpose of the perimeter and remedial action exposure monitoring is to determine whether the concentrations of COCs are below the project goals and to protect the RA workers and the offsite receptors surrounding the site.

Air monitoring will be conducted in accordance with the SSHP. Real time air monitoring will be conducted at all times during subsurface activities. Monitoring for volatile and semi volatile compounds will be performed with a photo ionization detector (PID) with a probe rating above 10.6 eV and draeger tubes specifically for benzene. Fugitive dust will be measured continually with a DataRAM. Hydrogen sulfide gas (as a potential component of #6 Fuel Oil will be measured with a Multigas detector. All readings will be recorded in a field log book, air monitoring data sheet, or in an excel spreadsheet format. Measurements will be taken at the edge of the excavation, in the breathing zone, and in upwind and downwind locations. Notes on weather, temperature, humidity, wind direction, wind speed, monitor placement, and daily site work activities will be collected along the real-time data. Sample air monitoring logs are included in Appendix D.

In the event that the SSHO determines that personal samples are necessary, then those samples will be collected from the "most at risk" equipment operators/technicians and will be submitted for laboratory analysis of respirable dust by NIOSH 0600, and lead by NIOSH 7300 on a daily basis until sufficient data is collected and the SSHO determines that the sampling frequency can be down-graded. All personal air samples will be submitted to Galson Laboratories.

Groundwater or Surface Water Confirmation Samples

Post-excavation confirmation sampling refers to the sampling of the bottom surface area and along the sidewalls of the excavation areas from which the USTs and impacted soil has been removed. After excavating the USTs and impacted soil, samples will be collected to document that the remedial goals have been met.

In the event that water is encountered in the excavation(s), a minimum of one (1) water sample will be collected from the water encountered in each respective excavation and analyzed for the constituents associated with the UST(s) in that particular excavation. If a contaminant release has occurred or is suspected based on visual observations, field screening, odors, and/or laboratory analytical results and groundwater is not encountered in the excavation, then a boring may be required to be advanced to groundwater or thirty (30) feet below ground surface as per the IDEM 329 IAC 9. However, a waiver of groundwater sampling requirement may be obtained from IDEM dependent on soil sampling results.

If a groundwater boring or monitoring well is required, soil will be continuously sampled as it is advanced to the first saturated ground water zone or to a total depth of thirty (30) feet below grade at the area of suspected or confirmed release. If auger refusal is encountered before encountering groundwater or reaching thirty (30) feet bgs then a soil sample will be collected at the bottom of the boring. In the event there are no PID readings during soil screening activities, soil samples that are stained will be the next priority for analytical testing. Otherwise, soil samples collected at the deepest interval will be shipped to the laboratory for analysis. If groundwater is not encountered within a depth of thirty (30) feet, a soil sample will be collected at the base of the boring. Each soil sample will be screened using a PID and only the soil sample with the highest reading from each location shall be sent to the laboratory for analysis. However, if bedrock is encountered in a boring before a depth of thirty (30) feet is reached, and a saturated ground water zone is not encountered in the boring, the Navy Project Manager will be notified and the IDEM will be contacted to determine an acceptable soil sampling strategy based on field observations.

Information provided for each of these activities includes a brief description of the field activity, media to be sampled, number and location of samples to be collected, sampling method, and field and laboratory analytical methods to be performed. Sampling activities will be performed in accordance with KEMRON's Corporate Standard Operating Procedures (SOPs). The following SOPs are included as Appendix C:

- 12-UTL-01 Utility Clearance
- 07-DCN-00 Sampling Equipment and Container Decontamination
- 12-SSP-01 Soil Sampling
- 12-GWS-01 Ground Water Sampling
- 12-SWS-01 Surface Water Sampling

- 12-MWI-00 Monitoring Well Installation
- 12-MWC-00 Monitoring Well Construction
- 07-PAS-00 Sample Packaging and Shipping
- 07-DOC-00 Field Documentation
- 07-FQC-00 Field Quality Control

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:

Soil confirmation and waste characterization samples will be collected utilizing a hand auger or stainless steel spoons. Soil samples will be analyzed for COCs which include:

- VOCs (specifically BTEX, MTBE, and naphthalene) by EPA Method 8260B
- PAHs by EPA Method 8270 SIM

The confirmation sampling will include the following types of samples:

- Floor samples: These will be collected from soil below both ends and the middle of the USTs
- Wall samples: Sidewall samples will be collected every twenty (20) feet of perimeter distance around each of the former UST excavations at a point half the distance from the surface to the bottom of the underground storage tank excavation.
- Piping samples: Native soil beneath piping will be sampled every twenty (20) feet, along the piping run beneath elbows and connectors.
- Dispenser Island samples: One soil sample will be collected under each former dispenser island (if any) that is identified during closure activities.

Groundwater and surface water samples will be collected utilizing temporary monitoring wells and low flow sampling techniques. Groundwater samples will be analyzed for COCs which include:

- VOCs (specifically BTEX, MTBE, and naphthalene) by EPA Method 8260B
- PAHs by EPA Method 8270 SIM

Backfill borrow source samples will be collected utilizing a hand auger or stainless steel spoons. The backfill source samples will be analyzed for the following:

- VOCs (specifically BTEX) by EPA Method 8260B
- TPH by EPA Method 8015
- PCBs by EPA Method 8082A
- Pesticides by EPA Method 8081

- Lead by EPA Method 6010B

Air Monitoring samples, if required, will be collected utilizing personal monitoring pump. Sample air monitoring logs are included in Appendix D. The air samples will be analyzed for the following:

- Respirable Dust by NIOSH 0600
- Lead by NIOSH 7300

Sample collection will be performed in general accordance with the SOPs, which are included in Appendix C.

Quality Control Sampling

Throughout the soil and groundwater sampling, QC samples will be collected. Types of QC samples collected include field blanks, rinse blanks, field duplicates, and matrix spike and matrix spike duplicates (MS/MSD). Collection methods and frequencies were as follows:

- One (1) field blank will be collected each day by pouring deionized water into sampling containers.
- One (1) rinse blank will be collected each day or every twenty (20) samples, by pouring deionized water over or through the decontaminated sampling equipment and then into the sampling containers.
- One (1) blind field duplicate sample will be collected for every twenty (20) samples collected. The field duplicate soil and sediment samples will be collected at the same time as the actual sample and from the homogenized mixture of sediments in stainless steel bowls. Surface water and groundwater field duplicate samples will be collected simultaneously to the actual samples.
- One (1) MS/MSD sample will be collected for every twenty (20) samples collected. The MS/MSD samples shall be collected at the same time as the actual sample and from the homogenized mixture of sediments and soils in stainless steel bowls. Surface water and groundwater MS/MSD samples will be collected simultaneously to the actual samples.

QAPP Worksheet #18
Sampling Locations and Methods / SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)[2]	Sampling SOP Reference[1]	Rationale for Sampling Location
UST Closure Confirmation Samples	Soil	Samples will include subsurface floor and wall confirmation samples	VOCs (8260B) PAHs (8270 SIM)	Low	Up to 55 Samples 3 Duplicates 3 MS/MSD 5 Rinse Blanks 5 Field Blanks	12-SSP-00 FP-4	See Worksheet 17
	Groundwater	TBD[2]	VOCs (8260B) PAHs (8270 SIM)	Low	Up to 8 Samples 1 Duplicates 1 MS/MSD 1 Rinse Blank 1 Field Blank	12-GWS-00 12-SWS-01	See Worksheet 17
Offsite Backfill Source Sample	Soil	Grab samples will be collected from virgin ground or backfill stockpile	VOCs (8260B) TPH (8015) PCBs (8082) Pesticides (8081) Lead (6010)	Low	Up to 2 Samples 1 Duplicates 1 MS/MSD 1 Rinse Blank 1 Field Blank	12-SSP-00 FP-4	See Worksheet 17

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)[2]	Sampling SOP Reference[1]	Rationale for Sampling Location
Waste Characterization Sample	Soil	Grab samples will be collected from soil stockpile	VOCs (8260B) PAHs (8270 SIM)	Low	Up to 20 Samples 1 Duplicates 1 MS/MSD 0 Rinse Blank 0 Field Blank	12-SSP-00 FP-4	1 per 50 cubic yards of material
Air Monitoring	Air	NA	Respirable Dust (NIOSH 0600) Lead (NIOSH 7300)	Low	Up to 10 Samples	NA	See Worksheet 17

[1] Specify the appropriate letter or number from the Project Sampling SOP References table.
[2] The actual depth, number of samples and field duplicates, will be based on the field observation of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.

QAPP Worksheet #19
Analytical SOP Requirements Table

Matrix	Analytical Group	Conc. Level	Analytical And Preparation Method / SOP Reference	Sample Volume	Containers (number, size and type)[1,2]	Preservation Requirements (chemical, temperature, light protected	Maximum Holding Time (preparation analysis)
Soil	VOCs	Low	SW-846 EPA Method 8260B	5 grams of soil	3 containers (40-mL glass vials with Teflon®-lined septum cap)	Cool to 4°C ± 2°C or ship frozen on ice	14 days to extraction, 40 days to analysis
Soil	PAHs	Low	SW-846 EPA Method 8270 SIM	Full jar[3]	4 oz. Glass jar	Cool to 4°C ± 2°C or ship frozen on ice	14 days to extraction, 40 days to analysis
Soil	TPH[3]	Low	SW-846 EPA Method 8015D	Full jar[3]	4 oz. Glass or plastic jars	Cool to 4°C ± 2°C	7 days to extraction, 40 days to analysis
Soil	PCBs[4]	Low	SW-846 EPA Method 8082A	Full jar[3]	4 oz. Glass or plastic jars	Cool to 4°C ± 2°C	7 days to extraction, 40 days to analysis
Soil	Pesticides[4]	Low	SW-846 EPA Method 8081	Full jar[3]	4 oz. Glass or plastic jars	Cool to 4°C ± 2°C	7 days to extraction, 40 days to analysis
Soil	Metals (Lead)[4]	Low	SW-846 EPA Method 6010B	Full jar[3]	4 oz. Glass or plastic jars	N/A	6 months

Matrix	Analytical Group	Conc. Level	Analytical And Preparation Method / SOP Reference	Sample Volume	Containers (number, size and type)[1,2]	Preservation Requirements (chemical, temperature, light protected	Maximum Holding Time (preparation analysis)
Water	VOCs	Low	SW-846 EPA Method 8260B	Full jar[3]	3 – 40 ml glass vials with Teflon®-lined septum cap)	Cool to 4°C ± 2°C and HCl to pH <2	14 days
Water	PAHs	Low	SW-846 EPA Method 8270 SIM	Full jar[3]	2 – 1 Liter Amber Glass Jar	Cool to 4°C ± 2°C	7 days to extraction, 40 days to analysis

[1] The sample containers used for each chemical parameter must be certified as being clean or have been decontaminated by the laboratory.
[2] The type and number of containers required may be adjusted to reflect the requirements of the individual laboratories selected to perform the analyses. The laboratories may allow combining analyses requested per jar to reduce the number of jars required.
[3] Each MS/MSD set is considered an individual sample and requires two (2) additional jars.
[4] Analytical Test for Backfill Requirements

QAPP Worksheet #20
Field Quality Control Sample Summary

Matrix	Analytical Group	Conc. Level	Analytical And Preparation Method / SOP Reference	No. of Sampling Locations[1]	No. of Field Duplicate Pairs	Inorganic No. of MS	No. of Field Blanks	No. Equip. Blanks	No. PT Samples	Total No. of Samples to Lab
Soil	VOCs	Low	SW-846 EPA Method 8260B	55	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Soil	PAHs	Low	SW-846 EPA Method 8270 SIM	55	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Soil	TPH[2]	Low	SW-846 EPA Method 8015D	2	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Soil	PCBs[2]	Low	SW-846 EPA Method 8082A	2	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Soil	Pesticides[2]	Low	SW-846 EPA Method 8081	2	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade	1 per 20 samples or 1 per day during	N/A	TBD[1]

Matrix	Analytical Group	Conc. Level	Analytical And Preparation Method / SOP	No. of Sampling Locations[1]	No. of Field Duplicate	Inorganic	No. of Field Blanks	No. Equip. Blanks	No. PT Samples	Total No. of Samples
							deionized water is used.	sampling		
Soil	Metals (Lead)[2]	Low	SW-846 EPA Method 6010B	2	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Water	VOCs	Low	SW-846 EPA Method 8260B	TBD[1]	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]
Water	PAHs	Low	SW-846 EPA Method 8270 SIM	TBD[1]	1 per 20 samples	1 per 20 samples	1 per source of decontaminatio n water. Not applicable if laboratory grade deionized water is used.	1 per 20 samples or 1 per day during sampling	N/A	TBD[1]

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical Test for Backfill Requirements

QAPP Worksheet #21
Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
07-DCN-00	Standard Operating Procedure for Sampling Equipment and Container Decontamination	KEMRON	Reusable sampling equipment (hand auguer, split spoon samplers, pumps, etc.)	N	Includes descriptions and procedures for sampling equipment and container decontamination.
07-FQC-00	Standard Operating Procedure for Field Quality Control	KEMRON	N/A	N	Includes basic procedures for collecting various quality control samples.
07-PAS-00	Standard Operating Procedure for Sample Packing and Shipping	KEMRON	N/A	N	Includes descriptions and procedures for packaging of samples and addresses proper shipping protocols.
12-GWS-00	Standard Operating Procedure for Groundwater Sampling	KEMRON	N/A	N	Includes descriptions and procedures for a variety of sampling techniques for collecting representative groundwater samples with a variety of sampling equipment.
12-SWS-01	Standard Operating Procedure for Surface Water Sampling	KEMRON	N/A	N	Provides technical guidance on surface water sampling.
12-MWI-00	Standard Operating Procedure for Monitoring Well Installation or Supervision of Monitoring Well Installation	KEMRON	N/A	N	Provides a method for over-site and installation for temporary or permanent monitoring well installation and hand auguring in accordance with an approved work plan or field assignment form.
12-MWC-00	Standard Operating Procedure for Monitoring Well Construction	KEMRON	N/A	N	Provides the general steps for permanent and temporary monitoring well construction.
12-SSP-00	Standard Operating Procedure for Soil Sampling in the Field	KEMRON	N/A	N	Provides technical guidance on soil sampling including decontamination of and types and uses of sampling equipment.
12-UTL-01	Standard Operating Procedure for Utility Clearance	KEMRON	N/A	N	Provides technical guidance on subsurface utility clearance.
07-DOC-00	Field Documentation	KEMRON	N/A	N	Provides guidance on field documentation and record keeping.

QAPP Worksheet #23
Analytical SOP References Table

Reference Number[1]	Title, Revision Date and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)	Variance from the QSM? (Y/N)
ME600F	Standard Operating Procedure for Thermo Jarrell Ash IRIS Advantage Inductively Coupled Plasma Atomic Emission Spectroscopy – SW 846 Method 6010B/ EPA Method 200.7	Definitive	Metals	ICP-AES	Microbac	N	N
ME700	Standard Operating Procedure for Metals by Perkin Elmer ELAN 6100 Inductively Coupled Plasma – Mass Spectrometer - SW 846 Method 6020/ EPA Method 200.8	Definitive	Metals	ICP-MS	Microbac	N	N
MSVO1	Standard Operating Procedure for Analysis of Volatile Organic Analytes by Methods 8260A and 8260B	Definitive	VOCs	GC/MS	Microbac	N	N
MSS03	Standard Operating Procedure for Low Level PAHs Method 8270	Definitive	PAHs	GC/MS	Microbac	N	N
EXA02	Standard Operating Procedure Sonication Extractions for Poly Aromatic Hydrocarbons - SW 846 Methods 3550B and 3550C	Definitive	PAHs	N/A	Microbac	N	N
EXP02	Standard Operating Procedure Sonication Extractions For Pesticides and PCB's SW846 Method 3500 and 3550/Revision #15 Issue/Implementation Date: 15 October 2010 Last Review Date: 15 April 2012	Definitive	Solid	Ultrasonic Cell Disruptor/Nitrogen Evaporator/Solid Phase Extraction Manifold	Microbac	N	N
GCS09	Standard Operating Procedure Organic Analysis of Pesticides Method 8081/Revision#14 Issue/Implementation Date: 15 August 2012 Last Review Date: 15 August 2012	Definitive	Solid	GC/FID	Microbac	N	N
GCV05	Standard Operating Procedure For Gasoline Range Organics SW846 8015/Revision #12 Issue/Implementation Date: 15 March 2010 Last Review Date 15 April 2012	Definitive	Water/Solid	GC/FID/PID	Microbac	N	N

Reference Number[1]	Title, Revision Date and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)	Variance from the QSM? (Y/N)
GCS02	Standard Operating Procedure Analysis of Diesel Range Organics Method 8015/Revision #14 Issue/Implementation Date: 29 June 2012 Last Review Date: 29 June 2012	Definitive	Water/Solid	GC/FID	Microbac	N	N
GCS10	Standard Operating Procedure Organic Analysis of PCBs Method 8082/8082A	Definitive	Pesticides / PCBs	GC/ECD	Microbac	N	N
GCS09	Standard Operating Procedure Organic Analysis of Pesticides Method 8081A/8081B	Definitive	Pesticides	GC/ECD	Microbac	N	N

[1] SOPs are located in Appendix E.

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS (VOCs)	ICAL	As needed; when CCV out of criteria	1. Average response factor (RF) for SPCCs: VOCs > 0.30 for chlorobenzene and 1,1,2,2-Tetrachloroethane, > 0.1 for chloromethane, bromoform, and 1,1-dichlororethane. 2. RSD for RFs for CCCs: VOCS < 30% and one option below; Option 1: Average RRF if RSD for each analyte < 15% Option 2: linear least squares regression r > 0.995 Option 3: non-linear regression - coefficient of determination r2 > 0.99	Correct problem; Repeat calibration	Analyst / Supervisor	MSV01
	ICV	After each initial calibration	Value of all analytes within + 25% of expected value	Reanalyze ICV; upon 2nd failure, repeat calibration		
	CCV	Daily; every 12 hours	1. Average response factor (RF) for SPCCs: VOCs > 0.30 for chlorobenzene and 1,1,2,2-Tetrachloroethane, > 0.1 for chloromethane, bromoform, and 1,1-dichlororethane. 2. % Difference /Drift for CCCs: All analytes < 20%D	Reanalyze CCV; if failure, repeat ICAL		
	BFB Tune	Every 12 hours	Mass Criteria Mass 50: 15.0-40.0 percent of mass 95 Mass 75 30.0-60 percent of mass 95: Mass 95 Base peak, 100 percent relative abundance Mass 96 5.0 to 9.0 percent of mass 95 Mass 173 Less than 2.0 percent of mass 174 Mass 174 Greater than 50.0 percent of mass 95 Mass 175 5.0 to 9.0 percent of mass 174 Mass 176 95.0 to 101 percent of mass 174 Mass 177 - 5.0 to 9.0 percent of mass 176	Retune and/or clean source		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS (PAHs)	Tune (DFTPP)	Daily prior to standard and sample analysis	m/z Ion Abundance Criteria 51 30.0-80.0% of m/z 198 68 <2.0% of m/z 69 69 Present 70 <2.0% of m/z 69 127 25.0-75.0% of m/z 198 197 <1.0% of m/z 198 198 Base peak, 100% relative abundance 199 5.0-9.0% of m/z 198 275 10.0-30.0% of m/z 198 365 >0.75% of m/z 198 441 Present, but <m/z 443 442 40.0-110.0% of m/z 198 443 15.0-24.0% of m/z 442	Correct problem; tune instrument; repeat tune.	Analyst / Supervisor	MSS03
	Minimum 5-pt initial calibration (ICAL) curve	Prior to sample analysis. Set-up, major maintenance and/or continuing calibration standards do not meet.	%RSD ≤20% of the response factor from the ICAL curve.	Correct problem; repeat ICAL.		
	Continuing Calibration Verification (CCV)	Daily before sample analysis.	%D ±15% of the response factor from the ICAL curve.	Reanalyze CCV; if fails, ICAL must be repeated.		
	Minimum 5-pt initial calibration (ICAL) curve	Prior to sample analysis. Set-up, major maintenance and/or continuing calibration standards do not meet	RRF>0.01 (Advisory), RRF >0.05 for SPCCs. %RSD ≤15%, RSD ≤30% for CCC compounds. Lab may use first or higher order regression fit (r≥0.99) if %RSD >15%.	Correct problem; repeat ICAL.		
	Continuing Calibration Verification (CCV)	12 hours	RRF > 0.01 (Advisory), RRF > 0.05 for SPCCs. Percent difference for CCCs must be ≤20%. If criteria are met, the relative response factors of all compounds are calculated.	Reanalyze CCV; if fails, ICAL must be repeated.		
ICP (Metals)	Minimum 1-pt initial calibration curve	Daily or major maintenance, instrument modification, replacement of the torch, replacement of the mirror	r > 0.995 for all elements r: linear correlation coefficient	Correct problem; repeat ICAL.	Analyst / Supervisor	ME600F

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	Initial/continuing calibration verification (ICV/CCV)	Every 10 samples or 2 per 8 hr and end of run.	Recovery ±10% of true value	Reanalyze CCV. If the CCV fails second time, terminate analysis, correct problem, re-calibrate instrument, and re-verify calibration prior to continuing sample analysis.		
	Interference check sample (ICS)	Beginning of each sample analytical run.	Recovery ±20% of true value.	Terminate the analysis, correct the problem, re-calibrate, re-verify the calibration, and reanalyze the samples.		
	Interference check sample (ICS)	Beginning of each sample analytical run.	Recovery ±20% of true value.	Terminate the analysis, correct the problem, re-calibrate, re-verify the calibration, and reanalyze the samples.		
GC (PCBs)	Minimum 5-pt initial calibration (ICAL) curve	Prior to sample analysis Set-up, major maintenance	curve RSD for all analytes <20% or linear regression where the coefficient of correlation is ≥0.995	Correct problem; repeat ICAL.	Analyst / Supervisor	GCS10/ SOC-8082Ar
	Second Source - ICV Standard	Once after each ICAL	%D < 20% of true value	Correct problem and verify second source standard. Re-run second source verification. If that fails, correct problem and repeat ICAL		GCS10

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
	Continuing Calibration Verification (CCV)	Daily before sample analysis and after every 10 samples or every 12 hours, whichever is first	%D < 20% of true value	Reanalyze the CCV. If the CCV fails a second time, ICAL must be repeated.		GCS10/ SOC-8082Ar
GC/FID/PID	ICAL	Initially and upon failure of two consecutive CCVs	Use Average RF else linear least squares regression r≥ 0.995 else non-linear regression r2 ≥ 0.99	Correct problem - Repeat calibration	Analyst / Supervisor	GCV05
	ICV	After each initial calibration	% Drift ± 15%	Reanalyze ICV; if failure, repeat ICAL		
	CCV	Prior to sequence, every 10 samples and end of sequence	SW8015B % Drift ± 15% SW8015C/D % Drift + 20%	Reanalyze CCV; if failure, repeat ICAL		
GC/FID	ICAL	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: Linear least squares regression: r > 0.995: Option 3: non-linear regression: coefficient of determination (COD) r2≥0.99 (6 points shall be used for second order, and 7 points shall be used for third order).	Correct problem then repeat ICAL.	Analyst / Supervisor	GCS09
	ICV	Immediately following ICAL.	GC methods: All project analytes within + 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.		
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	GC methods: All project analytes within + 20% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/FID	ICAL	ICAL prior to sample analysis and upon failure of two consecutive CCVs.	% RSD ≤ 20 for each analyte. If % RSD > 20, linear regression with r ≥ 0.995 for each analyte.	Evaluate/correct problem; repeat calibration; or qualify data and discuss in narrative.	Analyst / Supervisor	GCS02
	ICV	Immediately following ICAL.	≤ 20% drift for each analyte (≤ 15% for 8081A)	Correct problem; reanalyze ICV; upon second failure, repeat initial calibration.		
	Daily before sample analysis, every ten samples, and at the end of the analysis sequence.	≤ 20% drift for each analyte (≤ 15% for 8081A)	Samples bracketed by an unacceptable CCV must be reanalyzed. Repeat initial calibration after two consecutive failures of the CCV.	Daily before sample analysis, every ten samples, and at the end of the analysis sequence.		

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #28
QC Samples Table

Matrix	Soil and Groundwater
Analytical Group	VOCs
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-01; FP-4 Groundwater: 12-GWS-00 Surface Water: 12-SWS-01
Analytical Method / SOP Reference	SW-8260B MSV01
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories, Inc.
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blanks	Every 12 hours	No analytes detected ≥ LOQ	Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank. Document source of contamination.	Lab Representative	Accuracy/ Bias/Contamination	No analytes detected ≥ LOQ
LCS	Every batch	Specified QC limits (See List in Appendix B)	Qualify associated data biased high or biased low as appropriate.	Lab Representative	Accuracy/ Bias	Specified QC limits (See List in Appendix B)
Matrix Spike and Duplicate	1 per 20 per matrix	Specified QC limits (See List in Appendix B)	If MS/MSD results do not meet criteria, the reviewer should review the data in conjunction with other QC results to determine if the problem is specific to	Lab Representative	Accuracy/ Bias	Specified QC limits (See List in Appendix B)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			the QC samples or systematic.			
Duplicate Sample	5% of field samples per matrix	RPD ≤ 25% aqueous; RPD ≤ 35% solid	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservation/Bias	To verify sample cooler temperature during transport.
Trip Blank	1 trip blank per cooler of VOA samples	No target analytes, volatiles in water.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To determine if cross contamination occurs during shipment or storage with volatile

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
						organic analyses (VOA) samples.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To characterize decontamination water source; not applicable if laboratory grade deionized water is used for decontamination rinse.

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.

Matrix	Soil & Groundwater
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-00; FP-4 Groundwater: 12-GWS-00 Surface Water: 12-SWS-01
Analytical Method / SOP Reference	SW-8270 SIM MSS03
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent reference standard (LCS)	1 per batch	Specified QC Limits (See Lists in Appendix B)	Correct problem, then reanalyze. If still out, reprep and reanalyze the LCS and all samples in the affected analytical batch. Qualify associated data biased high or biased low as appropriate.	Lab Representative	Accuracy/ Bias	Specified QC Limits (See Lists A and E in Appendix B)
Method blanks	1 per batch	No target analytes.≥ RL	Correct	Lab	Accuracy/	No target

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits			Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
					problem then reprep and analyze method blank and all samples processed with the contaminated blank. Document source of contamination.	Representative	Bias/Contamination	analytes.≥ RL
Matrix spike and duplicate	1 per 20 samples per matrix	Standards Acenaphthene; Acenaphthylene; Anthracene; Benzo (a) Anthracene; Benzo (a) Pyrene; Benzo (b) Fluoranthene; Benzo (g,h,i) Perylene; Benzo (k) Fluoranthene; Chrysene; Dibenzo (a,h) anthracene; Fluorene	Aqueous %R 45–110 50–105 55–110 55–110 55–110 45–120 40–125 45–125 55-110 40–125 50–110 RPD ≤30 for all	Solid %R 45–110 45–105 55–105 50–110 50–110 45–125 55–115 45–125 55-110 40-125 55-125 RPD ≤50 for all	Data reviewer may use the MS and MSD results in conjunction with other QC sample results to determine the need for some qualification of the data.	Lab Representative	Accuracy/ Bias	QC Limits (See Lists A and E in Appendix B)
Duplicate Sample	5% of field samples per matrix	RPD ≤ 25% aqueous; RPD ≤ 35% solid			Data reviewer should review and judge all of the target	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			compounds against the acceptance criteria.			analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To ensure the decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservation/Bias	To verify sample cooler temperature during transport.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To characterize decontamination water source; not applicable if laboratory grade

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			acceptance criteria.			deionized water is used for decontamination rinse

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.

Matrix	Soil
Analytical Group	TPH[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-00; FP-4 Groundwater: 12-GWS-00 Surface Water: 12-SWS-01
Analytical Method / SOP Reference	SW-8015/GCV05 SW-8015/GCS02
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per matrix/batch ; maximum of 20 samples per batch.	GRO < ½ reporting limit DRO < reporting limit See limits on Worksheet #15	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative.	Analyst / Supervisor	Bias/ Contamination	GRO < ½ reporting limit DRO < reporting limit See limits on Worksheet #15
Laboratory Control Sample (LCS)	One per matrix/batch ; maximum of 20 samples	Target compounds within the designated ranges; use project QAPP or standard control criteria See limits on Worksheet #15	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in	Analyst / Supervisor	Accuracy / bias	Target compounds within the designated ranges; use project QAPP or standard control criteria See limits on Worksheet

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			narrative.			#15
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One per matrix/batch ; maximum of 20 samples	Target compounds within the designated ranges; See limits on Worksheet #15	Qualify data and/or address in narrative.	Analyst / Supervisor	Accuracy / bias	Target compounds within the designated range See limits on Worksheet #15
Sample/Sample Duplicate	One per matrix/batch ; maximum of 20 samples	Target compounds within the designated ranges; See limits on Worksheet #15	Qualify data and/or address in narrative.	Analyst / Supervisor	Accuracy / bias	Target compounds within the designated range See limits on Worksheet #15
Surrogate Spike	"Every sample, standard, and quality control sample "	See limits on Worksheet #15	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative.	Analyst / Supervisor	Accuracy / bias	See limits on Worksheet #15
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To ensure the decontaminati on of sampling equipment has been

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			the target compounds against the acceptance criteria.			adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservation/Bias	To verify sample cooler temperature during transport.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To characterize decontamination water source; not applicable if laboratory grade deionized water is used for decontamination rinse

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical Test for Backfill Requirements

Matrix	Soil
Analytical Group	Metals[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-00; FP-4
Analytical Method / SOP Reference	SW-6010B/6020/7471/7470A ME600F/ME700/ME405/ME404
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Continuing calibration blank (CCB)	Every 10 samples, end of analytical run	Concentration < 3 X s of the background mean (ICP). No target analytes.	If the average in not within criteria, terminate the analysis, correct the problem, re-calibrate, and reanalyze all samples analyzed since the last acceptable CCB.	Lab Representative	Accuracy/ Bias/Contamination	Concentration < 3 X s of the background mean (ICP). No target analytes.
Serial Dilution (ICP)	1 per 20 samples per matrix for samples > 50x MDL	Difference between diluted and undiluted sample <10%.	Chemical or physical interference should be suspected. Investigate to determine cause.	Lab Representative	Accuracy/ precision	Difference between diluted and undiluted sample <10%.
Preparation	1 per batch per	No target analytes.	Documented	Lab	Accuracy/	No target

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
blank	matrix		source of contamination.	Representative	Bias/Contamination	analytes.
Laboratory Control Sample	1 per analytical batch	80%≤%Rec.≤120%	Qualify associated data biased high or biased low as appropriate.	Lab Representative	Accuracy/ Bias	80%≤%Rec.≤ 120%
Matrix spike and duplicate and sample duplicate	1 per analytical batch	80%≤%Rec.≤120%; %RPD<20%; If spike(s) outside of limits, analyze PDS.	If matrix spike recovery does not meet criteria (except Ag), a post digestion spike is required for all methods except GFAA. Qualify results in accordance with Regional criteria.	Lab Representative	Accuracy/ Bias	80%≤%Rec.≤ 120%; %RPD<20%; If spike(s) outside of limits, analyze PDS.
Duplicate Sample	5% of field samples per matrix	RPD ≤ 25% aqueous; ≤ 35% solid	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance	Project Manager/Field Team Leader	Accuracy/ Bias/Contamination	To ensure the decontamination of sampling equipment has been adequately performed; to

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
			criteria.			assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservatio n/Bias	To verify sample cooler temperature during transport.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/ Bias/Conta mination	To characterize decontaminati on water source; not applicable if laboratory grade deionized water is used for decontaminati on rinse

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical Test for Backfill Requirements

Matrix	Soil
Analytical Group	PCBs[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-01
Analytical Method / SOP Reference	SW-8082 GCS10/SOC-8082Ar
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories/Columbia Analytical Services
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits			Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent reference standard (LCS)	1 per batch	Specified QC Limits (See List in Appendix B)			Qualify associated data biased high or biased low as appropriate.	Lab Representative	Accuracy/Bias	Specified QC Limits (See List in Appendix B)
Method blanks	1 per batch	No target analytes detected ≥ Reporting Limits			Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank. Document source of contamination.	Lab Representative	Accuracy/Bias/Contamination	No target analytes detected ≥ Reporting Limits
Matrix spike and matrix spike duplicate (1016/1260 mix)	1 per 20 samples per matrix	Standards PCB-1016 PCB-1260	Aqueous %R 25–145 30–145 RPD ≤ 30 for all	Solid %R 40–140 60–130 RPD ≤50 for all	If possible, MS/MSDs will be reprepped and reanalyzed. Data reviewer may use the MS and MSD results in conjunction with other QC sample results to determine the need for some qualification of the data.	Lab Representative	Accuracy/Bias	QC Limits (See List in Appendix B)
Duplicate Sample	5% of field samples per matrix	≤50% RPD, Specified QC Limits (See List L in Appendix B)			Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples	No target analytes			Data reviewer should	Project	Accuracy/Bi	To ensure the

Matrix	Soil
Analytical Group	PCBs[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-01
Analytical Method / SOP Reference	SW-8082 GCS10/SOC-8082Ar
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories/Columbia Analytical Services
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
	or 1 per day		review and judge all of the target compounds against the acceptance criteria.	Manager/Field Team Leader	as/Contamination	decontamination of sampling equipment has been adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservation /Bias	To verify sample cooler temperature during transport.

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical Test for Backfill Requirements

Matrix	Soil
Analytical Group	Pesticides[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-01
Analytical Method / SOP Reference	SW-8081 GCS09
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blanks	Per extraction batch	No analytes detected ≥ LOQ	Document source of contamination.	Lab Representative	Accuracy/Bias/Contamination	No analytes detected ≥ LOQ
LCS	Every batch	Specified QC Limits (See List in Appendix B)	Correct problem then reanalyze. If still out, reprep and reanalyze the LCS and all samples in the affected analytical batch. Qualify associated data biased high or biased low.	Lab Representative	Accuracy/Bias	Specified QC Limits (See List in Appendix B)
Matrix spike and duplicate	1 per 20 samples per matrix	Specified QC limits (See List in Appendix B)	If MS/MSD results do not meet criteria, the reviewer should review the data in conjunction with other QC results to determine if the problem is specific to the QC samples or systematic. If possible, MS/MSDs will be reprepped and reanalyzed.	Lab Representative	Accuracy/Bias	Specified QC limits (See List in Appendix B)
Duplicate Sample	5% of field samples per matrix	≤50% RPD, Specified QC Limits (See List in Appendix B)	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Precision	To ensure precision in sample homogeneity during collection and analysis
Rinse Blank	1 per 20 samples or 1 per day	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/Bias/Contamination	To ensure the decontamination of sampling equipment has been

Matrix	Soil
Analytical Group	Pesticides[2]
Concentration Level	Low
Sampling SOP	Soil: 12-SSP-01
Analytical Method / SOP Reference	SW-8081 GCS09
Sampler's Name	Field Team
Field Sampling Organization	KEMRON Environmental
Analytical Organization	Microbac Laboratories
No. of Sample Locations	TBD[1]

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
						adequately performed; to assess cross contamination and/or incidental contamination to the sample container.
Temperature Blank	1 temperature blank per cooler.	Temperatures inside cooler are within acceptable range upon arrival at the laboratory.	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Sample preservation /Bias	To verify sample cooler temperature during transport.
Source Water	1 per source	No target analytes	Data reviewer should review and judge all of the target compounds against the acceptance criteria.	Project Manager/Field Team Leader	Accuracy/Bias/Contamination	To characterize decontamination water source; not applicable if laboratory grade deionized water is used for decontamination rinse.

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical Test for Backfill Requirements

QAPP Worksheet #30 Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations / ID Numbers[1]	Analytical SOP[2]	Data Package Turnaround Time	Laboratory / Organization (Name and Address, Contact Person and Tel. No.)	Backup Laboratory / Organization[3] (Name and Address, Contact Person and Tel. No.)[3]
Soil and Groundwater	8260B	Low	See Worksheets 18 and 20	**MSV01**	Hardcopy Level 2 CLP Like & EDD = 14 Calendar Days	Microbac Laboratories, Inc. 158 Starlite Drive Marietta, OH 45750 Stephanie Mossburg (740) 373-4071	NA
Soil and Groundwater	8270 SIM	Low	See Worksheets 18 and 20	**MSS03**			NA
Soil	8015D[4]	Low	See Worksheets 18 and 20	**GCV05 GCS02**			NA
Soil	6010B[4]	Low	See Worksheets 18 and 20	**ME600F**			NA
Soil	8082A[4]	Low	See Worksheets 18 and 20	**GCS10/SOC-8082A**			NA
Soil	8081[4]	Low	See Worksheets 18 and 20	**GCS09**			NA

[1] The exact number of sampling locations are to be determined (TBD) based on the field observations of UST size, shape, and primary soil sample results. Detail of the Sampling Rational can be found on Worksheet 17.
[2] Analytical SOPs are included in Appendix F.
[3] A Secondary or Backup Laboratory has not been established for this project. In the event that a modification is required and a backup laboratory is to be used, then KEMRON will notify the Navy and provide current NELAP and DoD ELAP certifications for the backup lab.
[4] Analytical Test for Backfill Requirements

QAPP Worksheet #34
Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (Name, Organization)
Field Notes	Field notes will be collected at the end of each sampling event and reviewed for completeness against any collected and packed samples. Any required corrective actions will be addressed and implemented prior to the next sampling session. Field notes will be transcribed into the final project report and copies will be maintained with the project file.	I	Project Manager and/or Field Team Leader, KEMRON
Sample Plan	Provides the location and specific analytes for each sampling point. The collected samples will be compared to the original sampling plan to determine completeness of the project.	I	Project Manager and/or Field Team Leader, KEMRON
Sample Method	The sampling method will be included in this QAPP as an SOP. Documentation of adherence to the SOP will be reviewed and maintained in the project file.	I	Project Manager and/or Field Team Leader, KEMRON
Sample Instrument Calibration Log	Instruments used during sample collection will be calibrated according to this QAPP and manufacturers' criteria. All calibration records will be reviewed daily and maintained in the project file.	I	Project Manager and/or Field Team Leader, KEMRON
Sample Collection, Containment, and ID	Sample collection, containment, and identification will be conducted under the SOPs referenced in this QAPP. Each of these measures will be reviewed prior to sample packaging and submittal.	I	Project Manager and/or Field Team Leader, KEMRON
QC Sample Collection and Identification	QC samples will be collected and identified according to the frequencies and labeling systems defined in this QAPP. QC sampling will be reviewed daily as samples are collected.	I	Project Manager and/or Field Team Leader, KEMRON
Sample Handling Procedures	Sample handling procedures will be reviewed prior to packaging and shipment of samples. Any records will be maintained in the project file.	I	Project Manager and/or Field Team Leader, KEMRON
Chain of Custody	Chain of Custody forms will be reviewed when samples are collected for delivery to the laboratory both in the field and at the laboratory. The forms will be maintained in the project file.	I/E	Project Manager and/or Field Team Leader, KEMRON and Lab Representative, Microbac
Sample Condition Upon Receipt	The laboratory will note the conditions of the samples upon arrival including, temperature, presence of ice, custody seals, signed COC, intact containers, note holding times and preservative, etc.	E	Lab Representative, Microbac
Analysis Requested	The analysis requested for each sample on the COC will be compared against the original Sampling Plan to ensure the proper analysis is conducted on each sample.	I/E	Project Manager, KEMRON and Lab Representative, Microbac
Analytical Data Package	All laboratory data packages will be verified internally by the laboratory performing the work for completeness prior to submittal.	I/E	Project Manager, KEMRON and Lab Representative, and Microbac

Verification Input	Description	Internal / External	Responsible for Verification (Name, Organization)
	All Received data packages will be review externally according to the data validation procedures specified in worksheet #36		
Waste Characterization and sample collection, and profiling	Ensure that waste characterization and sample collection is representative of the waste stream. Profiles for each waste stream will be reviewed against the project analytical records for representativeness. Records will be kept in the project file.	I	Project Manager and/or Field Team Leader, KEMRON

QAPP Worksheet #35
Validation (Step IIa and IIb) Process Table

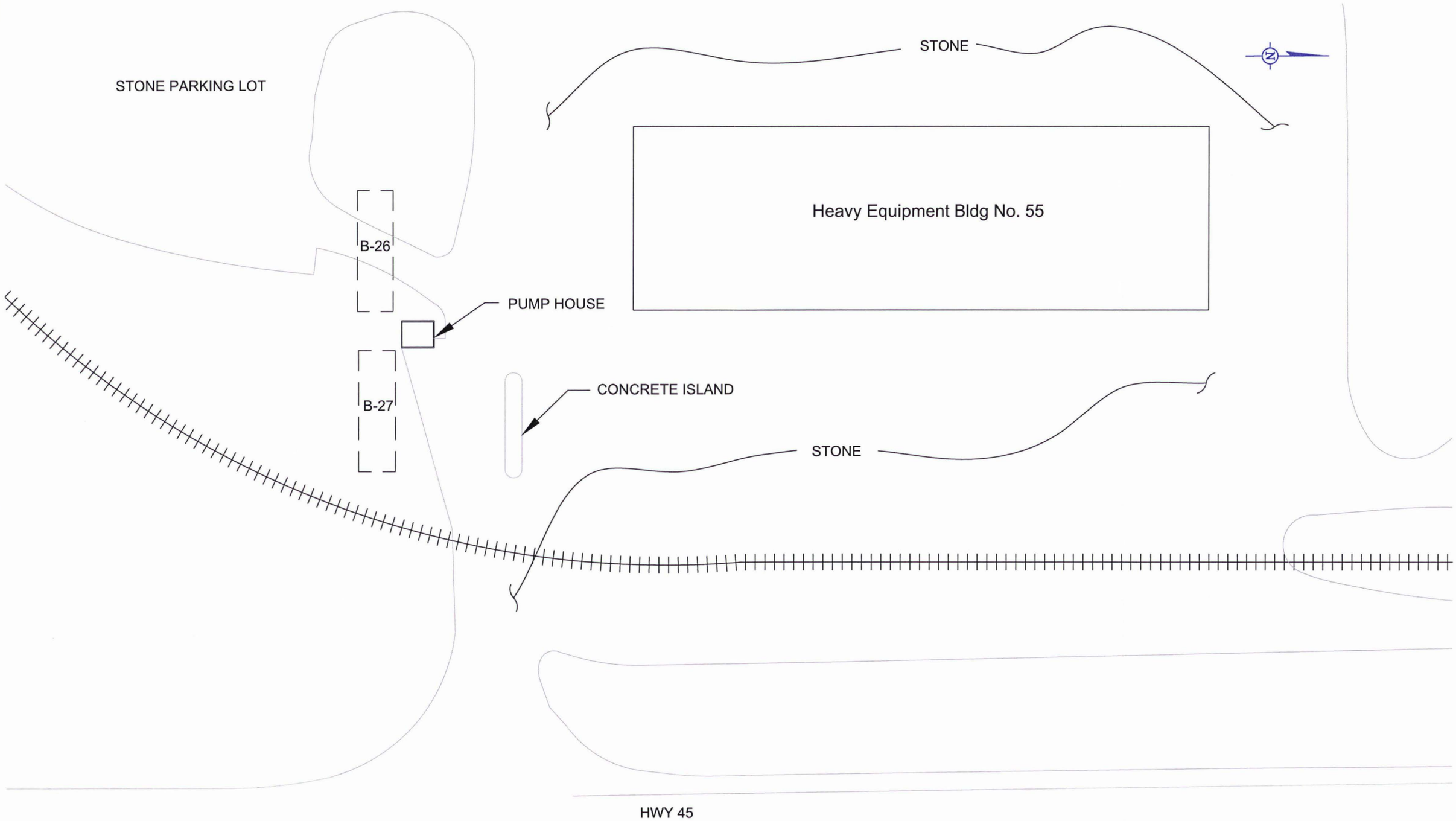
Step IIa / IIb	Validation Input	Description	Responsible for Verification (Name, Organization)
IIa	Evaluate Data Deliverables, analytes, chain-of-custody	Review of the following elements for completeness and accuracy per site specific sampling and analysis plan: • Number, type and location of samples collected; • SOP and site specific plan conformance of sampling methods/procedures; • Analyses requested by sample and matrix; • Chain-of-custody procedures	KEMRON QA Manager and SS
IIa	Evaluate sample receipt, preservation and holding times on 100% of the data	Review of the following elements for completeness and accuracy per US EPA established standards: • Chain of custody forms; • Sample handling procedures; • Analyses requested; • Sample IDs; • Sample holding times; • Sample Preservation; • Cooler receipt forms.	Microbac Laboratories, Inc. and KEMRON QA Manager

QAPP Worksheet #36
Validation (Step IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Name, Title, Organization)
IIa	100% Soil	VOCs	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Soil	PAHs	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Soil	TPH[1]	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Soil	Metals[1]	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Soil	PCBs[1]	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Soil	Pesticides[1]	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Water	VOCs	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS
IIa	100% Water	PAHs	Low	QAPP Worksheets #12, #15, #24, and #28	KEMRON QA Manager and SS

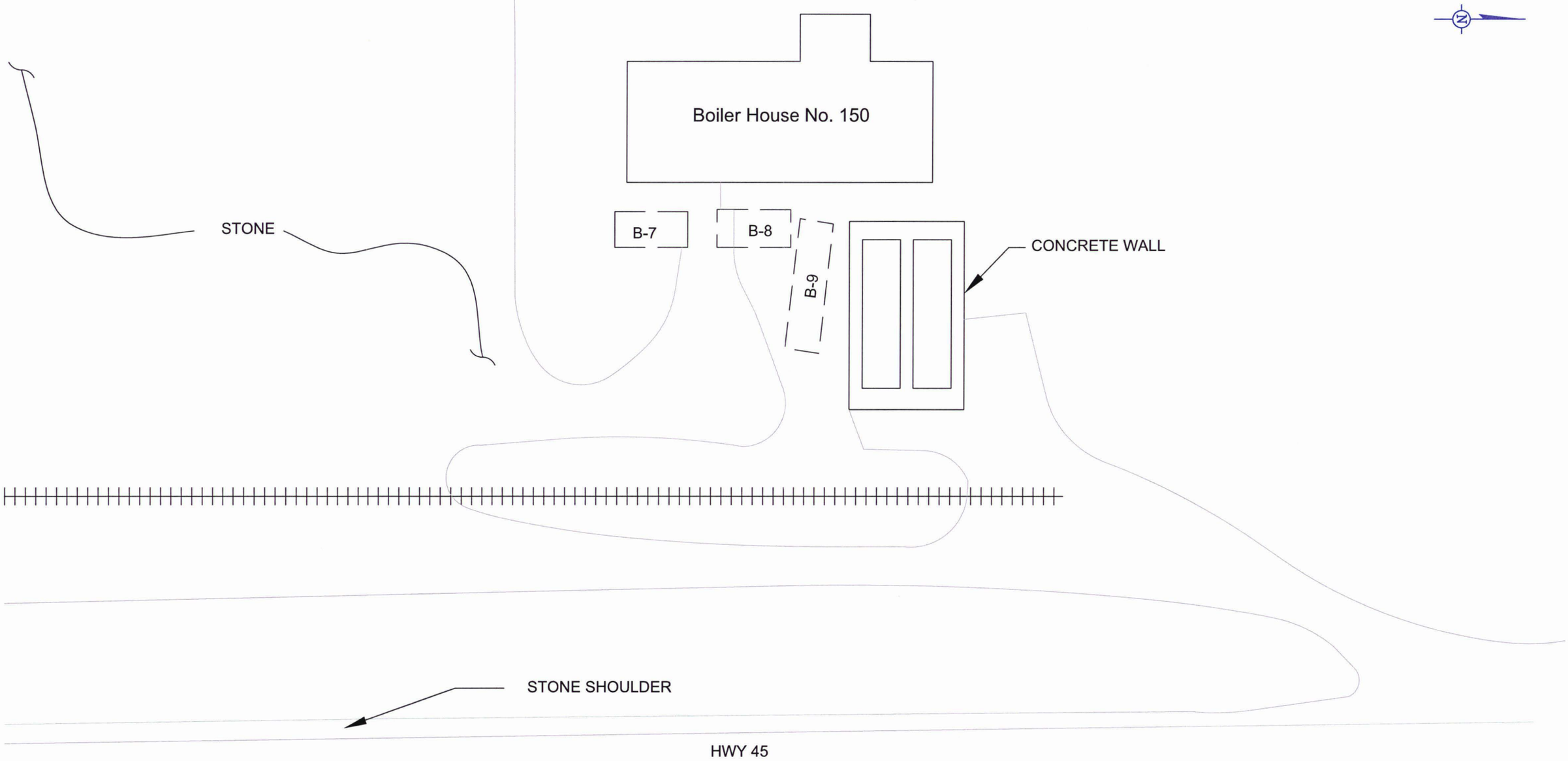
[1]Analytical Test for Backfill Requirements

FIGURES



KEMRON ENVIRONMENTAL SERVICES	0 10 40 SCALE 1"=20'	DRAWN BY: JRM	DATE 24 AUG. 2012	FIGURE-1A	SITE MAP BOILER HOUSE NO. 55
		REVIEWED: JG R. 1	PROJECT NO. CH0020.001		U.S. NAVAL AMMUNITION DEPOT CRANE, INDIANA
		APPROVED: JG	DWG. FILE NO. two bldgs.dwg		

PLOTTED: September 11, 2012 – 3:43 PM, BY: Jarvis Miller



FILE PATH: (LOCATION) C:\USERS\JMILLER\DOCUMENTS\RFP\CRANE BLDGS\TWO BLDGS.DWG [BL 150] 8/24/12

KEMRON ENVIRONMENTAL SERVICES	0 10 40 SCALE 1"=20'	DRAWN BY: JRM	DATE 24 AUG. 2012	FIGURE-1B	SITE MAP BOILER HOUSE NO. 150
		REVIEWED: JG R. 5	PROJECT NO. CH0020.001		U.S. NAVAL AMMUNITION DEPOT CRANE, INDIANA
		APPROVED: JG	DWG. FILE NO. two bldgs.dwg		

APPENDIX A
Laboratory Certification

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

MICROBAC LABORATORIES, INC.
OHIO VALLEY DIVISION
158 Starlite Drive
Marietta, OH 45750
Leslie Bucina Phone: 740-373-4071
Email address: lbucina@microbac.com

ENVIRONMENTAL

Valid To: December 31, 2013 Certificate Number: 2936.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, LC/MS/MS, Ion Chromatography, Misc.-Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), IR Spectrometry, Titrimetry, Total Organic Carbon

Parameter/Analyte	**Nonpotable Water (1)**	**Solid and Chemical Materials (2)**
Metals		
Aluminum	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C

5301 Buckeystown Pike, Suite 350 | Frederick, Maryland 21704-8373 | Phone: 301 644 3248 | Fax: 301 662 2974 | www.A2LA.org

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Antimony	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Arsenic	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Barium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Beryllium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Boron	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Cadmium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Calcium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Chromium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Cobalt	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
	EPA 3015A/6020A	
Copper	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Iron	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Lead	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Lithium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Magnesium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Manganese	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Mercury	EPA 245.1 EPA 7470A	EPA 7471A EPA 7471B
Molybdenum	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Nickel	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Phosphorus	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Potassium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Selenium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Silicon	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	----------
Silver	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Sodium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Strontium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Thallium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Tin	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Titanium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Uranium	EPA 200.8 EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Vanadium	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Zinc	EPA 200.7 EPA 200.8 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C EPA 3015/6020 EPA 3015A/6020 EPA 3015/6020A EPA 3015A/6020A	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C EPA 3051/6020 EPA 3051A/6020 EPA 3051/6020A EPA 3051A/6020A
Zirconium	EPA 200.7 EPA 3005A/6010B EPA 3015/6010B EPA 3015A/6010B EPA 3005A/6010C EPA 3015/6010C EPA 3015A/6010C	EPA 3051/6010B EPA 3051A/6010B EPA 3051/6010C EPA 3051A/6010C
Nutrients		
Ammonia (as N)	EPA 350.1 SM 4500-NH3 B	EPA 350.1 SM 4500-NH3 B
Kjeldahl nitrogen	EPA 351.2	----------
Nitrate (as N)	EPA 300.0 EPA 9056 EPA 9056A EPA 353.2	EPA 9056 EPA 9056A
Nitrate-nitrite (as N)	EPA 300.0 EPA 9056 EPA 9056A EPA 353.2	EPA 9056 EPA 9056A
Nitrite (as N)	EPA 300.0 EPA 9056 EPA 9056A EPA 354.1	EPA 9056 EPA 9056A
Orthophosphate (as P)	EPA 365.2 SM 4500-P E	EPA 365.2
Total phosphorus	EPA 365.4	----------
Demands		
Biochemical oxygen demand	SM 5210 B	----------
Chemical oxygen demand	EPA 410.4 HACH 8000	----------
Total organic carbon	EPA 415.1 EPA 9060A SM5310 C	LLOYDKAHN

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Wet Chemistry		
Alkalinity	EPA 310.1 EPA 310.2 SM 2320 B	----------
Bromide	EPA 300.0 EPA 9056 EPA 9056A	EPA 9056 EPA 9056A
Chloride	EPA 300.0 EPA 9056 EPA 9056A EPA 325.2 SM 4500-CL E	EPA 9056 EPA 9056A EPA 325.2 SM 4500-CL E
Cyanide	SM 4500 CN-C,E EPA 9010C/9014	EPA 9010C/9014
Amenable Cyanide	SM 4500 CN-G EPA 9010C/9014	EPA 9010C/9014
Fluoride	EPA 300.0 EPA 9056 EPA 9056A SM 4500 F,C	EPA 300.0 EPA 9056 EPA 9056A SM 4500 F,C
pH	SM 4500-H^+ B EPA 9040C	EPA 9040C EPA 9045D
Oil and Grease	EPA 1664A	EPA 1664A
Phenols	EPA 420.1	EPA 420.1
Total residue	EPA 160.3 SM 2540 B	----------
Filterable residue	EPA 160.1 SM 2540 C	----------
Nonfilterable residue	EPA 160.2 SM 2540 D	----------
Sulfate	EPA 300.0 EPA 9056 EPA 9056A EPA 375.4 SM 426C	EPA 9056 EPA 9056A EPA 375.4 SM 426 C
Sulfide	EPA 376.1 SM 4500-S F	EPA 9030B/9034
Flashpoint	EPA 1010A	EPA 1010A
Ferrous Iron	SM 3500-Fe B	----------
Hexavalent chromium	SM 3500-Cr D 19th Ed SM 3500-Cr B 20th and 21st Ed EPA 7196A	SM 3500-Cr D 19th Ed SM 3500-Cr B 20th and 21st Ed

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Purgeable Organics (Volatiles)		
Acetone	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Acetonitrile	EPA 624 EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Acrolein	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Acrylonitrile	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Allyl chloride	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
T-amylmethylether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Benzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Bromobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Bromochloromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Bromodichloromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Bromoform	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Bromomethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,3-Butadiene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
2-Butanone	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
n-Butyl alcohol	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
ert-Butyl alcohol	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
n-Butylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Sec-Butylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Tert-Butylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Carbon disulfide	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Carbon tetrachloride	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Chlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Chloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
-Chloroethyl vinyl ether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Chloroform	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Chloroprene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1-Chlorohexane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Chloromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
2-Chlorotoluene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
4-Chlorotoluene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Cyclohexane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Cyclohexanone	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Dibromochloromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Dibromofluoromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624 EPA 5030B/8260B EPA 5030C/8260B EPA 8011	EPA 5035/8260B EPA 5035A/8260B
Dibromomethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2Dibromomethane (EDB)	EPA 624 EPA 5030B/8260B EPA 5030C/8260B EPA 8011	EPA 5035/8260B EPA 5035A/8260B
1,2-Dichlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,3-Dichlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,4-Dichlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Trans-1,4-Dichloro-2-butene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Dichlorodifluoromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1-Dichloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2-Dichloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1-Dichloroethene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
cis-1,2-Dichloroethene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
trans-1,2-Dichloroethene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
1,2-Dichloropropane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,3-Dichloropropane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
2,2-Dichloropropane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1-Dichloropropene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
cis-1,3-Dichloropropene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
trans-1,3-Dichloropropene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Diethyl ether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Di-isopropyl ether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Dimethyldisulfide	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Dimethyl sulfide	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,4-Dioxane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Ethyl acetate	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Ethyl –t-butyl ether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Ethyl methacrylate	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Ethyl benzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Gas Range Organics (GRO)	EPA 5030B/8015B EPA 5030C/8015B EPA 5030B/8015C EPA 5030C/8015C EPA 5030B/8015D EPA 5030C/8015D NWTPH GX WADOE VPH MA VPH C5-C8 Aliphatics MA VPH C9-C12 Aliphatics MA VPH C9-C10 Aromatics	EPA 5035/8015B EPA 5035/8015C EPA 5035/8015D EPA 5035A/8015B EPA 5035A/8015C EPA 5035A/8015D NWTPH GX WADOE VPH MA VPH C5-C8 Aliphatics MA VPH C9-C12 Aliphatics MA VPH C9-C10 Aromatics
2-Hexanone	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Hexachlorobutadiene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Isoprene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Isopropylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,4-Isopropyltoluene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Iodomethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Isobutyl alcohol	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Isopropyl alcohol	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Methacrylonitrile	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Methyl acetate	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Methylcyclohexane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Methyl methacrylate	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Alpha-Methylstyrene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Methyl tert-butyl ether	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Methylene chloride	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
4-Methyl-2-pentanone	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Naphthalene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
2-Nitropropane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
n-Propylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Propionitrile	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Styrene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1,1,2-Tetrachloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1,2,2-Tetrachloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Tetrachloroethene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Tetrahydrofuran	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Toluene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1,1-Trichloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,1,2-Trichloroethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Trichloroethene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Trichlorofluoromethane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2,3-Trichlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2,3-Trichloropropane	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2,4-Trichlorobenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2,4-Trimethylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,3,5-Trimethylbenzene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Vinyl acetate	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Vinyl chloride	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Xylenes, total	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,2-Xylene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,3-Xylene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
1,4-Xylene	EPA 624 EPA 5030B/8260B EPA 5030C/8260B	EPA 5035/8260B EPA 5035A/8260B
Headspace Organics		
Carbon dioxide	EPA 5021/RSK175	----------
Methane	EPA 5021/RSK175	----------
Ethane	EPA 5021/RSK175	----------
Ethene	EPA 5021/RSK175	----------
Acetylene	EPA 5021/RSK175	----------

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Acenaphthene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Acenaphthylene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Acetophenone	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Acetylaminofluorene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
4-Aminobiphenyl	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Anilene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Anthracene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Aramite	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
;enzidine	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Benzoic acid	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
enzo (a) anthracene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Benzo (b) fluoranthene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Benzo (k) fluoranthene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Benzo (ghi) fluoranthene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Benzo (a) pyrene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Benzyl alcohol	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Benzaldehyde	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Biphenyl	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Bis(2-chloroethoxy) methane	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Bis (2-chloroethyl) ether	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Bis(2-chloroisopropyl) ether	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Bis (2-ethylhexyl) phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
4-Bromophenylphenylether	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Butyl benzyl phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
:aprolactam	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Carbazole	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
-Chloroaniline	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Chlorobenzilate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
4-Chloro-3-methylphenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1-Chloronaphthalene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Chloronaphthalene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Chlorophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
-Chlorophenylphenyl ether	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Chrysene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
resols	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Diallate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Dibenzo (a,c) anthracene	EPA 625 EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Dibenzo (a,h) anthracene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Dibenzofuran	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1,2-Dichlorobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,3-Dichlorobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1,4-Dichlorobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
,3'-Dichlorobenzidine	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2,4-Dichlorophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
2,6-Dichlorophenol	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Diethyl phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Dimethoate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
4-Dimethylaminoazobenzene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,12-Dimethylbenz(a)anthracene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
3,3'-Dimethylbenzidine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
lpha-,alpha- Dimethylphenethylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2,4-Dimethylphenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Dimethyl phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Di-n-butyl phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Di-n-octyl phthalate	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2,4-Dinitrophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,4-Dinitrotoluene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2,6-Dinitrotoluene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
4-Dioxane	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Diphenylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
1,2-Diphenylhydrazine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Disulfoton	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
DRO/ORO	EPA 3510C/8015B EPA 3510C/8015C EPA 3510C/8015D NWTPH DX WADOE EPH MA EPH C9-C18 Aliphatics MA EPH C19-C36 Aliphatics MA EPH C11-C22 Aromatics, Unadjusted	EPA 3545/8015B EPA 3545A/8015B EPA 3550B/8015B EPA 3550C/8015B EPA 3545/8015C EPA 3545A/8015C EPA 3550B/8015C EPA 3550C/8015C EPA 3545/8015D EPA 3545A/8015D EPA 3550B/8015D EPA 3550C/8015D EPA 3546/8270C EPA 3546/8270D NWTPH DX WADOE EPH MA EPH C9-C18 Aliphatics MA EPH C19-C36 Aliphatics MA EPH C11-C22 Aromatics, Unadjusted

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
thyl methanesulfonate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Ethyl parathion	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
amphur	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Fluoroanthene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Fluorene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Hexachlorobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Hexachlorobutadiene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Hexachlorocyclopentadiene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
lexachloroethane	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Hexachlorophene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
lexachloropropene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Indeno (1,2,3-cd) pyrene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Isodrin	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Isophorone	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Isosafrole	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Kepone	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Methapyrilene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
3-Methylcholanthrene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
-Methyl-4,6-Dinitrophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Methyl methanesulfonate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
1-Methylnaphthalene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Methylnaphthalene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Methyl parathion	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Naphthalene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,4-Naphthoquinone	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1-Naphthylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
-Naphthylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Nitroaniline	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
3-Nitroaniline	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
4-Nitroaniline	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Nitrobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
5-Nitro-o-toluidine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
2-Nitrophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
4-Nitrophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Nitroquinoline-1-oxide	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
n-Nitrosodiethylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
n-Nitrosodimethylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
n-Nitroso-di-n-butylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
n-Nitrosodi-n-propylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
n-Nitrosodiphenylamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
-Nitrosomorpholine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
n-Nitrosopiperidine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
-Nitrosopyrrolidine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pentachlorobenzene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Pentachloroethane	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pentachloronitobenzene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pentachlorophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Perylene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
'henacetin	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Phenanthrene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
'henol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1,4-Phenylenediamine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Phorate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2-Picoline	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pronamide	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pyrene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
yridine	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Safrole	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
ulfotepp	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1,2,4,5-Tetrachlorobenzene	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
2,3,4,6-Tetrachlorophenol	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
o,o,o-Triethyl phosphorothioate	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Thionazin	EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
1,2,4-Trichlorobenzene	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,4,5-Trichlorophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
2,4,6-Trichlorophenol	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
)-Toluidine	EPA 625 EPA 3510C/8270C EPA 3520C/8270C EPA 3510C/8270D EPA 3520C/8270D	EPA 3545/8270C EPA 3545A/8270C EPA 3550B/8270C EPA 3550C/8270C EPA 3580A/8270C EPA 3545/8270D EPA 3545A/8270D EPA 3550B/8270D EPA 3550C/8270D EPA 3580A/8270D EPA 3546/8270C EPA 3546/8270D
Pesticides/Herbicides/PCBs		
Aldrin	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
alpha-BHC	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Beta-BHC	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
delta-BHC	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
gamma-BHC	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Chlordane (technical)	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
alpha-chlordane	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
gamma-chlordane	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
4,4'-DDD	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
4,4'-DDE	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
,4',-DDT	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Dieldrin	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endosulfan I	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endosulfan II	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endonsulfan sulfate	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endrin	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endrin aldehyde	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Endrin ketone	EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
Heptachlor	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Heptachlor epoxide	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Methoxychlor	EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
Toxaphene	EPA 608 EPA 3510C/8081A EPA 3510C/8081B	EPA 3550B/8081A EPA 3550C/8081A EPA 3580A/8081A EPA 3550B/8081B EPA 3550C/8081B EPA 3580A/8081B
PCB-1016 (Aroclor)	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
PCB-1221	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
PCB-1232	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
PCB-1242	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
PCB-1248	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
PCB-1254	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
PCB-1260	EPA 608 EPA 3510C/8082 EPA 3510C/8082A	EPA 3550B/8082 EPA 3550C/8082 EPA 3580A/8082 EPA 3550B/8082A EPA 3550C/8082A EPA 3580A/8082A
2,4-D	EPA 8151A	EPA 8151A
Dalapon	EPA 8151A	EPA 8151A
2,4-DB	EPA 8151A	EPA 8151A
Dicamba	EPA 8151A	EPA 8151A
Dichloroprop	EPA 8151A	EPA 8151A
Dinoseb	EPA 8151A	EPA 8151A
ICPA	EPA 8151A	EPA 8151A
MCPP	EPA 8151A	EPA 8151A
Pentachlorophenol	EPA 8151A	EPA 8151A
2,4,5-T	EPA 8151A	EPA 8151A
2,4,5-TP	EPA 8151A	EPA 8151A
HPLC		
1,3,5-Trinitrobenzene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
1,3-Dinitrobenzene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
2,4,6-Trinitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
2,4-Dinitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
2,6-Dinitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
2-Amino-4,6-dinitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
2-Nitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
3-Nitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Amino-2,6-dinitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B

Parameter/Analyte	Nonpotable Water (1)	Solid and Chemical Materials (2)
4-Nitrotoluene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Nitrobenzene	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Nitroglycerin	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
HMX	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
PETN	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
RDX	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Tetryl	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Nitroglycerin	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Nitroguanidine	EPA 3535A/8330A EPA 3535A/8330B	EPA 8330A EPA 8330B
Nitrocellulose	EPA USATHAMA/353.2/353.3	EPA USATHAMA/353.2/353.3
Hazardous Waste Characteristics		
Corrosivity	EPA 9040C	EPA 9040C EPA 9045D
Ignitibility	EPA 1010A	EPA 1010A
Reactivity	EPA SW 846 Ch 7	EPA SW 846 Ch 7
Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311	EPA 1311
LC/MS/MS		
Perchlorate	EPA 6850 EPA 331	EPA 6850
PFOA/PFOS	EPA 8321B	EPA 8321B
Benzotriazole	EPA 8321B	----------
Tolytriazole	EPA 8321B	----------

(1) Method List includes Clean Water Act and RCRA water parameters.
(2) Method List includes RCRA parameters only.

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A2LA has accredited

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OHIO VALLEY DIVISION

Marietta, OH

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality System Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 30th day of January 2012.

President & CEO
For the Accreditation Council
Certificate Number 2936.01
Valid to December 31, 2013
Revised June 1, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

APPENDIX B

Laboratory Quality Control Limits

TPH Water

Compound	CAS Number	Units	LCL	UCL	RPD
Gasoline Range Organics	8006-61-9	ug/kg	85	120	20
Diesel Range (C10-C28)	68334-30-5	ug/kg	32	160	45
Chlorobenzene(s)	108-90-7	ug/kg	74	138	
o-Terphenyl	84-15-1	ug/kg	49	174	
Octacosane	630-02-4	ug/kg	26	152	

TPH Solid

Compound	CAS Number	Units	LCL	UCL	RPD
Gasoline Range Organics	8006-61-9	ug/kg	85	120	20
Diesel Range (C10-C28)	68334-30-5	ug/kg	53	127	26
Chlorobenzene(s)	108-90-7	ug/kg	64	148	
o-Terphenyl	84-15-1	ug/kg	43	136	
Octacosane	630-02-4	ug/kg	25	162	

List K Pesticides in Soils

Compound	CAS Number	Units	LCL	UCL	RPD
Aldrin	309-00-2	ug/L	45	140	30
Alpha-BHC	319-84-6	ug/L	50	145	30
Beta-BHC	319-85-7	ug/L	55	130	30
Delta-BHC	319-86-8	ug/L	55	130	30
Gamma-BHC (Lindane)	58-89-9	ug/L	50	135	30
Alpha-Chlordane	5103-71-9	ug/L	65	130	30
Gamma-Chlordane	57-74-9	ug/L	55	135	30
4,4'-DDD	72-54-8	ug/L	40	140	30
4,4'-DDE	72-55-9	ug/L	70	140	30
4,4'-DDT	50-29-3	ug/L	50	150	30
Dieldrin	60-57-1	ug/L	65	125	30
Endosulfan I	959-98-8	ug/L	30	130	30
Endosulfan II	33213-65-9	ug/L	35	140	30
Endosulfan sulfate	1031-07-8	ug/L	55	135	30
Endrin	72-20-8	ug/L	60	140	30
Endrin Aldehyde	7421-93-4	ug/L	35	145	30
Endrin Ketone	53494-70-5	ug/L	60	130	30
Heptachlor	76-44-8	ug/L	50	140	30
Heptachlor expoxide	1024-57-3	ug/L	50	120	30
Methoxychlor	72-43-5	ug/L	55	145	30
Toxaphene	8022-04-6	ug/L	25	138	30
			0	0	0
			0	0	0
			0	0	0
2,4,5,6-tetrachloro-m-xylene	877-09-8	% Recovery	39	130	
Decachlorobiphenly	2051-24-3	% Recovery	33	143	

List J VOCs Solid

Compound	CAS Number	Units	LCL	UCL	RPD
1,1,1,2-Tetrachloroethane	630-20-6	ug/kg	71	137	30
1,1,1-Trichloroethane	71-55-6	ug/kg	70	135	30
1,1,2,2-Tetrachloroethane	79-34-5	ug/kg	55	130	30
1,1,2-Trichloroethane	79-00-5	ug/kg	60	125	30
1,1-Dichloroethane	75-34-3	ug/kg	75	125	30
1,1-Dichloroethene	75-35-4	ug/kg	65	135	30
1,1-Dichloropropene	563-58-6	ug/kg	57	138	30
1,2,3-Trichlorobenzene	87-61-6	ug/kg	60	135	30
1,2,3-Trichloropropane	96-18-4	ug/kg	65	130	30
1,2,4-Trichlorobenzene	120-82-1	ug/kg	65	130	30
1,2,4-Trimethylbenzene	95-63-6	ug/kg	75	132	30
1,2-Dibromo-3-chloropropane	96-12-8	ug/kg	40	135	30
1,2-Dibromoethane	106-93-4	ug/kg	69	128	30
1,2-Dichlorobenzene	95-50-1	ug/kg	70	130	30
1,2-Dichloroethane	107-06-2	ug/kg	63	133	30
1,2-Dichloropropane	78-87-5	ug/kg	72	130	30
1,3,5-Trimethylbenzene	108-67-8	ug/kg	74	133	30
1,3-Dichlorobenzene	541-73-1	ug/kg	70	130	30
1,3-Dichloropropane	142-28-9	ug/kg	65	128	30
1,4-Dichlorobenzene	106-46-7	ug/kg	70	130	30
2,2-Dichloropropane	594-20-7	ug/kg	66	135	30
2-Butanone	78-93-3	ug/kg	37	180	30
2-Chloroethyl vinyl ether	110-75-8	ug/kg	35	154	30
2-Chlorotoluene	95-49-8	ug/kg	63	147	30
2-Hexanone	591-78-6	ug/kg	45	145	30
4-Chlorotoluene	106-43-4	ug/kg	70	138	30
4-Methyl-2-pentanone	108-10-1	ug/kg	47	146	30
Acetone	67-64-1	ug/kg	20	160	30
Benzene	71-43-2	ug/kg	70	130	30
Bromobenzene	108-86-1	ug/kg	72	131	30
Bromochloromethane	74-97-5	ug/kg	70	130	30
Bromodichloromethane	75-27-4	ug/kg	72	137	30
Bromoform	75-25-2	ug/kg	49	146	30
Bromomethane	74-83-9	ug/kg	37	143	30
Carbon disulfide	75-15-0	ug/kg	39	139	30
Carbon tetrachloride	56-23-5	ug/kg	59	136	30
Chlorobenzene	108-90-7	ug/kg	70	130	30
Chloroethane	75-00-3	ug/kg	52	135	30
Chloroform	67-66-3	ug/kg	74	129	30
Chloromethane	74-87-3	ug/kg	30	131	30
cis-1,2-Dichloroethene	156-59-2	ug/kg	70	130	30
cis-1,3-Dichloropropene	10061-01-5	ug/kg	70	142	30
Chlorodibromomethane	124-48-1	ug/kg	59	136	30
Dibromomethane	74-95-3	ug/kg	69	130	30
Dichlorodifluoromethane	75-71-8	ug/kg	25	130	30
Ethylbenzene	100-41-4	ug/kg	70	130	30

List J VOCs Solid

Compound	CAS Number	Units	LCL	UCL	RPD
Hexachlorobutadiene	87-68-3	ug/kg	65	135	30
Isopropylbenzene	98-82-8	ug/kg	68	129	30
m-,p-Xylene	179601-23-1	ug/kg	70	130	30
Methylene chloride	75-09-2	ug/kg	74	128	30
n-Butylbenzene	104-51-8	ug/kg	70	136	30
n-Propylbenzene	103-65-1	ug/kg	72	136	30
Naphthalene	91-20-3	ug/kg	50	146	30
o-Xylene	95-47-6	ug/kg	70	130	30
p-Isopropyltoluene	99-87-6	ug/kg	72	128	30
sec-Butylbenzene	135-98-8	ug/kg	71	132	30
Styrene	100-42-5	ug/kg	74	130	30
tert-Butylbenzene	98-06-6	ug/kg	72	130	30
Tetrachloroethene	127-18-4	ug/kg	72	130	30
Toluene	108-88-3	ug/kg	77	126	30
trans-1,2-Dichloroethene	156-60-5	ug/kg	72	127	30
trans-1,3-Dichloropropene	10061-02-6	ug/kg	65	139	30
Trichloroethene	79-01-6	ug/kg	72	126	30
Trichlorofluoromethane	75-69-4	ug/kg	48	154	30
Vinyl acetate	108-05-4	ug/kg	10	150	30
Vinyl chloride	75-01-4	ug/kg	45	140	30
Chlorobenzene-d5	3114-55-4		0	0	0
Fluorobenzene	462-06-6		0	0	0
1,4-Dichlorobenzene-d4	3855-82-1		0	0	0
4-Bromofluorobenzene	460-00-4	% Recovery	74	121	
Toluene-d8	2037-26-5	% Recovery	81	117	
1,2-Dichloroethane-d4	17060-07-0	% Recovery	80	120	
Dibromofluoromethane	1868-53-7	% Recovery	80	120	

List H VOCs Water

Compound	CAS Number	Units	LCL	UCL	RPD
1,1,1,2-Tetrachloroethane	630-20-6	ug/L	80	130	20
1,1,1-Trichloroethane	71-55-6	ug/L	80	134	20
1,1,2,2-Tetrachloroethane	79-34-5	ug/L	79	125	20
1,1,2-Trichloroethane	79-00-5	ug/L	80	125	20
1,1-Dichloroethane	75-34-3	ug/L	80	125	20
1,1-Dichloroethene	75-35-4	ug/L	80	132	20
1,1-Dichloropropene	563-58-6	ug/L	75	130	20
1,2,3-Trichlorobenzene	87-61-6	ug/L	55	140	20
1,2,3-Trichloropropane	96-18-4	ug/L	75	125	20
1,2,4-Trichlorobenzene	120-82-1	ug/L	65	135	20
1,2,4-Trimethylbenzene	95-63-6	ug/L	80	125	20
1,2-Dibromo-3-chloropropane	96-12-8	ug/L	50	130	20
1,2-Dibromoethane	106-93-4	ug/L	80	129	20
1,2-Dichlorobenzene	95-50-1	ug/L	80	125	20
1,2-Dichloroethane	107-06-2	ug/L	80	129	20
1,2-Dichloropropane	78-87-5	ug/L	80	120	20
1,3,5-Trimethylbenzene	108-67-8	ug/L	80	127	20
1,3-Dichlorobenzene	541-73-1	ug/L	80	120	20
1,3-Dichloropropane	142-28-9	ug/L	80	120	20
1,4-Dichlorobenzene	106-46-7	ug/L	80	120	20
2,2-Dichloropropane	594-20-7	ug/L	80	133	20
2-Butanone	78-93-3	ug/L	10	170	20
2-Chloroethyl vinyl ether	110-75-8	ug/L	45	160	20
2-Chlorotoluene	95-49-8	ug/L	80	127	20
2-Hexanone	591-78-6	ug/L	55	130	20
4-Chlorotoluene	106-43-4	ug/L	80	126	20
4-Methyl-2-pentanone	108-10-1	ug/L	64	140	20
Acetone	67-64-1	ug/L	40	180	20
Benzene	71-43-2	ug/L	80	121	20
Bromobenzene	108-86-1	ug/L	80	120	20
Bromochloromethane	74-97-5	ug/L	65	130	20
Bromodichloromethane	75-27-4	ug/L	80	131	20
Bromoform	75-25-2	ug/L	70	130	20
Bromomethane	74-83-9	ug/L	30	145	20
Carbon disulfide	75-15-0	ug/L	58	128	20
Carbon tetrachloride	56-23-5	ug/L	65	140	20
Chlorobenzene	108-90-7	ug/L	80	120	20
Chloroethane	75-00-3	ug/L	60	135	20
Chloroform	67-66-3	ug/L	80	125	20
Chloromethane	74-87-3	ug/L	40	125	20
cis-1,2-Dichloroethene	156-59-2	ug/L	70	125	20
cis-1,3-Dichloropropene	10061-01-5	ug/L	70	130	20
Chlorodibromomethane	124-48-1	ug/L	60	135	20
Dibromomethane	74-95-3	ug/L	75	125	20
Dichlorodifluoromethane	75-71-8	ug/L	40	160	20
Ethylbenzene	100-41-4	ug/L	80	122	20

List H VOCs Water

Compound	CAS Number	Units	LCL	UCL	RPD
Hexachlorobutadiene	87-68-3	ug/L	72	132	20
Isopropylbenzene	98-82-8	ug/L	80	122	20
m-,p-Xylene	179601-23-1	ug/L	80	122	20
Methylene chloride	75-09-2	ug/L	80	123	20
n-Butylbenzene	104-51-8	ug/L	80	131	20
n-Propylbenzene	103-65-1	ug/L	80	129	20
Naphthalene	91-20-3	ug/L	59	149	20
o-Xylene	95-47-6	ug/L	80	122	20
p-Isopropyltoluene	99-87-6	ug/L	80	122	20
sec-Butylbenzene	135-98-8	ug/L	80	127	20
Styrene	100-42-5	ug/L	80	123	20
tert-Butylbenzene	98-06-6	ug/L	80	126	20
Tetrachloroethene	127-18-4	ug/L	80	124	20
Toluene	108-88-3	ug/L	80	124	20
trans-1,2-Dichloroethene	156-60-5	ug/L	80	127	20
trans-1,3-Dichloropropene	10061-02-6	ug/L	80	130	20
Trichloroethene	79-01-6	ug/L	80	122	20
Trichlorofluoromethane	75-69-4	ug/L	62	151	20
Vinyl acetate	108-05-4	ug/L	10	190	20
Vinyl chloride	75-01-4	ug/L	50	170	20
Chlorobenzene-d5	3114-55-4		0	0	0
Fluorobenzene	462-06-6		0	0	0
1,4-Dichlorobenzene-d4	3855-82-1		0	0	0
4-Bromofluorobenzene	460-00-4	% Recovery	86	115	
Toluene-d8	2037-26-5	% Recovery	88	110	
1,2-Dichloroethane-d4	17060-07-0	% Recovery	80	120	
Dibromofluoromethane	1868-53-7	% Recovery	86	118	

List E PAHs Water

Compound	CAS Number	Units	LCL	UCL	RPD
Phenanthrene	85-01-8	ug/L	33	122	40
Indeno(1,2,3-cd)pyrene	193-39-5	ug/L	42	147	40
Naphthalene	91-20-3	ug/L	33	112	40
Acenaphthene	83-32-9	ug/L	22	116	40
1-Methylnaphthalene	90-12-0	ug/L	31	111	40
Acenaphthylene	208-96-8	ug/L	21	116	40
Anthracene	120-12-7	ug/L	34	121	40
Benzo(a)anthracene	56-55-3	ug/L	55	150	40
Benzo(a)pyrene	50-32-8	ug/L	55	150	40
Benzo(b)fluoranthene	205-99-2	ug/L	51	148	40
Benzo(g,h,i)perylene	191-24-2	ug/L	29	149	40
Benzo(k)fluoranthene	207-08-9	ug/L	42	157	40
Chrysene	218-01-9	ug/L	50	155	40
Pyrene	129-00-0	ug/L	55	138	40
Dibenzo(a,h)anthracene	53-70-3	ug/L	23	150	40
Fluoranthene	206-44-0	ug/L	51	141	40
Fluorene	86-73-7	ug/L	23	118	40
2-Methylnaphthalene	91-57-6	ug/L	32	115	40
Naphthalene-d8	1146-65-2		0	0	0
Chrysene-d12	1719-03-5		0	0	0
Acenaphthene-d10	15067-26-2		0	0	0
Phenanthrene-d10	1517-22-2		0	0	0
Perylene-d12	1520-96-3		0	0	0
Nitrobenzene-d5	4165-60-0	% Recovery	35	114	
2-Fluorobiphenyl	321-60-8	% Recovery	43	116	
p-Terphenyl-d14	1718-51-0	% Recovery	33	141	

List C PCBs Soil

Compound	CAS Number	Units	LCL	UCL	RPD
Aroclor-1254	11097-69-1	ug/kg	60	130	40
Aroclor-1260	11096-82-5	ug/kg	60	130	40
Aroclor-1016	12674-11-2	ug/kg	40	140	40
Aroclor-1242	53469-21-9	ug/kg	64	136	40
Aroclor-1248	12672-29-6	ug/kg	64	136	40
Aroclor-1221	11104-28-2	ug/kg	64	136	40
Aroclor-1232	11141-16-5	ug/kg	64	136	40
2,4,5,6-Tetrachloro-m-Xylene	877-09-8	% Recovery	29	133	
Decachlorobiphenyl	2051-24-3	% Recovery	30	173	

List A PAHs Solid

Compound	CAS Number	Units	LCL	UCL	RPD
Phenanthrene	85-01-8	ug/kg	35	92	40
Indeno(1,2,3-cd)pyrene	193-39-5	ug/kg	42	132	40
Naphthalene	91-20-3	ug/kg	37	87	40
Acenaphthene	83-32-9	ug/kg	30	87	40
1-Methylnaphthalene	90-12-0	ug/kg	35	89	40
Acenaphthylene	208-96-8	ug/kg	31	87	40
Anthracene	120-12-7	ug/kg	37	94	40
Benzo(a)anthracene	56-55-3	ug/kg	43	136	40
Benzo(a)pyrene	50-32-8	ug/kg	45	136	40
Benzo(b)fluoranthene	205-99-2	ug/kg	39	130	40
Benzo(g,h,i)perylene	191-24-2	ug/kg	42	132	40
Benzo(k)fluoranthene	207-08-9	ug/kg	40	133	40
Chrysene	218-01-9	ug/kg	43	131	40
Pyrene	129-00-0	ug/kg	47	118	40
Dibenzo(a,h)anthracene	53-70-3	ug/kg	40	135	40
Fluoranthene	206-44-0	ug/kg	41	123	40
Fluorene	86-73-7	ug/kg	31	86	40
2-Methylnaphthalene	91-57-6	ug/kg	35	92	40
Naphthalene-d8	1146-65-2		0	0	0
Chrysene-d12	1719-03-5		0	0	0
Acenaphthene-d10	15067-26-2		0	0	0
Phenanthrene-d10	1517-22-2		0	0	0
Perylene-d12	1520-96-3		0	0	0
Nitrobenzene-d5	4165-60-0	% Recovery	23	120	
2-Fluorobiphenyl	321-60-8	% Recovery	30	115	
p-Terphenyl-d14	1718-51-0	% Recovery	18	137	

APPENDIX C

Field Sampling Standard Operating Procedures

Document Control #: 07-DCN-00

STANDARD OPERATING PROCEDURE FOR SAMPLING EQUIPMENT AND CONTAINER DECONTAMINATION

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

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The following persons have read and understand this SOP:

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1. PURPOSE

By complying with procedures specified in this SOP, KEMRON will ensure the removal of contaminants of concern from sampling, drilling and other field equipment.

2. SCOPE

This procedure provides technical guidance on decontamination of sampling equipment and sample containers. The following two categories of cleaning are covered in this procedure:

- Equipment cleaning procedures
- Emergency field sample container cleaning

3. RESPONSIBILITIES

3.1 Project Manager Responsibilities

The KEMRON project manager ensures that any project specific deviations from this procedure will be provided to the Field Staff.

3.2 Field Staff Responsibilities

The KEMRON field staff follow procedures specified in this SOP unless written site-specific protocol is provided by the Project Manager.

4. DEFINITIONS / SPECIFICATIONS

4.1 Emergency Field Sample Container Cleaning Procedures

Emergency field sample container cleaning procedures are restricted procedures to be used when events occur outside the scope of the sampling plan.

4.2 Soap

Soap is a standard brand of phosphate-free laboratory detergent such as Liquinox®. Use of other detergent is justified and documented in the field logbooks and inspection or investigative reports.

4.3 Solvent

Solvent is pesticide-grade isopropanol. Other solvents may be substituted for a particular purpose if required. Pesticide-grade acetone or methanol are acceptable.

4.4 Tap water

Tap water may be used from any municipal water treatment system (spigot at the office). Use of an untreated potable water supply is not an acceptable substitute for tap water.

4.5 Deionized water

Deionized water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits).

4.6 Disinfectant soap

Disinfectant soap is a standard brand of disinfectant cleaner.

4.7 Nitric acid solution (10%)

Nitric acid solution (10%) is made from reagent-grade nitric acid and deionized water.

4.8 Containment

Containment includes securing all waste materials generated during site operations. Routine practice should include all efforts to keep used decontamination solutions at the site. This is especially the case for equipment used to collect samples of hazardous materials or toxic wastes or materials from hazardous waste sites or RCRA facilities.

5. HANDLING OF AND CONTAINERS FOR CLEANING SOLUTIONS

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. The following are acceptable materials used for containing the specified cleaning solutions:

5.1 Standard Use Solutions

5.1.1 Soap is kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.

5.1.2 Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose. All secondary containers are labeled as to the contents.

5.1.3 Deionized water is stored in clean containers that can be closed prior to use. It can be applied from plastic squeeze bottles. All secondary containers are labeled as to the contents.

5.2 Non-standard Use Solutions

5.2.1 Disinfectant soap should be kept in clean containers until use. It should be poured directly from the container.

5.2.2 Solvent is stored in the unopened original containers until used. They may be applied using the low pressure nitrogen system fitted with a Teflon® nozzle, or using Teflon® squeeze bottles. All secondary containers are labeled as to the contents.

5.2.3 Nitric acid should be kept in the glass container it is received in, and placed in squeeze bottles prior to application. All secondary containers are labeled as to the contents.

Note: Hand pump sprayers generally are not acceptable storage or application containers for the above materials (with the exception of tap water). This also applies to stainless steel sprayers. All hand sprayers have internal oil coated gaskets and black rubber seals that may contaminate the solutions.

6. EQUIPMENT CLEANING PROCEDURES

All equipment taken to the field is assumed to be clean. The following procedures are completed during a sampling event for multiple uses of the equipment, at the conclusion of equipment use at the site and if visual evidence indicates a need at the onset of equipment use.

6.1 Specifications for Decontamination Pads

The size of the project will dictate the method of containing decontamination fluids. Examples of containment include the use of drums, buckets, decon pads, etc. For additional information see Appendix C.

6.2 Sampling Equipment Cleaning Procedures

The following procedures are used for all sampling equipment (well casings, well screens, or split spoon samplers, etc.), unless otherwise specified in the project documents or unless it is observed that additional cleaning is necessary. For additional guidance on decontamination and cleaning of equipment, see Appendix A.

1) Clean with tap water and soap using a brush if necessary to remove particulate matter and surface films. Equipment may be steam cleaned (soap and high pressure hot water) as an alternative to brushing; however, PVC or plastic items should not be steam cleaned.
2) Rinse thoroughly with tap water.
3) Rinse thoroughly with deionized water.
4) If equipment is to be used during the next field sampling operations, remove from the decontamination area and cover with plastic. If equipment cleaning is a final project cleaning or will be used during a later sampling phase, wrap equipment in one layer of aluminum foil. Seal the foil wrapped equipment in plastic and label.
5) For information related to contaminant of decontamination solutions, see Appendix F.

After collecting samples that contain oil, grease, or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone, hexane or petroleum either to remove the materials before proceeding with the first step of cleaning; or in extreme cases, it may be necessary to steam clean the field equipment before the first step of cleaning; or discarding the equipment may be necessary. Special considerations need to be addressed with the Project Manager before deciding to use solvent rinses since waste handling and disposal are regulated activities.

6.3 Cleaning Procedures for Specialized Equipment

For specialized field equipment the manufacturer's manual should state the cleaning procedures KEMRON should use. For additional information see Appendix A.

6.4 Handling of Cleaned Equipment

After field cleaning, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the cleaning area to prevent recontamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in

aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

7. SUBCONTRACTOR EQUIPMENT CLEANING PROCEDURES

Heavy equipment rental and operation is usually subcontracted and the subcontractor is responsible for equipment decontamination. KEMRON personnel are responsible to direct any additional cleaning should there be a need. For additional information see Appendix D.

8. EQUIPMENT CLEANING PROCEDURES AT THE OFFICE

Additional cleaning and decontamination procedures maybe warranted when equipment or parts of equipment arrival back at the office (field or home). See Appendix B for examples of cleaning procedures and Appendix G for disposal of office cleaning solutions.

9. PROCEDURES FOR DISPOSABLE SAMPLE CONTAINER CLEANING AND USE: IN EMERGENCY FIELD SITUATIONS

The following container use and cleaning procedures are restricted for use in emergency situations only which maybe necessary if sampling occurs outside planned events; or in the case of an ER mobilization where additional containers may be needed in a pinch. In such emergencies, new one-pint or one-quart mason jars may be used to collect samples for analyses of organic compounds and metals in waste and soil samples. These containers would also be acceptable on an emergency basis for the collection of water samples for extractable organic compounds, pesticides, and metals analyses. These jars cannot be used for the collection of water samples for volatile organic compound analyses. The rubber sealing ring should not be in contact with the jar and aluminum foil should be used, if possible, between the jar and the sealing ring. If possible, the jar and aluminum foil should be rinsed with pesticide-grade isopropanol and allowed to air dry before use. Several empty bottles and lids maybe submitted to the laboratory as blanks for quality control purposes.

10. PREPARATION OF DISPOSABLE SAMPLE CONTAINERS

Routine sampling procedures call for precleaned containers supplied by the laboratory. However, if disruption of the supply of pre-cleaned, certified sample bottles occurs, sampling may have to continue regardless. In such situations, the laboratory is contacted immediately for the shipping of additional containers and approval of any field cleaning procedures of sample containers in the interim. For examples of cleaning disposable sample containers under such conditions, see Appendix E.

11. REFERENCE

1) Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, US EPA Region 4, November 2001

APPENDIX A: ADDITIONAL FIELD CLEANING PROCEDURES

Well Sounders, Tapes, or Interface Probes

The following cleaning procedures are used for well sounders or tapes:

1) Wash with soap and tap water.
2) Rinse with tap water.
3) Rinse with deionized water.

Pumps

Cleaning of pumps not specified in this section will be cleaned in a similar fashion.

1) Goulds® Pump Cleaning Procedure
   CAUTION - During cleaning always disconnect the pump from the generator. The Goulds© pump should be cleaned prior to use and between each monitoring well. The following cleaning procedure is used:
   a) Using a brush, scrub the exterior of the contaminated hose and pump with soap and tap water.
   b) Rinse the soap from the outside of the pump and hose with tap water.
   c) Rinse the tap water residue from the outside of pump and hose with deionized water.
   d) Place the pump and hose in a clean plastic bag.

2) Redi-Flo2® Pump
   CAUTION - Make sure the pump is not plugged in. The Redi-Flo2® pump should be cleaned prior to use and between each monitoring well. The following cleaning procedure is used:
   a) Using a brush, scrub the exterior of the pump, electrical cord and garden hose with soap and tap water. Do not wet the electrical plug.
   b) Rinse with tap water.
   c) Rinse with deionized water.
   d) Place the equipment in a clean plastic bag.

3) Redi-Flo2® Ball Check Valve
   The following procedure is required to clean the Redi-Flo2® ball check valve:
   a) Completely dismantle ball check valve. Check for wear and/or corrosion, and replace as needed.
   b) Using a brush, scrub all components with soap and tap water.
   c) Rinse with deionized water.
   d) Reassemble and re-attach the ball check valve to the Redi-Flo2® pump head.

Automatic Sampler Tubing

Whenever possible use new or dedicated tubing. In cases where new or dedicated tubing can not be used, Silastic® and Tygon® tubing previously used in the automatic samplers may be field cleaned as follows:

a) Flush tubing with tap water and soap.
b) Rinse tubing thoroughly with tap water.
c) Rinse tubing with deionized water.

Full-face, ½-face, and SCBA Face-masks

When there is a potential for exposure, face masks are used. This being the case, cleaning of face masks always follows use.

a) Wash face-mask thoroughly inside and out with hot tap water and disinfectant soap. Use only soft brushes. Do not use scouring pads of any type.
b) Rinse face-mask thoroughly inside and out with tap water.
c) Hang face-mask up until completely dry.
d) Place face-mask in plastic bag and return to SCBA case.

APRs are completely dismantled prior to cleaning. Then steps as outlined above for SCBA face masks are used. When completely dry, the APR is reassembled and placed in a plastic bag.

APPENDIX B: PROCEDURES FOR OFFICE CLEANING

See the web at the following web address for additional cleaning procedures (see reference section): http://www.epa.gov/region4/sesd/eisopqam/eisopqam.pdf

APPENDIX C: EXAMPLE OF CONSTRUCTION & USE OF A DECONTAMINATION PAD

1) The pad should be constructed in an area known or believed to be free of surface contamination.
2) The pad should not leak excessively.
3) If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
4) Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
5) Water should be removed from the decontamination pad frequently.
6) A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.
7) At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. No solvent rinsates will be placed in the pit.
8) Solvent rinsates should be collected in separate containers for proper disposal. If the decontamination pad has leaked excessively, soil sampling may be required.

APPENDIX D: SUBCONTRACTOR EQUIPMENT CLEANING PROCEDURES

This section is provided as a guide to KEMRON employees to ensure that the subcontractor equipment brought on-site and cleaned in the field has been cleaned to a level equivalent to that stated here. In addition, the procedures stated provide an example of text that should be included in the SOW the subcontractor is to follow where it is specified that such steps are to be completed either prior to arrival at the site or between each drilling event, as applicable.

These procedures are used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses, and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

Preliminary Equipment Cleaning (prior to site arrival)
The subcontractor should bring drilling and related equipment pre-cleaned and ready for use. The following outlines the equivalent procedures that should be used by the subcontractor:

1) All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (soap and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.
2) Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other hazardous waste sites before being brought on site.
3) Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can supply materials without the printing and/or writing if specified when ordered.

Drill Rig Field Cleaning Procedure for Multiple Boreholes
Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (soap and high pressure hot water) between boreholes.

Field Cleaning Procedure for Drilling Equipment
The following is a standard procedure for subcontractor field cleaning of augers, drill stems, rods, tools, and associated equipment.

1) Clean with tap water and soap, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with soap) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.
2) Rinse thoroughly with tap water.
3) Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

APPENDIX E: PREPARATION OF DISPOSABLE SAMPLE CONTAINERS

1. Introduction
No disposable sample container (with the exception of the glass and plastic compositing containers) may be reused. All disposable sample containers will be stored in their original packing containers. When packages of uncapped sample containers are opened, they will be placed in new plastic garbage bags and sealed to prevent contamination during storage. Specific pre-cleaning instructions for disposable sample containers are given in the following sections.

2. Plastic Containers used for "Classical" Parameters
Plastic containers used for oxygen demand, nutrients, classical inorganics, and sulfides have no precleaning requirement. However, only new containers may be used.

3. Glass Bottles for Semi-Volatile GC/MS Analytes
These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Laboratory approval is necessary prior to implementation of these procedures. If desired, pesticide-grade methylene chloride may be substituted for pesticide-grade isopropanol. In addition, 1:1 nitric acid may be substituted for the 10% nitric acid solution.

   a) Wash bottles and jars, Teflon® liners, and caps in hot tap water and soap.
   b) Rinse three times with tap water.
   c) Rinse with 10% nitric acid solution.
   d) Rinse three times with deionized water.
   e) Rinse bottles, jars, and liners (not caps) with solvent.
   f) Oven dry bottles, jars, and liners at 125EC. Allow to cool.
   g) Place liners in caps and close containers.
   h) Store in contaminant-free area.

4. Glass Bottles for Volatile GC/MS and TOX Analyses
These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Prior QA approval is necessary prior to implementation of these procedures.
   a) Wash vials, bottles and jars, Teflon liners and septa, and caps in hot tap water and laboratory detergent.
   b) Rinse all items with deionized water.
   c) Oven dry at 125oC and allow to cool.
   d) Seal vials, bottles, and jars with liners or septa as appropriate and cap.
   e) Store in a contaminant free area.

5. Plastic Bottles for ICP Analytes
These procedures are to be used only if the supply of pre-cleaned, certified sample bottles is disrupted. Prior QA approval is necessary prior to implementation of these procedures.
   a) Wash bottles and caps in hot tap water with soap.
   b) Rinse both with 10% nitric acid solution.
   c) Rinse three times with deionized water.
   d) Invert bottles and dry in contaminant free environment.
   e) Cap bottles.
   f) Store in contaminant free area.

APPENDIX F: DISPOSAL OF FIELD CLEANING SOLUTIONS

Disposal of Field Cleaning Solutions
Procedures for the safe handling and disposition of investigation derived waste (IDW), including used wash water, rinse water, and spent solvents are specified in the project plan. The following are provided as general guidelines:

1) Minimum Requirements for Non-hazardous Disposal of IDW
   a) Liquid and soil/sediment IDW is containerized and analyzed before disposal.
   b) The collection, handling, and proposed disposal method is specified in the approved project plan.

2) Minimum Requirements for Management of Hazardous IDW
   a) Spent solvents should remain in the field for proper disposal or recycling.
   b) All hazardous IDW is containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

DISPOSAL of IDW

Type	Hazardous	Non-Hazardous
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator, otherwise return to field office for proper disposal.	N/A
Decontamination Vater	Containerize in 55-gallon drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with site manager for testing and disposal.	Containerize in 55-gallon drum with tight fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with site manager for testing and disposal.

APPENDIX G: DISPOSAL OF OFFICE (FIELD OR HOME) CLEANING SOLUTIONS

Since equipment is cleaned in the field at the conclusion of field activities, any additional cleaning completed at the office will have differing waste disposal procedures for cleaning solutions than those in the field. Procedures for safe handling and disposition of such spent cleaning solutions, including washwater, rinse water, spent acid solutions, and spent solvents are as follows:

1) Washwater
   Since equipment is decontaminated before its return to the office, the washwater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is washed outside, it may wash onto the ground without recovery of the washwater.

2) Rinsewater
   Since equipment is decontaminated before its return to the office, the rinsewater may be disposed in the sanitary drain in the washroom. When large equipment (vehicles, augers, etc.) is rinsed outside, it may go onto the ground without recovery.

3) Nitric Acid
   Nitric acid cleaning solutions are to be diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom. If used outdoors, this material should be captured and diluted to a pH greater than 2.0, and flushed down the sanitary drain in the washroom.

4) Solvent
   All solvents used should be captured, properly labeled, and stored on the premises of the office until arrangements for proper disposal are made. Used solvents can be classified as either "solvent for recovery" or "solvent for disposal". Solvent for recovery is that which was used in the standard field cleaning or office cleaning of equipment. Solvent used for cleaning badly contaminated equipment (e.g., tar removal, etc.) should be designated for disposal. The two groups should be labeled "For Recovery" or "For Disposal" and stored separately at the office.

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STANDARD OPERATING PROCEDURE FOR SAMPLE PACKAGING AND SHIPPING

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The following persons have read and understand this SOP:

Signature	Date

1. PURPOSE

The purpose of this procedure is to ensure consistent sample packaging and shipping at KEMRON.

2. SCOPE

This procedure addresses the packaging of samples and proper shipping protocol.

3. RESPONSIBILITIES

3.1 Project Manager

To ensure the field staff follows the procedure as stated and/or to provide written deviations to the SOP.

3.2 Field staff

To follow SOP as stated and any written deviations provided by the Project Manager.

4. POLICY

Samples collected during field investigations or in response to a hazardous materials incident are classified prior to shipment, as either environmental or hazardous materials samples. See Attachment B for the KEMRON policy on shipping, entitled "Shipping Guidelines".

5. DEFINITIONS

5.1 Hazardous Materials

Materials which meet specific definitions under US DOT regulations which are subject to restrictive shipping requirements including employee training, packaging, labeling, and documentation. See Attachment A

5.2 Environmental Sample

A sample of solid waste, water, soil or air which is collected for the sole purpose of testing to determine characteristics or composition and are not subject to 40 CFR Part 262 through 268 (including use of hazardous waste manifests), if certain conditions of shipment are met.

(Refer to Appendix D of the Sample Shipping Procedures, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, EPA Region 4, November 2001, (http://www.epa.gov/region4/sesd/eisopqam/eisopqam.html), and "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), US-EPA, April 13, 1981.

5.3 Dangerous Goods

US DOT classified Hazardous Materials may be classified as Dangerous Goods when shipped by air and be subject to further regulatory requirements and/or shipping restrictions than that stated for Hazardous Materials.

According to the International Air Transportation Association (IATA), Dangerous goods are articles or substances which are capable of posing a risk to health, safety, property or the environment and which are shown in the list of dangerous goods in these Regulations or which are classified according to these Regulations.

6. SHIPMENT OF DANGEROUS GOODS (BY AIR)

The Project Manager is responsible for determining if samples collected during a specific field investigation meet the definitions for hazardous materials and/or dangerous goods. When shipping dangerous goods by air, all samples are packaged by a trained and certified employee according to Attachment B. Check with the air shipping carrier to ensure all carrier shipping requirements are met. For additional information, see the IATA Manual.

7. SHIPMENT OF ENVIRONMENTAL SAMPLES

Samples determined by the Project Manager to be environmental samples are shipped using the following protocol, developed jointly between US-EPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples" (3).

Untreated wastewater and sludge from POTW's are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

7.1 Shipping Environmental Samples by Commercial Carrier (Air or Ground)

Environmental samples should be packed prior to shipment using the following procedures and in compliance with all carrier requirements:

7.1.1 Allow a small amount of headspace in all bottles (except VOC containers with a septum seal) to compensate for any pressure and temperature changes.

7.1.2 Be sure the lids on all bottles are tight (will not leak). Lids maybe taped or sealed with custody seals as added protection or as required.

7.1.3 Place bottles in separate and appropriately sized polyethylene bags and seal the bags with tape.

7.1.4 Include one temperature blank per cooler.

7.1.5 Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape.

7.1.6 Line the cooler with a large heavy duty plastic bag.

7.1.7 Place an appropriate amount of bubble wrap in the bottom of the cooler and then place the bottles in the cooler with sufficient space to allow for the addition of bubble wrap between the bottles and cans.

7.1.8 Put "double bagged" ice (in heavy duty polyethylene bags and properly sealed) on top of and/or between the samples. Fill all remaining space between the bottles or cans with bubble wrap.

7.1.9 Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

7.1.10 Place the Chain-of-Custody Record into a plastic bag, and tape the bag to the inner side of the cooler lid.

7.1.11 Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut.

7.1.12 Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

7.1.13 Shipping containers must be marked "THIS END UP", and arrow labels which indicate the proper upward position of the container should be affixed to the container. A label containing the name and address of the shipper should be placed on the outside of the container. Labels used in the shipment of hazardous materials (e.g., Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of containers used to transport environmental samples.

7.2 **Hand Delivery of Environmental Samples (by employee or courier)**

7.2.1 Bottles (required for VOC containers with a septum seal) can be filled completely with sample.

7.2.2 Be sure the lids on all bottles are tight (will not leak).

7.2.3 Place bottles in a sturdy cooler with sufficient space to allow for the addition of bubble wrap between the bottles.

7.2.4 Include one temperature blank per cooler.

7.2.5 Put "double bagged" ice (in heavy duty polyethylene bags and properly sealed) on top of and/or between the samples.

7.2.6 Place COC in plastic bag and tape to the inside of the cooler lid, close lid, seal with custody seals and transfer the courier. Alternatively, when delivering by hand, close the cooler and hand deliver with the chain of custody.

8. ASBESTOS SHIPPING

See Figure 1

8.1 Place each asbestos sample in a small Ziploc bag.

8.2 Place the bags of asbestos samples in a large Ziploc bag, as needed.

8.3 Place in a sturdy box for shipping (i.e., FEDEX box)

8.4 Use bubble wrap to prevent the sample(s) from moving around in the box.

8.5 Insert COC in the box.

8.6 Seal box and place "Danger" stickers on the box.

8.7 Affix chain-of-custody seals to the top and sides of the box within any securing tape so that the box cannot be opened without breaking the seal.

9. AIR QUALITY SAMPLE SHIPPING

9.1 Place air samples in a sturdy box suitable for the type of sampling media (i.e., tenax tubes, tedlar bags, summa canisters)

9.2 Use bubble wrap to prevent the sample(s) from moving around in the box.

9.3 Insert COC in the box

9.4 Seal box

9.5 Affix chain-of-custody seals to the top and sides of the box within any securing tape so that the box cannot be opened without breaking the seal.

10. REFERENCES

10.1 Dangerous Goods Regulations, International Air Transport Authority (IATA). Current Edition.

10.2 EPA Order 1000.18, February 16, 1979.

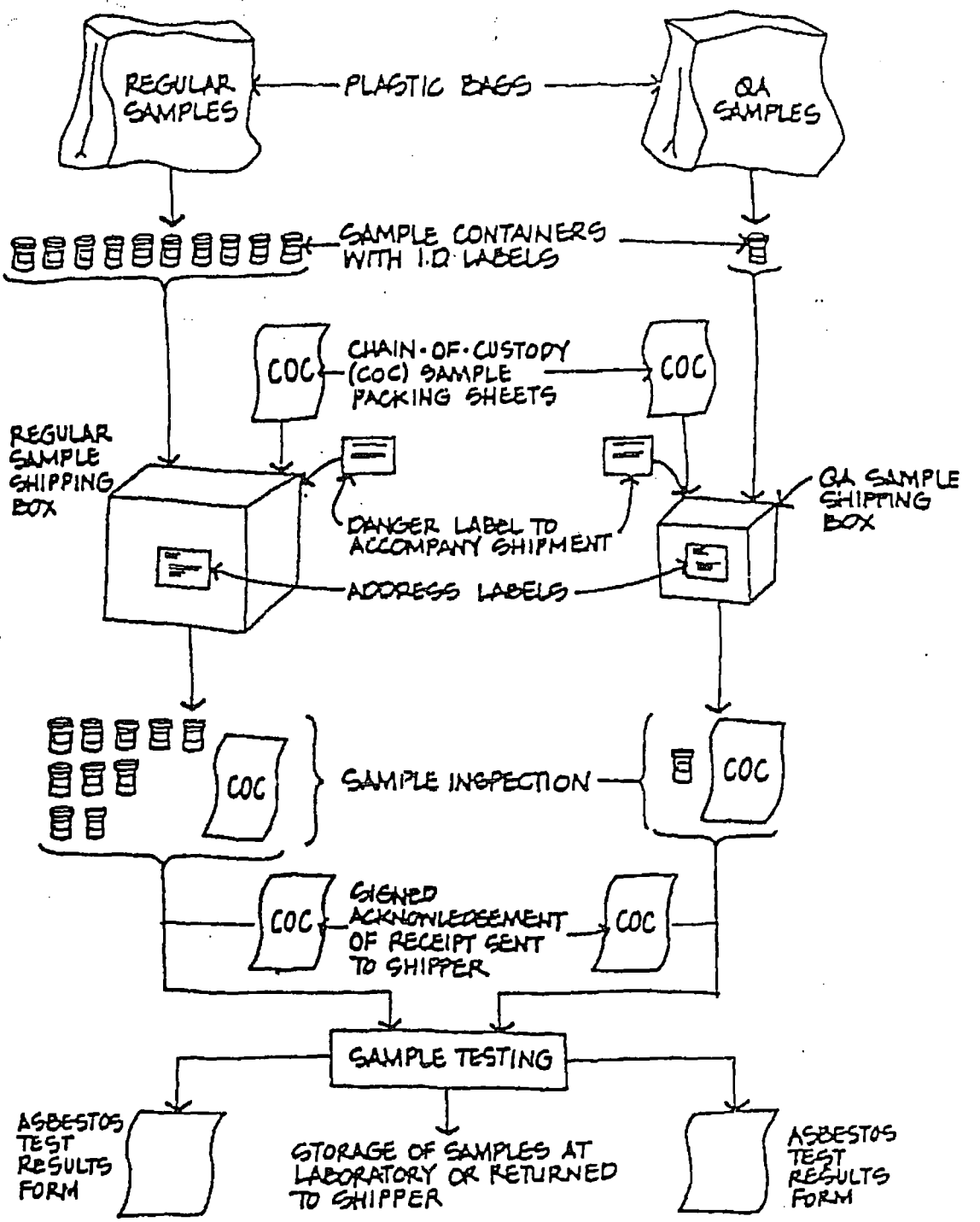
10.3 "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), US-EPA, April 13, 1981.

10.4 40 CFR 136.3. July 1, 2001. See Table 11, Footnote 3.

10.5 Environmental Investigation Standard Operating Procedures and Quality Assurance Manual, USEPA, Region 4, 11/2001

FIGURE 1 – ASBESTOS SAMPLE PACKAGING PROCEDURES

EXHIBIT K-2
SAMPLE HANDLING PROCEDURES



ATTACHMENT A – DEFINITION OF HAZARDOUS MATERIALS

HAZARDOUS MATERIAL DEFINITION

DOT Definition of Hazardous Material:

A hazardous material is defined as any substance or material could adversely affect the safety of the public, handlers or carriers during transportation. All DOT hazardous materials are listed in the DOT's Hazardous Material Table.

There are nine classes of hazardous materials:

Hazard Class 1: Explosives	1.1 mass explosion hazard 1.2 projectile hazard 1.3 minor blast/projectile/fire 1.4 minor blast 1.5 insensitive explosives 1.6 very insensitive explosives
Hazard Class 2: Compressed Gases	2.1 flammable gases 2.2 non flammable compressed 2.3 poisonous
Hazard Class 3: Flammable Liquids	Flammable (flash point below 141°) Combustible (flash point 141°-200°)
Hazard Class 4: Flammable Solids	4.1 flammable solids 4.2 spontaneously combustible 4.3 dangerous when wet
Hazard Class 5: Oxidizers and Organic Peroxides	5.1 Oxidizer 5.2 Organic Peroxide
Hazard Class 6: Toxic Materials	6.1 Material that is poisonous 6.2 Infectious Agents
Hazard Class 7: Radioactive Material	Radioactive I Radioactive II Radioactive III
Hazard Class 8: Corrosive Material	Destruction of the human skin Corrode steel at a rate of 0.25 inches per year
Hazard Class 9: Miscellaneous	A material that presents a hazard during shipment but does not meet the definition of the other classes

ATTACHMENT B – SHIPPING GUIDELINES

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ENVIRONMENTAL SERVICES

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www.kemron.com

MEMO

TO: Lisa Larson, Heather Moorehead, Okey Tucker, Jeannette Hamm, Doug Barnett, Victoria Anderson

FROM: Marilyn Zumbro MZ

CC: Matt Davey, Chris Amick, Tom O'Hara, Marsha Robinson, Pat Bolger, John Dwyer, Kelly Clemons

DATE: May 13, 2004

RE: SHIPPING GUIDELINES

The purpose of this memo is to provide information and additional instruction regarding procedures for the classification and shipment of materials by KEMRON personnel. During the course of normal project activities, KEMRON personnel may be required to ship materials including samples, wastes, chemicals, supplies, calibration solutions/gases, etc. Certain materials which meet specific definitions under US Department of Transportation (DOT) regulations are considered "hazardous materials" and are subject to restrictive shipping requirements including employee training, packaging, labeling, and documentation. Such materials, when shipped by air, may be classified as "dangerous goods' and be subject to further regulatory requirements and/or shipping restrictions.

The following guidelines will be used by KEMRON personnel in determining if materials being offered for shipment are classified as hazardous under DOT regulations. All shipments of hazardous materials by KEMRON must conform to DOT requirements and be prepared and offered for shipment ONLY by personnel who have documented training in accordance with regulatory requirements. ALL shipments of hazardous materials must be approved/authorized by Program/Regional Management.

The following materials are considered hazardous by DOT and must be shipped in accordance with DOT HMT requirements:

- Materials designated as hazardous by listing in the Hazardous Materials Table (49 CFR 172.101);
- Hazardous wastes requiring manifesting;
- Hazardous substances listed in Appendix A to 49 CFR Part 172.101, and shipped in a quantity in one package in excess of the listed RQ;
- Mixtures or solutions of hazardous substances exceeding concentration limits corresponding to RQs as listed in table in 49 CFR Part 171.8;
- Substances or samples of substances meeting the defining criteria of any DOT hazard class or division;
- Other specifically listed DOT regulated hazardous materials such as marine pollutants and elevated temperature materials.

It is important to remember that a hazardous waste, for purposes of DOT, is a material which is subject to the Hazardous Waste Manifest Requirements under USEPA regulations contained in 40 CFR Part 262. Per 40 CFR Part 261.4, samples of solid waste, water, soil, or air which are collected for the sole purpose of testing to determine characteristics or composition, are not subject to 40 CFR Part 262 through 268 (including use of hazardous waste manifests), if certain conditions of shipment are met. Many samples shipped by KEMRON will meet the RCRA criteria for exemption from

provisions of 40 CFR Parts 262 through 268, including hazardous waste manifesting, and will therefore, not meet the DOT definition of a hazardous waste.

This does not mean that all environmental samples are exempt from DOT requirements. Your shipment must pass all the definitional tests in order be excluded by definition. Ask the following questions about the shipment.

- Is it designated in the HM table (49 CFR 172.101)?
- Is it a hazardous waste requiring manifesting?
- Is it a hazardous substance (listed in Appendix A to 49 CFR Part 172.101) in a quantity in one package which exceeds the RQ?
- If it is a hazardous substance mixture or solution, is it in a concentration by weight that exceeds the corresponding RQ of the material as listed in the table found in 49 CFR Part 171.8 *Hazardous Substance* (3)(ii)?
- Does it meet the defining criteria of any DOT hazard class or division?
- Is it specifically listed as a marine pollutant?
- Does it meet the definition of an elevated temperature material?

If the answer to any of these questions is yes, the shipment is regulated by DOT as a hazardous material and subject to specific shipping requirements. Consult with your management to assure that properly trained personnel prepare and offer the shipment and that the shipment meets all regulatory requirements.

ATTACHMENT C – CHAIN OF CUSTODY

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

KEMRON
ENVIRONMENTAL SERVICES

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project #:	Project Name:
Sampler (print):	Signature:

Program
- ☐ NPDES
- ☐ AFCEE
- ☐ RCRA
- ☐ USAGE
- ☐ Other _____

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF CONTAINERS	Hold																ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp in °C	Remarks:

*Homogenize all composite samples prior to analysis

Page _____ of _____

Document Control #: 07-FQC-00

STANDARD OPERATING PROCEDURE FOR FIELD QUALITY CONTROL

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

KEMRON Environmental Services

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Approved by:

Marilyn K. Zumbro

11/8/04

Marilyn Zumbro, Regional Manager — Date

11/8/04

John Dwyer, Vice President — Date

Kelly McClemons

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. PURPOSE

This procedure is followed in conjunction with both project specific guidance to determine which quality control samples are necessary and field procedures for technical collection procedures. Following this procedure will ensure that the quality control samples collected will be collected in a consistent manner.

2. SCOPE

This SOP provides basic procedures for collecting various types of quality control samples.

3. RESPONSIBILITIES

3.1 Project Manager Responsibilities

The KEMRON Project Manager ensures that any project specific deviations from this procedure will be provided to the Field Staff.

3.2 Field Staff Responsibilities

The KEMRON field staff follow procedures specified in this SOP unless written site-specific protocol is provided by the Project Manager.

4. DEFINITIONS

4.1 Quality Control Samples

Quality control (QC) samples are collected during field studies for various purposes which include the isolation of site effects (control samples), define background conditions (background sample), evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples).

4.2 Control Sample

Control samples are typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

4.3 Background Sample

A background sample is a sample (usually a grab sample) collected from an area, water body, or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

4.4 Split Sample

A split sample is a sample which has been portioned into two or more containers from a single sample container or sample mixing container. The primary purpose of a split sample is to measure sample handling variability. A split sample will also measure inter- or intra-laboratory precision.

4.5 Duplicate Sample

Duplicate samples are two or more samples collected and representative of a given population. The purpose of a duplicate sample is to estimate the variability of a given characteristic or contaminant associated with a population.

4.6 Trip Blanks

A trip blank is prepared at the time of the sample kit preparation (by lab or field office) and remains unopened until analysis. Trip blanks indicate if volatile contamination in the environment of container / sample handling is affecting the associated volatile samples.

4.7 Spikes (also known as proficiency test (pt) samples)

Spikes are a sample with known concentrations of contaminants. Spike samples are often packaged for shipment with other samples and sent for analysis. At no time after their preparation are the sample containers opened before they reach the laboratory. Spiked samples are used to measure bias due to sample handling or analytical procedures.

4.8 Equipment Field Blanks

An equipment field blank is used to determine if contaminants have been introduced by contact of the sample medium with sampling equipment. Equipment field blanks are often associated with collecting rinse blanks of equipment that has been decontaminated.

4.9 Temperature Blanks

A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 4°C (ice). The temperature of the blanks are measured at the time of sample receipt by the laboratory.

4.10 Preservative Blanks

A preservation blank is a sample that is prepared in the field and used to determine if the preservative used during field operations was contaminated, thereby causing a positive bias in the contaminant concentration.

4.11 Field Blanks

A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample).

4.12 Material Blanks

A material blank is a sample of sampling materials (e.g., material used to collect wipe samples, etc.), construction materials (e.g., well construction materials), or reagents (e.g., deionized water generated in the field, water from local water supplies used to mix well grout, etc.) collected to measure any positive bias from sample handling variability. Commonly collected material blanks are listed below:

4.12.1 Wipe Sample Blanks

Wipe sample blanks are a sample of the material used for collecting wipe samples.

4.12.2 Grout Blanks

Grout blanks are a sample of the material used to make seals around the annular space in monitoring wells.

4.12.3 Filter Pack Blanks

Filter pack blanks are a sample of the material used to create an interface around the screened interval of a monitoring well.

4.12.4 Construction Water Blanks

Construction water blanks are a sample of the water used to mix or hydrate construction materials such as monitoring well grout.

4.12.5 Deionized Water Blanks

A deionized water blank is a sample collected from a field deionized water generating system. The purpose of the deionized water blank is to measure positive bias from sample handling variability due to possible localized contamination of the deionized water generating system or contamination introduced to the sample containers during storage at the site.

5. PREPARATION OF FIELD QUALITY CONTROL (QC) SAMPLES

The following procedures are basic procedures, should be used in conjunction with the field SOP and may not apply to a particular project. Project documents will specify what types of QC are necessary.

5.1 Control Sample

5.1.1 Collect the control sample in the same way a study sample is collected, i.e., composite, grab, etc.

5.1.2 Handle the control sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.2 Background Sample

5.2.1 Collect the background sample in the same way a study sample is collected, i.e., composite, grab, etc. and in the area prescribed by the project documents.

5.2.2 Handle the background sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.3 Split Sample

5.3.1 Collect a study sample which is approximately double in size than what is normally used.

5.3.2 Homogenize the sample.

5.3.3 Split the sample into two portions.

5.3.4 Aliquot each portion in the appropriate sample containers with the appropriate preservation.

5.3.5 Ship one portion of the split sample with the batch of samples to the laboratory prescribed in the project documents and ship the second portion to a second laboratory as prescribed in the project documents.

Note: Split samples are not collected for volatiles.

5.4 Duplicate Sample

5.4.1 Collect a second study sample from the same location / area / bucket in the same manner as that of the study sample.

5.4.2 Handle the control sample in the same way a study sample is collected, i.e., container, labeling, preservation.

5.5 Trip Blank

Trip blanks are prepared at the time of sample kit preparation which may occur at the laboratory or the field office.

5.5.1 Fill a VOA vial full with deionized water, cap and ensure that there are no air bubbles.

5.5.2 Transport the unopened trip blank with the sample kit to the field.

5.5.3 Transport the unopened trip blank with the samples to the laboratory for analysis

Note: The trip blank is analyzed for volatile organic compounds.

5.6 Spikes

Spikes are generally prepared at the laboratory prior to analysis; however, there are occasions where the project may require field prepared spike samples.

5.6.1 Purchase or prepare a spike or proficiency test sample as stated in the project documents. Prepare a spike sample by fortifying a background sample with the target analytes of interest. Prepare an unfortified background sample also.

5.6.2 Submit the spike sample, and background sample as necessary, to the laboratory for analysis as a "blind" sample.

5.6.3 Evaluation occurs upon receipt of the sample results.

5.7 Equipment Blanks

5.7.1 Bring extra deionized water to the field.

5.7.2 Pour deionized water over the sampling equipment following equipment cleaning and collect in an appropriate sample container.

5.7.3 Handle the sample container in the same fashion as an aqueous sample is handled, i.e. container, preservation, etc.

5.7.4 Collect enough equipment blanks to analyze for all the target analytes of interest or as stated in the project documents.

5.7.5 Transport the sample to the laboratory for analysis with the samples.

5.8 Temperature Blanks

5.8.1 Fill a small container with water. (Sample kits should already contain a temperature blank).

5.8.2 Transport the temperature blank with the samples to the laboratory.

5.8.3 Provide a temperature blank with each cooler or sample transport container.

5.9 Preservative Blanks

5.9.1 Prepare a preservative blank at the appropriate stage and in the frequency as stated in the project documents.
5.9.2 Bring extra deionized water to the field.
5.9.3 Fill the same type bottle used for sample collection with deionized water
5.9.4 Add the appropriate type and amount of chemical preservative.
5.9.5 Transport the sample to the laboratory with the samples.

5.10 Field Blanks

5.10.1 Bring extra deionized water to the field.
5.10.2 Pour the water into the appropriate sample containers at pre-designated locations at the site, according to project documents.
5.10.3 Collect in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.
5.10.4 Add the appropriate chemical preservative according to the analysis.
5.10.5 Transport the sample to the laboratory with the samples.

5.11 Wipe Sample Blanks

5.11.1 Bring extra wipe samples to the site.
5.11.2 Handle, package and transport unused wipe material as that for wipes used for sampling.

5.12 Grout Blanks

5.12.1 Take a sample of the grout used to seal the monitoring well.
5.12.2 Place in a suitable container and transport to the laboratory.

5.13 Filter Pack Blanks

5.13.1 Take a sample of the filter pack used in assembling of the monitoring well.
5.13.2 Place in a suitable container and transport to the laboratory.

5.14 Construction Water Blanks

5.14.1 Take a sample of the water used in preparing construction materials.
5.14.2 Place in a suitable container and transport to the laboratory.

5.15 Deionized Water Blanks

5.15.1 Take a sample of the water generated from the field deionized water source at the end of sampling activities or in intervals as stated in the project documents.
5.15.2 Take the sample in clean environment so external conditions are not affecting the sample.
5.15.3 Place in a suitable container and transport to the laboratory.

6. REFERENCES

Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, EPA Region IV, November 2001

Document Control #: 07-DOC-00

STANDARD WORK INSTRUCTION FOR FIELD DOCUMENTATION

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

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11/8/04

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Kelly McClemens

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. POLICY

All field activities are documented. The extent of the documentation is such that all activities can be recreated either through real time note taking or through reference to other documents and/or logbooks. All logbooks have the KEMRON home office address and phone number, toll free if available written on the inside of the front cover.

2. PURPOSE

This procedure establishes the field documentation at KEMRON.

3. SCOPE

The technical documentation addressed in this procedure applies to field activities that KEMRON directly participates in or where project over-ste is provided by KEMRON.

4. DEFINITIONS AND ACRONYMS

Not Applicable

5. RESPONSIBILITIES

5.1 Project Manager

To ensure that the Field documentation procedures are followed.

5.2 Field Staff

At a minimum, to complete field documentation as stated in this SOP.

6. Field Documentation

The two types of field documentation routinely completed at KEMRON include bound notebooks and field data sheets. Specific types of field data sheets may be required by the client which are used in place of any generic field sheets used by KEMRON.

6.1 Bound Field Logbooks

Bound field logbooks are assigned to the job or job tasks for larger/multiphase work. The front of each logbook will state "KEMRON", book number, project name, project number, and start/end dates for the logbook. The spine of the logbook will contain the following minimally: project number and start and end dates. Field notes recorded in bound notebooks will be recorded with a ball point pen, on all- weather bound environmental field notebooks with consecutively numbered pages. See section 7 for information to be recorded. Upon completion of the data entry and entry review (see section 6.3) the logbooks are immediately placed in a central project location for retrival by other KEMRON personnel.

6.2 Field Data Sheets

When field data sheets are used a reference to the frequency of site visits is established by either of the following: (1) client/work agreement (i.e., take data every month); or (2) the data sheets are referenced in a bound logbook. When referenced in a bound notebook, the date, time, number of pages and description of the content of the field data sheets is included. All recorded notes are made with a ball point pen. If multiple field data sheets are used for any given site visit, the pages are numbered using the following format: "page 1 of 3, page 2 of 3, page 3 of 3" in a conspicuous location on each data sheet.

6.3 QA/QC of Field Documentation

6.3.1 Making Corrections

If an entry is in need of a correction, the erroneous information is crossed out with a single strike mark and initialed and dated, where the correction is recorded as near to the error as possible.

6.3.2 Field Staff

The field staff who collected the data signs and dates the daily entry in the bound notebook or field data sheet(s). This is completed the day the data is taken.

6.3.3 Independent Reviewer

Each set of collected data is reviewed and signed by an independent reviewer. The review is completed as soon as possible following data collection. At a minimum, documentation and validity of the following items should be verified (will be dependent on type of field activity): Correct study area designation and sample numbers; date and time (24-hour system recordings); and ensure that any unused portion of the data sheet and/or bound logbook is lined out, initialed and dated.

7. Types of Field Documentation and Procedures

7.1 Documentation for Sampling Events

7.1.1 General Entries: Logbooks and Field Data Sheets

a. Page heading to include location, date, project/client (for easy reference when copying);
b. Time onsite and offsite;
c. Parties involved (list all individuals and companies);
d. Vehicle (type, function, etc.);
e. Weather and site conditions (to include temperature, humidity, barameter,wind dir/speed, rainfall);
f. Health and Safety topics discussed;
g. Detailed descriptions of sample locations that identify the area of concern (make references to maps, GPS location, redlines, manmade objects, terrain, site history, etc.);
h. Sampling equipment used, including reference to the calibration/maintenance logbook;
i. Any equipment decontamination and maintenance completed in the field and reference to who completed the decontamination and field maintenance if other than the field staff and when it was completed;
j. All collected field measurements (PID /OVA, pH, temperature, etc.);
k. Sample number, samplers signature, fractions sampled, date sampled, time (military format) sampled and preservation method for each sample;
l. Identification and types of QC samples collected;
m. Photographic information as specified in section 7.3;
n. Reference to and summary of information on any field data sheets;

o. Fed-ex number, if applicable;
p. Cross through any remaining empty portions of page(s) at conclusion of entries with initials in crossed area;
q. Number pages and sign;

7.1.2 Specific Entries

See the specific technical SOPs for specific items to be recorded for the specific application. Project specific plans may include documentation specifications as well.

7.1.3 Survey Documentation

For surveying well heads, the following are examples of what can be recorded:

a. The benchmark can be a true known elevation or an assumed elevation of 100.0 labeled as TBM (Temporary Benchmark).
b. The Back Sight (BS) is the rod reading taken from the known benchmark or the arbitrary benchmark established as 100.0'.
c. The Back Sight rod reading is simply added to the benchmark elevation to establish the Height of Instrument.
d. The Fore Sight (FS) rod readings are those rod readings taken from unknown elevation points.
e. Establish the elevations of previously unknown points by subtracting the FS rod readings from the HI.
f. A Turning Point (TP) is a newly surveyed point that becomes a new benchmark once the instrument is moved.
g. The above example shows MW-3 becoming a Turning Point (TP 1) as the instrument needed moved to get a line of site on the final two points.
h. Once the instrument is moved a new HI is established by simply taking a rod reading (BS) on your TP.

The above information can be recorded in columns in the logbook with the following column headers: Station ID, BS (+) Rod Reading, HI (height of Instrument), FS (-) Rod Reading, and Elevation.

STATION ID	BS (+) Rod Reading	HI (Height of Instrument)	FS (-) Rod Reading	ELEVATION
Benchmark (BM)	4.5	673.15		668.65
MW-1			4.70	668.45
MW-2			5.25	667.90
MW-3			3.50	669.65
Turning Point (TP 1)	6.5	676.15		669.65
MW-4			5.65	670.50
MW-5			5.50	670.65

7.1.4 Chain of Custody

Cross-reference the Chain of Custody information in the field logbook. The Chain of Custody is completed when samples are taken. Whenever possible, a secondary review is completed. Additional details are recorded in the applicable SOPs or project documents.

7.1.5 Sample Labels

Cross-reference the sample labels information in the field logbook. The sample labels are completed when samples are taken. Sample labels contain information such as sample ID, sampler's signature or first initial and last name, date and time (military format) of sampling, preservative, fraction, etc. Whenever possible, a secondary review is completed.

7.2 Equipment Maintenance & Calibration Logbooks

Each piece of equipment has an assigned logbook which remains at the central facility (regional office or site office). The equipment serial number cross-references the equipment with the logbook. All maintenance and calibration information is included in the logbook(s). Any maintenance and/or calibration that is completed in the field is recorded first in the field log and then transcribed to the equipment maintenance & calibration logbook upon returning to the office / site office.

7.3 Photographic Records

Photographs may be digital or film photographs. Digital photographs will be maintained in digital files and/or a photo record book in the project file. Film photographs will be maintained in the project file. The number and subject of all photographs (digital and film) will be recorded in the field logbook(s) and, if necessary, cross-referenced to the photographic logbook, which maybe used for larger projects. The entries identify the photographs with the following information:

1. The project#, date, time, and location of the photograph, including direction facing;
2. The name of the photographer, and;
3. The signature of the photographer.

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STANDARD OPERATING PROCEDURE FOR UTILITY CLEARANCE

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Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date

John Dwyer, Executive Vice President — 10/11/2010 Date

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1. POLICY

KEMRON personnel will follow procedures established in the "SOP for Subsurface Utility Clearance" for all intrusive work, which is work that breaks the ground surface, such as drilling, probing, over-drilling, excavations and trenching. No intrusive work occurs without KEMRON present. Any deviations from this procedure require Regional Management approval.

2. PURPOSE

Subsurface utilities, product lines, and other unseen obstacles pose both potential safety and asset damage issues if encountered unexpectedly while performing drilling or excavating activities. This SOP standardizes subsurface utility identification at KEMRON in an effort to minimize the possibility of striking such obstacles.

3. SCOPE

This procedure provides technical guidance on subsurface utility identification.

4. RESPONSIBILITIES

4.1 Project Manager Responsibilities

The KEMRON Project Manager ensures that subsurface utility clearance is completed as stated in this SOP.

4.2 Field Technician Responsibilities

The KEMRON field technician(s) follow the applicable steps as stated in this procedure, in the site specific Health and Safety Plan, and as directed by the Project Manager.

4.3 Site Specific Health and Safety Officer (SSHO) Responsibilities

To review the completed Marked Utilities Verification Form.

4.4 Subcontractors

All subcontractors are secured under contract and required to meet all local, state, and federal requirements (i.e., 29 CFR 1910.333(c)(3); 29 CFR 1926.550(a)(15)(i), (ii), (iii)).

5. PLANNING

Planning will address all steps outlined in this procedure. Whether a private utility locator and/or public underground utility locating service will be used is determined during the planning stages.

5.1 Site-Specific Written Instructions for Utility Identification

The site specific health and safety plan will reference this SOP. Additionally, the forms used for this SOP (Attachments A and B), or this SOP in its entirety, will be attached to the site specific health and safety plan.

5.2 Written Instructions for Deviations

Any deviations from this SOP will be prepared and justified by the Project Manager and approved by the Regional Manager.

6. PRE-MOBILIZATION UTILITY IDENTIFICATION

6.1 Proposed Intrusive Work Locations

6.1.1 Request and conduct interview/site walk with the property owner representative (if available). Document as stated on the Utility Locator Form (Attachment A).

6.1.2 Identify and mark either with paint (white paint only) and/or with stakes the proposed intrusive work locations.

6.1.3 Check for the presence of over head utilities.

6.2 Pre-mobilization Documentation

6.2.1 Ensure the Utility Locator Form (Attachment A) is completed prior to mobilization. Advanced notice is necessary for the utilities to complete the utility location process prior to the start of field work.

6.2.2 Obtain written consent of off-site property owners (e.g., through an access agreement or right-of-entry agreement) prior to performing any work on non-client property.

6.2.3 Request client maps, plans or photos showing the locations of underground tanks, lines, utilities, pipelines, etc.

6.3 Utility Locator Process

6.3.1 Complete the following initial information on the Utility Locator Form (Attachment A):

- 6.3.1.1 Site Address
- 6.3.1.2 City / County/township
- 6.3.1.3 Locations of Work Onsite
- 6.3.1.4 Extent of work: Front / Side / Rear
- 6.3.1.5 Distance from Road / ROW
- 6.3.1.6 Nearest Street Intersection
- 6.3.1.7 Distance and Direction to Intersection or list streets if work is to occur between streets
- 6.3.1.8 Project Name
- 6.3.1.9 Project Number
- 6.3.1.10 Client

6.3.1.11 Contractor / Subcontractor
6.3.1.12 Dates of Work
6.3.1.13 Times of Work
6.3.1.14 Type(s) of Work (i.e., excavation, blasting, railroad ROW, Highway, vertical drilling, horizontal drilling)

6.3.2 Contact the local utility ONE-CALL Service (i.e. Miss Utility, DIG-TESS, J.U.L.I.E., OUPS, etc.), as applicable, and record the following information on the Utility Locator Form.

6.3.2.1 Name of the KEMRON employee calling the ONE-CALL
6.3.2.1.1 date and time of call,
6.3.2.1.2 ticket number,
6.3.2.1.3 dates the ticket is valid

6.3.2.2 Record the following specific information concerning utility owners to be contacted by the ONE-CALL service provider:
6.3.2.2.1 Utility type
6.3.2.2.2 Utility Company Name
6.3.2.2.3 Utility Company Contact Information
6.3.2.2.4 Method used by utility company to confirm completion of utility identification (i.e., fax, return call by KEMRON, etc.)

6.3.2.3 Ask for and record those utility owners that will not be contacted by the ONE-CALL service

6.3.3 Complete the following for each Utility owner that will not be contacted by the ONE CALL service

6.3.3.1 Contact each of the other utilities

6.3.3.2 Record the following on the Utility Locator Form:
6.3.3.2.1 Utility type
6.3.3.2.2 Utility Company Name
6.3.3.2.3 Utility Contact Information
6.3.3.2.4 Method used by utility company to confirm completion of utility identification (i.e., fax, return call by KEMRON, etc.)
6.3.3.2.5 Name of the KEMRON employee calling the utility company
6.3.3.2.6 Date and time of the call
6.3.3.2.7 Dates utility marking or clearance is valid
6.3.3.2.8 Record when confirmation is received

6.3.4 When confirmation of utility location is received from utility owners, record the method of confirmation and the date of receipt.

6.3.5 Review utility drawings (as-builts or plans), if available, and complete appropriate portion of the Utility Locator Form (Attachment A).

6.3.6 Under certain circumstances, the only available information will be gathered from a site walk with the property owner/representative (i.e. federal property) and/or review of utility drawings (as-builts or plans). If this is the case, the site walk completed in section 6.1.1 is documented on the appropriate portion of the Utility Locator Form (Attachment A). Information from review of any site utility drawings is recorded on the utility locator form. The site walk and/or map review should provide all of the available utility information.

6.4 Optional Utility Location Methods
Public utility locating services are usually only required to locate up to the service connection. When intrusive work is planned beyond the boundary of the public utility locating service (i.e. inside buildings, behind locked fences, etc.), use of a private utility locating service is recommended. Advanced locating technologies (i.e. ground penetrating radar) may be necessary when locating beneath steel reinforced concrete. KEMRON may opt to meet the locater on site. If this option is utilized, document the date and time contact was made, who was contacted, locating technology, and include other information as necessary.

7. MARKED UTILITIES VERIFICATION FORM

Verify that subsurface utilities have been cleared and/or marked prior to initiating intrusive site work by completing the Marked Utilities Verification Form (MUVF) (Attachment B). Subsurface utilities should be marked using standard color codes. If other color codes are used, the type of utilities marked should be verified with the locator service. The standard color codes are:

- White — Work Location
- Red — Electric
- Orange — Telephone/Cable
- Yellow — Gas/Oil
- Blue — Water
- Green — Sewer

Record the following on the MUVF.

7.1 Ticket number
7.2 Project name
7.3 Project number
7.4 Site location
7.5 Name of KEMRON employee completing the Marked Utility Verification Form
7.6 Date the form was prepared.

7.7 Verify and document that utilities are cleared/marked by recording the following on the Marked Utility Verification Form.

7.7.1 A "Y" or "N" indicating whether the utility is marked. (Note: if a utility is supposed to be marked and is not marked, call the ONE-CALL service or utility company as appropriate)

7.7.2 How the utility is marked (i.e., paint, color, stake, flag, etc.)

7.7.3 Any additional comments

8. TAILGATE SAFETY MEETINGS

Prior to any intrusive work, conduct a tailgate safety meeting where the following are completed:

8.1 The Tailgate Safety Form is completed as stated in the Corporate Health & Safety Plan and/or in the Site Specific Health and Safety Plan.

8.2 Utility identification and verification of identification will be an agenda item for the Tailgate Safety Meeting.

8.3 Document the meeting agenda and items discussed on the Tailgate Safety Meeting Form.

9. FIELD PROCEDURES

9.1 Visual check

Conduct a walk through of the area where intrusive work is to be performed and visually identify all above ground indications of subsurface utilities such as electrical/light poles, manholes, clean-outs, junction boxes, etc. Compare locations and line of sight potential underground connections with available drawings and utility markings. If site conditions indicate potential unmarked or mismarked subsurface utilities, contact the Project Manager to discuss field procedures. Also, review overhead utilities in relation to the work locations. Estimating the distance between the overhead power lines and the workers or work equipment should be made and the Project Manager is notified if the distance is less than 15 feet. (see Appendix 1) Drillers may have more stringent requirements regarding the proximity of the drill rig mast to the overhead lines and shielding requirements.

9.2 Intrusive Work Location vs. Marked Utilities

Actual intrusive work locations must be at least five feet away from all marked utility lines, unless authorized by the Project Manager or addressed in the work scope.

9.3 Surface Covering: Concrete or Asphalt

Use a concrete saw followed by a jackhammer to saw and break through an asphalt or concrete surface cover. The drill bit on the rig may also be used to auger through an asphalt cover, but cannot penetrate a concrete cover. **DO NOT** advance beyond the bottom of an asphalt or concrete cover with powered equipment until manual clearance is performed.

9.4 Manual or Vacuum Clearance for Drilling Activities

Prior to mechanical drilling, excavate the borehole location using a post-hole digger, hand auger, hand shovel, or other manual excavation method. Alternatively, vacuum excavation (potholing) of the location may be performed using soil vacuum equipment provided by some drillers. If vacuum excavation is used, verify volatile sampling protocol, if applicable. Excavate the borehole location to a diameter of at least two inches greater than the diameter of the drill bit on the lead auger, drill stem, or casing that is to be used. Advance the manual or vacuum borehole clearance to a minimum depth of 4 feet. If it is not possible to meet the diameter requirements using a single manual excavation boring, advance three or more hand auger borings to the required depth around the proposed location in such a manner that any subsurface utilities passing through the proposed boring location will be encountered while performing manual clearance.

If pea gravel, atypical fill material, or refusal is encountered, call the Project Manager, abandon and relocate. Pea gravel or granular fill material indicates that underground utilities or tanks may be located in the vicinity. If refusal is encountered, relocate to complete work or identify the source of refusal and continue as applicable (i.e, a rock). If relocating or if the source of refusal was a utility, call the PM; contact the utility, if identified; and, as applicable, periodically check location of initial attempt for observations indicating a puncture (i.e., presence of liquid). Prior to selecting a new location, review any subsurface utility drawings and location of overhead utilities. Proceed with the manual or vacuum clearance procedure at the revised location.

9.5 Target Soil within 4 feet of Surface

Use a hand auger to collect an undisturbed soil sample, if soil sample collection is required within the manual or vacuum clearance range (4 feet). Obtain PM approval for use of a direct-push drilling rig (i.e. Geoprobe) for sampling soil within 4 ft of surface.

9.6 Intrusive Work Location vs. Overhead Power Lines

All work (ranges of equipment use and personnel) occurs greater than 15 feet from the overhead power lines. If work is to be completed between 10-15 feet from the overhead power lines, verification that lines are not more than 50kV is required. See Appendix 1.

9.7 Limiting Access

If the intrusive work is not completed immediately after confirmed clearance, complete one of the following:

a) Surround the clearance area with barricades or cones and/or cover with a sheet of material, such as plywood, sufficient in strength to support the weight of a person or vehicle as necessary; or
b) Temporarily backfill the clearance location flush to ground surface with clean inert material.

10. REFERENCES

29 CFR 1910.333(c)(3); 29 CFR 1926.550(a)(15)(i), (ii), (iii)

ATTACHMENT A: UTILITY LOCATOR FORM

Site Address: _____

City/County/Township: _____

Locations of Work Onsite: _____

Extent of Work – Front/Sides/Rear: _____

Distance From Road/ROW: _____

Nearest Street Intersection: _____

Distance & Direction to Intersection: _____

(Alternately) Between Streets: _____

Project Name:

Project #:

Client:

Contractor/Sub:

Dates of Work:

Times of Work: _____

Type of Work:

_____ **Blasting** _____ **Excavation**

_____ **Railroad ROW** _____ **Vertical Drilling**

_____ **Highway** _____ **Horizontal Drilling**

ONE CALL - Utilities Contacted by One Call Service:

KEMRON Employee: _____

ONE CALL Operator: _____

Date and Time of Call: _____

Does ONE CALL locate in this area? ☐Yes ☐No

(if yes) Ticket #: _____

Dates Ticket Valid: _____

Utility	Company Name	Contact Information	Confirmation Mechanism (fax, etc..)	Confirmation Received? (y/n)

Utility	Company Name	Contact Information	Confirmation Mechanism (fax, etc..)	Confirmation Received? (y/n)

UTILITIES NOT CONTACTED BY ONE CALL SERVICE (must be contacted directly by KEMRON):

Utility	Company Name	Contact Information	Confirmation Received (y/n)?	KEMRON Employee	Date/Time of Contact	Dates Utility Marking or Clearance Valid

UTILITY DRAWINGS

1) Date utilities drawings requested _____
2) Requested by (KEMRON employee) _____
3) Utilities drawings requested from (client name, etc.) _____
4) Are utility drawings available for subject area? _____
5) List of drawings received (attach copies) _____
6) Drawings reviewed by (KEMRON employee) _____

INTERVIEWS / UTILITIES SITE WALK

1) Date utilities site walk requested

2) Utilities site walk requested from (client name, etc.)

3) Date and Time of utilities site walk

4) Utilities site walk attendees:

Name	Title

5) Utilities located

ATTACHMENT B: MARKED UTILITIES VERIFICATION FORM

Ticket #:		**Project Name:**	
Project #:		**Location of Site:**	
Prepared By:		**Date Prepared:**	

Instructions: A KEMRON employee completes this checklist while on site and before any intrusive work begins to ensure that all underground utility lines, other underground structures, as well as aboveground power lines are clearly marked out in the area selected for intrusive work. Utilities identified on the Utility Locator Form (ULF) for the site are required to be verified on-site and documented below. Any utilities identified on the ULF that are not visibly marked, or are not documented as "not present", require a call to the One-Call Company or utility company.

Emergencies: Follow the incident procedures as stated in the corporate Health and Safety Plan.

Verification:

Utility / Structure	Marked?		How Marked? (flags, paint, wooden stakes, etc.)	Comments
	YES	NO		
Natural Gas				
Electric Power				
Telephone Cable				
Sewer				
Storm Drain				
Water				
Steam				
Petroleum Product Lines				
Product Tank				
Septic Tank / Drain Field				
Overhead Power Line				

**** all marked "no" require a call to the One- Call company or to the utility company.***

APPROVAL OF UTILITY CLEARANCE:

SSHO/Field Representative:		Date:	

Color Codes: Red = Electric, Orange = Phone/Cable TV, Yellow = Gas, Blue = Water, Green = Sewer, White = Proposed Excavation

APPENDIX 1: OVERHEAD UTILITY REQUIREMENTS

Summary of the 2 Federal Regulations below

If working in the vicinity of overhead power lines, precautions are taken so that the equipment/personnel and power lines do not come into contact by implementing one of the following:

- de-energize and visibly ground electrical distribution and transmission lines at the point of work
- use insulated barriers that are not a part of the equipment to prevent contact with the lines
- if the power lines are not de-energized, operate equipment in the area ONLY if a safe minimum clearance is maintained as follows:
  - **At least 10 feet for lines rated 50 kilovolts (kV) or below**
  - At least 10 feet plus 0.4 inch for each kV above 50 kV; or maintain twice the length of the line insulator (but never less than 10-feet)

29 CFR 1910.333(c)(3)

(c) Working on or near exposed energized parts--(1) Application. This paragraph applies to work performed on exposed live parts (involving either direct contact or contact by means of tools or materials) or near enough to them for employees to be exposed to any hazard they present.

(2) Work on energized equipment. Only qualified persons may work on electric circuit parts or equipment that have not been deenergized under the procedures of paragraph (b) of this section. Such persons shall be capable of working safely on energized circuits and shall be familiar with the proper use of special precautionary techniques, personal protective equipment, insulating and shielding materials, and insulated tools.

(3) Overhead lines. If work is to be performed near overhead lines, the lines shall be deenergized and grounded, or other protective measures shall be provided before work is started. If the lines are to be deenergized, arrangements shall be made with the person or organization that operates or controls the electric circuits involved to deenergize and ground them. If protective measures, such as guarding, isolating, or insulating are provided, these precautions shall prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

Note: The work practices used by qualified persons installing insulating devices on overhead power transmission or distribution lines are covered by Sec. 1910.269 of this Part, not by Sec. Sec. 1910.332 through 1910.335 of this Part. Under paragraph (c)(2) of this section, unqualified persons are prohibited from performing this type of work.

(i) Unqualified persons. (A) When an unqualified person is working in an elevated position near overhead lines, the location shall be such that the person and the longest conductive object he or she may contact cannot come closer to any unguarded, energized overhead line than the following distances:

(1) For voltages to ground 50kV or below--10 ft. (305 cm);

(2) For voltages to ground over 50kV--10 ft. (305 cm) plus 4 in. (10 cm) for every 10kV over 50kV.

(B) When an unqualified person is working on the ground in the vicinity of overhead lines, the person may not bring any conductive object closer to unguarded, energized overhead lines than the distances given in paragraph (c)(3)(i)(A) of this section.

Note: For voltages normally encountered with overhead power lines, objects which do not have an insulating rating for the voltage involved are considered to be conductive.

29 CFR 1926.550(a)(15)(i), (ii), (iii)

(15) Except where electrical distribution and transmission lines have been deenergized and visibly grounded at point of work or where insulating barriers, not a part of or an attachment to the equipment or machinery, have been erected to prevent physical contact with the lines, equipment or machines shall be operated proximate to power lines only in accordance with the following:

(i) For lines rated 50 kV. or below, minimum clearance between the lines and any part of the crane or load shall be 10 feet;

(ii) For lines rated over 50 kV., minimum clearance between the lines and any part of the crane or load shall be 10 feet plus 0.4 inch for each 1 kV. over 50 kV., or twice the length of the line insulator, but never less than 10 feet;

(iii) In transit with no load and boom lowered, the equipment clearance shall be a minimum of 4 feet for voltages less than 50 kV., and 10 feet for voltages over 50 kV., up to and including 345 kV., and 16 feet for voltages up to and including 750 kV.

(iv) A person shall be designated to observe clearance of the equipment and give timely warning for all operations where it is difficult for the operator to maintain the desired clearance by visual means;

(v) Cage-type boom guards, insulating links, or proximity warning devices may be used on cranes, but the use of such devices shall not alter the requirements of any other regulation of this part even if such device is required by law or regulation;

(vi) Any overhead wire shall be considered to be an energized line unless and until the person owning such line or the electrical utility authorities indicate that it is not an energized line and it has been visibly grounded;

(vii) Prior to work near transmitter towers where an electrical charge can be induced in the equipment or materials being handled, the transmitter shall be de-energized or tests shall be made to determine if electrical charge is induced on the crane. The following precautions shall be taken when necessary to dissipate induced voltages:

(a) The equipment shall be provided with an electrical ground directly to the upper rotating structure supporting the boom; and

(b) Ground jumper cables shall be attached to materials being handled by boom equipment when electrical charge is induced while working near energized transmitters. Crews shall be provided with nonconductive poles having large alligator clips or other similar protection to attach the ground cable to the load.

(c) Combustible and flammable materials shall be removed from the immediate area prior to operations.

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STANDARD OPERATING PROCEDURE FOR SURFACE WATER SAMPLING

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Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date

John Dwyer, Executive Vice President — 10/11/2010 Date

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1. PURPOSE

The objectives of surface water sampling are to ensure valid and representative samples are collected that meet the requirements of the project, and to prevent deterioration and contamination of the samples before laboratory analysis. By complying with procedures specified in this Standard Operating Procedure (SOP), KEMRON will ensure that the following are successfully completed:

- Proper sampling technique.
- Prevent contamination or cross-contamination of surface water samples during collection, containerization, transit and storage.
- Obtain and record accurate and complete sampling data.
- Prepare complete and correct sample labels.
- Maintain sample chain-of-custody.

2. SCOPE

This procedure provides technical guidance on surface water sampling includingdecontamination procedures, types, and uses of sampling equipment. This SOP applies to KEMRON personnel and subcontractors collecting surface water samples for KEMRON projects. Client, federal, or state requirements, or project-specific requirements may dictate specific types of equipment or procedures to be used when applying this SOP to a particular project. Deviations from this SOP to accommodate site-specific requirements shall be documented in project planning documents and approved by the KEMRON Project Manager and Project Quality Assurance (QA) Manager, if applicable, prior to the performance of work.

3. RESPONSIBILITIES

Project Manager Responsibilities
The KEMRON Project Manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan. The KEMRON Project Manager ensures all field staff have reviewed and signed off on this SOP prior to beginning field work. A sign-off sheet covering all SOPs related to the project will be prepared and filed with project documents.

Field Staff Responsibilities
The KEMRON field staff prepares, organizes, and inspects all equipment required for the sampling event prior to the initiation of sampling activities. The field sampler and shipper are responsible for initiating, maintaining, and transferring chain-of-custody. Upon completion of the sampling event, the field staff returns all equipment to the designated storage area ensuring that the equipment is decontaminated and in a condition ready for reuse. KEMRON field staff are required to follow procedures specified in this SOP, or approved project-specific SOP, prior

to initiating the sampling event. Field staff are required to sign off that they have read and understand this SOP prior to beginning field work.

4. DEFINITIONS

Global Positioning System (GPS): A navigational system involving satellites and computers that can determine the latitude and longitude of a receiver on Earth by computing the time difference for signals from different satellites to reach the receiver. A GPS can be a hand-held device used to provide location information.

Sample Compositing; Discrete samples are taken and then proportionally combined into one sample container. Composite samples are generally used to estimate the average concentration of the individual samples that make up the composite.

Sampling: The act of collecting a portion of material for analytical purposes that accurately represents the material being sampled with respect to stated objectives.

Surface Water Samplers: Equipment designed to capture the portion of water necessary to meet project goals. Sampling of surface water can range from surface water to very deep water and the equipment needed is selected accordingly. Examples of surface water samplers include the following: bottle, Kemmerer, Van Dorn, and Niskin-Flow Bottle samplers.

5. PLANNING

The Project Manager provides written instructions of any project-specific information for the sampling crew. The written instructions can be as simple as a sampling requirements report or as detailed as a Sampling and Analysis Plan (SAP). Items in written sampling instructions may include the following:

- Vertical and horizontal sampling locations,
- Location of effluent discharges,
- Sampling points relative to the mixing zone,
- Method of sampling,
- Analytical parameters,
- Sample containers to be used for each analysis,
- Preservative for each analysis, and/or
- Frequency and type of QA/Quality Control (QC) samples.

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of QC samples such as field duplicate, equipment and/or field blank, and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the QAPP.

Information regarding the sample containers, preservation techniques and holding times for surface water samples will be described in the project specific SAP/QAPP.

Surface water sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project health and safety officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

A sufficient number of QC samples, appropriately distributed in time and space, should be collected to ensure that data quality objectives and requirements are met.

6. SAMPLE COLLECTION EQUIPMENT AND PROCEDURES

The tables in this section provide a description of equipment and sampling procedures for different surface water sampling methods. The project work plan will specify the method and any site-specific requirements. Once the surface water is sampled, the sample will be aliquoted in the following sequence or as stated in the project-specific planning documents: Field Parameters, Volatile Organic Compounds (VOCs), Semi-volatile Organic Compounds (SVOCs), Pesticides, Herbicides, PolyChlorinated Biphenyls (PCBs), Metals, and Wet Chemistry parameters.

Surface water sampling objectives may vary significantly between projects. Project objectives should be defined within the project-specific work plans. The list of required materials below identifies the types of equipment which may be used for a range of surface water sampling applications. The various types of sampling equipment which may be used include:

Field Instruments

- Individual or multi-parameter meter(s) to measure temperature, pH, specific conductance, dissolved oxygen (DO), oxidation reduction potential (ORP), and/or turbidity
- Flow meter

Sampling Equipment

- Sample bottles
- Extension rod with stainless steel clamp
- Kemmerer sampler
- Van Dorn or Niskin-flow sampler

Sample Preparation Equipment

- Filtration equipment
- Intermediate containers
- Sample kit (i.e., bottles, labels, preservatives, custody records, cooler)

General Equipment

- Project-specific sampling plans (SAP, QAPP, HASP) including map of wells to be sampled
- Sample collection records
- Field notebook/pen
- Waterproof marker pens
- Deionized water dispenser
- Sample cup
- Buckets
- Coolers or sample shuttles
- Instrument calibration solutions
- Power source (generator or battery)
- Equipment decontamination supplies
- Health and safety supplies (life jackets, PPE, etc)
- First-Aid kit
- Tool box
- Boat

Expendable Materials

- Deionized water supply
- 0.45 micron filters
- Paper towels
- Plastic sheeting
- Ice/blue ice for sample preservation
- Disposable latex powder-free glove liners
- Disposable nitrile gloves
- Plastic trash bags
- Ziplock® bags

This equipment list was developed to aid in field organization and should be used in preparation for each sampling event. Depending on the site-specific sampling plan, additional material and equipment may be necessary and should be determined before the scheduled sampling event. Similarly, not all of the items shown in this list may be necessary for any one sampling event.

TABLE 1: SURFACE WATER SAMPLING WITH BOTTLE SAMPLER

Item	Description
Sampling Equipment:	• Bottle or Container Sampler • Bottle Sampler with extension rod containing stainless steel clamp
Locating Samples:	Using a measuring gauge, if necessary, locate the horizontal distance from the bank to the sampling point as stated in the work plan, or use a GPS, or the watershed codes.
Getting to Sampling Point:	Get to the sampling point via any of the following: • From the bank, • From a structure (bridge, pier, etc.), • By wading into the current (avoid disturbing the sediment), or • From a motor-propelled boat traveling / sampling upstream whenever possible and, when necessary, idle into the current to maintain location.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Consider buoys to mark all sampling locations. Consider specific site factors, such as extent and nature of contaminant, flow, nearby outfalls, when selecting sample location. If required, locations may be adjusted based on site access, property boundaries, and surface obstructions. ***Sampling:*** 1. Immerse an inverted sampler to the vertical point of sampling and then re-righting (or unscrew cap of) the sampler into the direction of any current. 2. After the sampler is full, turn upright (or cap) and remove from water. 3. After aliquoting, if more volume is needed at the sampling point, collect as stated above and continue aliquoting. 4. Record sample observations in the field log book. ***Aliquot Sample:*** 1. For field measurements, transfer sample to a suitable container. 2. For laboratory analysis, transfer into the appropriate sample container(s) 3. Complete sample container label -include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. 4. Place in cooler and cool to 4° Celsius (C) with wet ice. ***Post-Sampling Activities:*** Several activities need to be completed and documented once surface water sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to Laboratory • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 2: SURFACE WATER SAMPLING WITH KEMMERER SAMPLER

Locating the Sampling Point:	Using a measuring gauge, if necessary, locate the horizontal distance from the bank to the sampling point as stated in the work plan, or use a GPS, or the watershed codes.
Getting to Sampling Point:	Get to the sampling point via any of the following: • From the bank, • From a structure (bridge, pier, etc.), • By wading into the current (avoid disturbing the sediment), or • From a motor-propelled boat traveling / sampling upstream whenever possible and, when necessary, idle into the current to maintain location.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Consider if buoys are necessary to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, flow, nearby outfalls, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. ***Sampling:*** 1. Obtain the sampler and check the knot at the bottom of the sampler for tightness and size. 2. To make a cast, cock the sampler by pulling the trip head into the trip plate. This is done by holding the top and bottom stoppers and giving a short, hard pull to the bottom stopper. 3. Tie the free end of the line to a fixed structure. 4. Lower the sampler to the desired depth. 5. When the sampler is at the desired depth, attach a messenger to the line. Hold the messenger over the deck until it is securely attached to the line. 6. Release the messenger to close the sampler. 7. Retrieve the sampler and aliquot sample collected. 8. Record the temperature of the water. 9. Record sample observations in the field log book ***Aliquot Sample:*** 1. For field measurements, transfer sample to a suitable container. 2. For laboratory analysis, transfer into the appropriate sample container 3. Complete sample container label - include initials, date and time of sample collection, sample ID, preservative and analysis required. 4. Place sample(s) in cooler and cool to 4° Celsius (C) with wet ice. ***Post-Sampling Activities:*** Several activities need to be completed and documented once surface water sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to Laboratory • Compiling all field data for site records.

	• Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 3: SURFACE WATER SAMPLING W/ VAN DORN OR NISKIN-FLOW SAMPLERS

Locating Samples:	Using a measuring gauge, if necessary, locate the horizontal distance from the bank to the sampling point as stated in the work plan, or use a GPS, or the watershed codes.
Getting to Sampling Point:	Get to the sampling point via any of the following: • From the bank, • From a structure (bridge, pier, etc.), • By wading into the current (avoid disturbing the sediment), or • From a motor-propelled boat traveling / sampling upstream whenever possible and, when necessary, idle into the current to maintain location.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP 6. Consider buoys to mark sampling locations. Consider specific site factors, including extent and nature of contaminant, flow, nearby outfalls, when selecting sample location. If required, locations may be adjusted based on site access, property boundaries, and surface obstructions. ***Sampling:*** 1. Measure the surface water depth to sediment. 2. Attach the sampler to the line (a winch line may be preferred). 3. Lower the sampler to the water. 4. Attach top sampler to the line at the appropriate point to meet required sampling depths. 5. Lower the samplers until the lower bottle is at the project specified depth. 6. Release the messenger down the wire to close the bottles. 7. Retrieve the bottles (using a winch may be preferred) and remove them from the line. 8. Record the temperature of the water. 9. Aliquot samples. 10. Record description of sampling in the field log book ***Aliquot Sample:*** 1. For field measurements, transfer sample to a suitable container. 2. For laboratory analysis, transfer into the appropriate sample container 3. Complete sample container label - include initials, date and time of sample collection, sample ID, preservative and analysis required. 4. Place sample(s) in cooler and cool to 4° Celsius (C) with wet ice. ***Post-Sampling Activities:*** Several activities need to be completed and documented once surface water sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to Laboratory • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

7. FIELD MEASUREMENTS

The following measurements will be taken unless otherwise specified in the project documents:

- Temperature
- pH
- Conductivity
- Turbidity
- Flow* at sampling point

*Flow is taken at 2-3 surface water depths at the sampling location, if required.

8. FIELD RECORDS

8.1 Field Logbook

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific surface water sampling documentation include the following:

- Site name;
- Date and Time of Arrival;
- Surface water depth;
- Weather conditions, including ambient air temperature and/or miscellaneous observations;
- Vertical and horizontal sampling location;
- Date and time of sampling;
- Equipment ID;
- Flow rate recorded (if applicable);
- Sample sequence number;
- Sampling strategy (bank, structure, wading, motor boat);
- Method of sampling (bottle, Kemmerer/Van Dorn Sampler, Niskin-Flow Bottle);
- Field parameters;
- Sample characteristics (color, turbidity, odor, etc.);
- Pertinent field observations and comments such as location of effluent discharges, mixing zones;
- Fractions sampled and preservation method; and
- Signature of sampler(s) and date, etc.

8.2 Chain-of-Custody

Completed chain-of-custody forms are maintained for all samples. A sample chain-of-custody form is included as Attachment A. The original form is sent to the laboratory with the samples and signed by the lab upon receipt. Original completed chain-of-custody forms are returned to KEMRON with the analytical report.

9. DECONTAMINATION

All equipment taken to the field will be clean. The following procedures, or that specified in the site-specific documents, are completed during a sampling event for multiple uses of equipment, at

the conclusion of equipment use at the site, and if visual evidence indicates a need at the onset of equipment use.

1. Hand wash, or flush/pump through automatic samplers / equipment, with a non-phosphate laboratory detergent dissolved in tap water;
2. Rinse with tap water;
3. Rinse thoroughly with deionized water;
4. Allow to air dry.
5. Wrap in aluminum foil and a plastic bag, if needed, and store in a clean, dry area.

Decontamination solvent such as isopropanol, acetone or hexane may be used for equipment exposed to high levels of organics (e.g., NAPL). Use of specific decontamination solvents should be addressed in project-specific planning documents and the waste solvents managed and disposed in accordance with applicable regulations.

10. SHIPPING

Equipment decontamination chemicals may need to be shipped to the site and samples may need to be shipped to the laboratory. All shipping is completed in accordance with the current federal and international regulations (DOT, EPA, IATA). Shipping stipulations are to be included in the project documents, but should you have any questions regarding shipping of any materials to and from the site, contact the authorized shipping representative in your home office.

11. ASSOCIATED SOPs

Field Log Book 07-DOC-00

12. REFERENCES

http://www.epa.gov/region09/qa/fieldsamp.html#surface

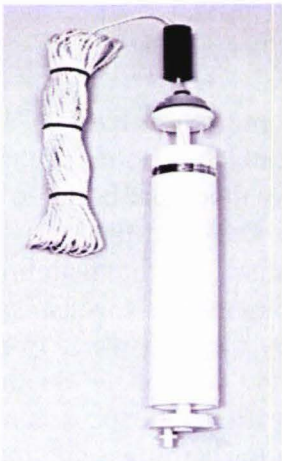
U.S. Geological Survey, 2006. Techniques of Water-Resources Investigations Book 9 Handbooks for Water-Resources Investigations National Field Manual for the Collection of Water-Quality Data Chapter A4. Collection of Water Samples *Revised 2006*

APPENDIX A: EXAMPLES OF SURFACE WATER SAMPLING EQUIPMENT

Bottle Sampler

A bottle that is used for surface water sampling where the bottle is lowered to the desired sampling depth, the lid is removed, the bottle is filled and recapped.

Kemmerer Sampler



Kemmerer samplers are used to collect surface water samples. Typically, Kemmerer samplers are used in shallow water where samples are to be collected at only one or two depths. The sampler is essentially a tube with stoppers on each end that can be held open when the sampler is lowered by a line to a desired depth. These same stoppers can then be triggered to close the ends of the tube when a metal cylinder called a messenger is dropped down the line holding the Kemmerer sampler. After the stoppers close the ends of the tube, the sampler is retrieved with the desired sample of water being uncontaminated by other water. Since only one sample of water can be obtained at a time, the process of obtaining several samples at various depths at the same sampling station is slow.

Van Dorn Sampler

Van Dorn samplers are used to collect surface water samples. Typically, Van Dorn samplers are used to collect samples from various depths at the same time. The Van Dorn bottles also allow greater depths to be explored than with the Kemmerer sampler. The Van Dorn sampler consists of an open ended clear plastic cylinder that can be attached to the hydrographic wire (the steel wire wound on the winch) and lowered to any desired depth. A deckhand operates the winch. The bottles also provide a platform to which thermometers can be attached to record the temperature of the water at the location of each Van Dorn bottle.

When it is time to lower the Van Dorn bottles into the water (this is called making a "cast"), a decision is made about the depth to which to send the bottles. This decision is based upon the depth of the water at the station and the number of samples needed. Normally only two water samples (surface and bottom) are taken. If a third water sample is required, the Kemmerer bottle may be used to obtain the surface sample. One Van Dorn bottle could then be used to obtain a mid-depth sample. In shallow areas, typically only take one water sample is taken.

Each end of the cylinder is fitted with a rubber cover. The Van Dorn bottle is attached to the line with the covers pulled out and twisted back and around to the side. The bottle is lowered to a pre-selected depth and left there until the thermometers attached inside come to thermal equilibrium with the water at that depth.

Two Van Dorn bottles can be attached to the same line and lowered to two different pre-selected depths. A metal weight called a "messenger" is

attached below the upper bottle. The water sample is taken by dropping a "messenger" down the wire. When the weight hits the catch on the upper Van Dorn bottle, the catch releases the rubber end covers. The two ends snap around and seal off the ends. The other "messenger" then travels down the winch line to the lower Van Dorn bottle causing it to be sealed.

When both bottles have been tripped, they are retrieved and returned to their storage rack. Water samples from each bottle can then be taken for analysis and the temperature read from the attached thermometers.

Niskin-Flow Bottle

The Niskin bottle is a plastic cylinder with stoppers at each end, connected by an elastic cord. The stoppers are held open by plastic cords attached to a release mechanism. Two clamps on the side of the cylinder are used to attach the bottle to a hydrographic line (usually a 3/16-inch steel cable with a 10-lb weight at the end) so that it can be lowered to a discrete depth in the water. When a small weight encircling the hydrographic line is released down the line, it strikes the release mechanism resulting in the two stoppers being pulled into the ends of the cylinder, thereby trapping water from that depth. The release mechanism also has the facility for holding a second messenger on the hydrographic line below the Niskin bottle such that when the mechanism is triggered, the second messenger is released down the line below the Niskin to trigger another Niskin further down the line. In this way several bottles can be triggered sequentially in one operation.

APPENDIX B: SPECIAL STIPULATIONS FOR SAMPLING

Special Stipulations for Sampling Running Water (rivers, streams, creeks)

- Collect grab samples at midstream and mid-depth where lateral mixing is complete, whenever possible.
- Do not collect samples from the surface (air-water interface).
- Take care not to disturb sediments prior to or during sampling while wading.
- When using a boat or wading, approach the station from downstream. Sample collection should be made upstream of the sampler.
- Collect samples in an order from downstream to upstream.
- Sample in areas of the stream where good vertical and horizontal mixing occurs (good current velocity and turbulence).
- Collect samples upstream of culverts (culverts tend to trap materials and debris moving downstream).
- At small bridges, if necessary, move upstream when sampling to avoid garbage and debris which may exist around the bridge.
- When sampling downstream of an effluent discharge, be aware of the location of the mixing zone and where samples are to be taken relative to the mixing zone.
- Near the confluence of two streams, collect samples at a sufficient distance downstream to ensure adequate mixing and at a sufficient distance upstream to avoid backwater from the other stream.
- When sampling from a motor-propelled boat, collect samples from the bow or upwind and/or upstream from the motor. Take care that no oil or gasoline leakage enters the water being sampled.
- Do not sample immediately after rainfall.

Special Stipulations for Sampling Confined Water (lakes, ponds, impoundments)

- Do not collect samples from the surface (air-water interface).
- Take care not to disturb sediments prior to or during sampling while wading.
- When sampling from a motor-propelled boat, collect samples from the bow or upwind and/or upstream from the motor. Take care that no oil or gasoline leakage enters the water being sampled.
- Composite samples should be collected, unless homogeneous mixing can be demonstrated.
- Do not sample immediately after rainfall.

KEMRON ENVIRONMENTAL SERVICES

ATTACHMENT A – EXAMPLE CHAIN OF CUSTODY FORM

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

Phone: 740-373-4071
Fax: 740-373-4835

KEMRON ENVIRONMENTAL SERVICES

CHAIN-OF-CUSTODY RECORD

Company Name:

Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project #:	Project Name:
Sampler (print):	Signature:

Program
- ☐ NPDES
- ☐ AFCEE
- ☐ RCRA
- ☐ USACE
- ☐ Other ________

ADDITIONAL REQUIREMENTS

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF CONTAINERS	Hold															ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp in °C	Remarks:

*Homogenize all composite samples prior to analysis.

Page ________ of ________

ATTACHMENT B – COLLECTION FOR WATER QUALITY DATA

Water Quality Physical Parameters Data

Methods for Collecting Water Quality Physical Parameters Data

During periods of flow, water samples were collected for the determination of nitrogen and phosphorus species that truly represent the stream cross-section. This was accomplished by collecting depth-integrated samples and point (grab) samples from mid-stream at 0.5- and 1.0-m depths for comparison. These samples were collected mostly upstream of the gated control structures at locations where most of the historical data were collected. Additionally, hydrologic data were collected concurrent with each sampling event for use in the determination of instantaneous discharge. Based on these data, statistical comparisons were made between point (grab) and depth-integrated samples, and models were developed to estimate total nitrogen and total phosphorus loads.

Depth-integrated and point (grab) samples for nutrients were collected from the gated control structures at the east coast canal sites in Miami-Dade County during periods of flow. Most of the samples were collected near low tide when the gates at the sites usually are open. Depth-integrated samples were collected by means of the equal-width-increment method, which commonly is used when a discharge measurement is not made before sampling. In this method, the width of the stream is subdivided into equal-width intervals with a sampling vertical associated with each interval.

In each cross section for the study, 8 to 10 verticals were used. The first vertical selected was half the distance of the first interval from the edge of the stream bank, and the other verticals were equally spaced apart across the stream from the first vertical. Because flow velocities of the east coast canals in Miami-Dade County are nearly always less than 2 ft/s (feet per second), the weighted-bottle method was used to collect each sample. The weighted bottle does not sample isokinetically (nozzle velocity equal to stream velocity); however, it can be used during low flows and when differences in water quality across the stream are believed to be insignificant. In the initial step, the vertical with the greatest velocity is selected; the weighted bottle is then lowered and raised at a constant rate so that it is not overfilled when returned to the surface. For each specific site, this transit rate was maintained throughout for all the verticals in the cross section, and a sample from each vertical was composited in a churn splitter for processing. The transit rate as well as the nozzle size was varied for each specific site in order to prevent overfilling of the bottle. During sample processing, the water in the churn was stirred by the churn disc at a rate of about 9 in/s (inches per second) to minimize error, with care being exercised to prevent the churn disc from breaking the surface. A total of 125 mL (milliliters) of this composite sample was siphoned off into an amber polyethylene bottle, chilled immediately, and within 48 hours was shipped to the USGS Water Quality Service Unit in Ocala, Fla., for analysis. The constituents determined were total organic nitrogen, ammonia, nitrite, nitrate, nitrite plus nitrate nitrogen, total phosphorus, and orthophosphate. A point (grab) sample was collected concurrently with each depth-integrated sample. Point (grab) samples, depending on the site, were collected at 0.5 or 1.0 m below the surface near the centroid of flow by using a Niskin bottle. This bottle is spring loaded so that a messenger can trip it shut at the appropriate depth. After collection of the point (grab) sample, 125 mL of the sample was transferred from the Niskin bottle into an amber polyethylene bottle, chilled, and shipped for analysis according to protocol.

Before each sampling trip, the Niskin bottle, weighted bottle, and churn splitter were cleaned with a dilute nonphosphate detergent and then rinsed with tap water followed by deionized water. Two rinses with native water were required for the samplers before sample collection at each east coast canal site. Between sampling sites, the samplers were rinsed with deionized water before being rinsed with native water.

To ensure the integrity of the field data collected for the study, quality assurance samples (equipment blanks, field blanks, and split samples) were used extensively in the data-collection phase. Blank solutions essentially are samples free of the analytes being determined in the environmental samples. An equipment blank is a blank solution that is processed through all of the equipment used in the collection and processing of the environmental samples.

Field blanks are actually equipment blanks done in the field and are subject to all aspects of the data-collection efforts as the environmental samples, including processing, preservation, transport, and laboratory handling. Two types of field and equipment blanks were used: one for the Niskin bottle and the other for the weighted bottle and churn splitter. These two field blank solutions consisted of inorganic blank water prepared at the USGS Water Quality Service Unit and were processed and analyzed according to protocol. Analytical results from the equipment blanks and field blanks indicated that most of the concentrations for the individual nitrogen and phosphorus species were below the detection limits for the analytical methods.

Split samples were collected concurrently by the USGS and DERM to verify inter-laboratory accuracy. The sampling data consisted of point (grab) samples that were collected from the same Niskin bottle by both agencies. The samples were sent to the USGS Water Quality Service Unit and to the DERM laboratory for analysis.

Document Control #: 12-SSP-01

STANDARD OPERATING PROCEDURE FOR SOIL SAMPLING IN THE FIELD

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Revision 01
Last Review/Implementation Date: October 11, 2010

Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date

John Dwyer, Executive Vice President — 10/11/2010 Date

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1. PURPOSE

The objectives of soil sampling are to ensure that valid and representative samples are collected that meet the requirements of the project, and to prevent deterioration and contamination of the samples before laboratory analysis. By complying with procedures specified in this Standard Operating Procedure (SOP), KEMRON will ensure that the following are successfully completed:

- Proper excavation.
- Proper sampling.
- Prevention of contamination or cross-contamination of soil samples during collection. containerization, transit and storage.
- Obtain and record accurate and complete sampling data.
- Preparation of complete and correct sample labels.
- Preparation and maintenance of sample chain-of-custody.

2. SCOPE

This procedure provides technical guidance on soil sampling including decontamination procedures, types, and uses of sampling equipment. This SOP applies to KEMRON personnel and subcontractors collecting soil samples for KEMRON projects. Client, federal, or state requirements, or project-specific requirements may dictate specific types of equipment or procedures to be used when applying this SOP to a particular project. Deviations from this SOP to accommodate site-specific requirements shall be documented in project planning documents and approved by the KEMRON Project Manager and Project Quality Assurance (QA) Manager, if applicable, prior to the performance of work.

3. RESPONSIBILITIES

Project Manager Responsibilities
The KEMRON Project Manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan. The KEMRON Project Manager ensures all field staff have reviewed and signed off on this SOP prior to beginning field work. A sign-off sheet covering all SOPs related to the project will be prepared and filed with project documents.

Field Staff Responsibilities
The KEMRON field staff prepares, organizes, and inspects all equipment required for the sampling event prior to the initiation of sampling activities. The field sampler and shipper are responsible for initiating, maintaining, and transferring chain-of-custody. Upon completion of the sampling event, the field staff returns all equipment to the designated storage area ensuring that the equipment is decontaminated and in a condition ready for reuse. KEMRON field staff are required to follow procedures specified in this SOP, or approved project-specific SOP, prior to

initiating the sampling event. Field staff are required to sign off that they have read and understand this SOP prior to beginning field work.

4. DEFINITIONS

bgs: - below ground surface

Dressing Soil Surfaces - Dressing soil surfaces includes removing the exposed soil surface prior to sampling using a stainless steel shovel, spatula, knife, or spoon. The depth of soil removed depends on the technique used to expose the soil (excavation with a backhoe requires more removal than excavation with a shovel).

Homogenization - The process of mixing samples in order to minimize any bias of sample representativeness introduced by the natural stratification of constituents within the sample. To homogenize a sample, place each grab or core material into a single mixing bowl (made of suitable material i.e. stainless steel), remove any large objects such as sticks, leaves or stones, etc. and stir thoroughly with a spoon to homogenize. Scrape the soil from the sides, corners, and bottom of the pan; roll to the middle of the pan; and complete a final mixing in the center of the pan. Flatten the sample, if needed, quarter the sample, and move to the four corners of the pan. Each quarter of the sample should be mixed individually, and then rolled back to the center of the container and the entire sample mixed again. Aliquot as appropriate.

Sample Compositing - Discrete samples are taken and then proportionally combined into one sample container. Composite samples are generally used to estimate the average concentration of the individual samples that make up the composite.

5. PLANNING

The Project Manager provides any project specific information in written instructions for the sampling crew. Written instructions can be as simple as a sampling requirements report or as detailed as a Sampling and Analysis Plan (SAP). Items in written sampling instructions may include a sampling grid, depths to sample, analyses to sample for, etc.

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific SAP and Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blank, and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the SAP/QAPP.

Information regarding the sample containers, preservation techniques and holding times for soil samples will be described in the project specific SAP/QAPP.

Soil sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project health and safety officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

6. EXCAVATION AND SAMPLE COLLECTION EQUIPMENT AND PROCEDURES

The tables in this section provide a description of equipment and sampling procedures for different soil sampling methods. The project work plan will specify the method and any site-specific requirements. Once the soil is sampled, the sample will be aliquoted in the following sequence or as stated in the project-specific planning documents: Volatile Organic Compounds (VOCs), Semi-volatile Organic Compounds (SVOCs), Pesticides, Herbicides, Polychlorinated Biphenyls (PCBs), and Metals.

Soil sampling equipment and supplies can include the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific HASP (PPE, etc.)
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera (extra batteries, extra memory chip)
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan - tools plated with chrome or other materials should not be used
- Sample containers
- Ziploc plastic bags
- Logbook
- Labels
- Chain-of-Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s) with thermometer for checking temperature
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger

- Extension rods
- T-handle
- Portable electronic scale
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
- Tubes
- Points
- Drive head
- Drop hammer
- Puller jack and grip
- Backhoe

Soil VOC samples for site investigations conducted under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) or the Resource Conservation and Recovery Act (RCRA), as well as some other state or federal regulatory programs, may require that collection and analysis in accordance with USEPA Method 5035. This method specifies the use of a special sampling device. USEPA instituted this type of sampling based upon studies that indicated this purge and trap method of sampling and analysis more accurately identified the types and concentrations of VOCs in soils than prior methods. USEPA and some states determined that prior methods introduced error based upon evaporative loss of VOCs that Method 5035 is believed to be eliminated.

When implementing Method 5035 sampling and analysis, an Encore™® or Terracore™® (or equivalent and EPA-approved) sampling device is required. The samplers are attached to a T-handle that serves to assist in pushing the sampler into the soil. Three (3) samplers are usually required to be used to complete a single sample; this provides the laboratory one sample for high contaminant analysis and two samples for low level analysis. The samples are either preserved by freezing upon receipt at the laboratory, or preserved within 48 hours with the appropriate preservative (methanol or sodium bisulfate). Please note that some regulatory programs do not accept freezing of samples as an adequate means of preservation. If freezing is not allowed, sample preparation in the laboratory must be performed within 48 hours of sample collection; therefore, advance coordination with the analytical laboratory regarding sample collection, delivery and preparation is critical to a successful sampling and analysis program.

The sampler must review the manufacturer's instructions that accompany the sampling device prior to use, and must be trained by KEMRON personnel experienced in use of the sampling device prior to conduct of sampling.

Additionally, hard clays and gravels may not be able to be sampled with Encore™® or Terracore™® samplers; the Project Manager should assess the site geology to determine feasibility of sampling protocol. The Project Manager also should be aware that EnCore samplers typically have a higher associated cost than Terracores.

Use of Terracore Samplers with Pre-prepared Vials:

An undisturbed soil sample is obtained by pushing the barrel of the coring tool into a freshly exposed surface and removing the corer once filed. An approximate five (5) gram weight sample is obtained for each Terracore™®, with sample weights determined in the field using a portable electronic scale. The sample is extruded into laboratory prepared, pre-tared and labeled 40 ml vials with a stir bar. The laboratory adds the necessary preservative upon log-in. A separate sample must be collected for soil moisture content measurement, using a wide-mouth glass jar; size of the sample is not critical.

Use of EnCore™® Samplers:

The EnCore™® sampler serves as the sample shipping container. An undisturbed sample is obtained by pushing the barrel of the coring tool into the freshly exposed surface and removing the corer once filled. The exterior of the barrel is quickly wiped with a clean disposable towel to ensure a tight seal. An airtight cap is then attached to create a complete sample package. The sample is labeled and inserted into a sealable (e.g., zip lock) pouch, immediately cooled to 4 ± 2°C, and prepared for shipment to the analytical laboratory.

TABLE 1: SOIL SAMPLING WITH SPOONS, SHOVELS, POST-HOLE DIGGERS, BOWLS

Item	Description
Soil Level Identification:	Surface Soils
Soil Depth:	Based on project specific requirements, typically in range of 0 to 12" bgs
Equipment:	Shovel, post-hole digger, stainless steel spoon, stainless steel bowl
Excavation Procedure:	Use the spoon, shovel or post-hole digger as intended.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** 1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade. 2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade. 3. If VOC analysis is to be performed via Method 5035 (Encore or equivalent) sampling, the sample is to be collected directly from the soil. Otherwise, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly. 4. Place samples in cooler and cool to 4° Celsius (C) with wet ice. 5. Record sample observations in field log book. ***Post-Sampling Activities:*** Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to

	proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible. • Processing all sample paperwork, including copies provided to laboratory. • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 2: HAND-AUGER OR THIN-WALL TUBE SAMPLER

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs to the depth that manual methods become impractical
Equipment:	Stainless steel hand auger, stainless steel spoon, stainless steel bowl
Excavation Procedure:	Typically, four ()-inch auger buckets with cutting heads are pushed and twisted into the ground and removed as the auger bucket is filled. The auger holes are advanced one bucket at a time. When the depth of a sampling interval has been reached, one auger bucket is used to advance the auger hole to the first desired sampling depth.
Sampling Procedure	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler. Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet. The following procedure is used for collecting soil samples with the auger: 1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod. 2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an

area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.
5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip, and the core from the device.
9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
10. If VOCs analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. If Method 5035 (Encore or equivalent) sampling is required, the sample is to be collected directly from the core. Otherwise, place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
11. Place samples in cooler and cool to 4° Celsius (C) with wet ice.
12. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
13. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.
14. Record sample observations in field log book.

Post-Sampling Activities:

Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.

	• Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 3: SHELBY OR "PUSH TUBE"

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs to depth that manual methods become impractical
Excavation Procedure:	Drive the tube into the soil with a drill rig, if necessary.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** 1. Drill to first sampling depth, as described in the project planning documents. 2. Place decontaminated Shelby tube sampler on center rods. 3. Drive Shelby tube sampler with the push head of the drill rig, as described in ASTM Method D1587. Retrieve the sampling tube and remove the disturbed material from the top of the tube. In addition, remove 1 inch of soil from the base of the tube. Place an impervious disk at both ends of the tube seal with a wax plug prior to shipment to the laboratory. 4. If Shelby tubes are to be extruded in the field for composite sampling, the driller will use a hydraulic extruder to obtain the sample. Samples will then be composited, as necessary, for analysis. 5. Screen sampler with PID (if required). 6. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples. The sample tube should be packed in Styrofoam™ plugs or other cushioning material to prevent disturbance of the sample while en route to the geotechnical laboratory for analyses. 7. Record sample observations in field log book. ***Post-Sampling Activities:*** Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to: • Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of

	individual responsible. • Processing all sample paperwork, including copies provided to laboratory. • Compiling all field data for site records. • Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 4: BACKHOE TRENCH OR BUCKET SAMPLING

Item	Description
Soil Level Identification:	• Surface Soils • Shallow Subsurface Soils
Soil Depth:	• Based on project specific requirements, typically in range of 0 to 12" bgs • 12 inches bgs – depth manual methods become impractical or the depth that a backhoe can reach
Excavation Procedure:	Backhoe operator is instructed what area to excavate.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** A backhoe can be used to remove sections of soil when detailed examination of soil characteristics is required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation. The following procedures are used for collecting soil samples from test pits or trenches: 1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities. 2. Review the site specific HASP and ensure that all safety precautions including appropriate monitoring equipment are installed as required. 3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than four feet deep must be sloped or protected by a shoring system, as required by OSHA regulations. 4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done. 5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket. KEMRON employees are not to enter trenches for the purposes of sample collection unless done so under specific health & safety requirements, work plans or procedures.

6. If VOC analyses are required, the Encore or equivalent sampling device is to be used to collect the sample. Otherwise, transfer the sample into an appropriate, labeled sample container with a stainless steel spoon or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
7. Place samples in cooler and cool to 4° Celsius (C) with wet ice.
8. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.
9. Record sample observations in field log book.

Post-Sampling Activities:

Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.
- Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 5: DRILL RIGS WITH SPLIT SPOON (BARREL) SAMPLER

Item	Description
Soil Level Identification:	Deep Subsurface Soils
Soil Depth:	> 20 feet
Excavation Procedure:	***Split-Spoon – Hammer driven:*** • Drive the split-spoon sampler either inside a hollow-stem auger or inside an open borehole after rotary drilling equipment has been temporarily removed. • Record the number of blows required to drive the spoon for each six-inch interval. • Remove the spoon. ***Continuous split-spoon:*** While drilling, advance the hollow-stem auger containing the split-spoon.
Sampling Procedure:	***Preparation:*** 1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required. 2. Obtain necessary sampling and monitoring equipment. 3. Decontaminate or pre-clean equipment, and ensure that it is in working order. 4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate. 5. Perform a general site survey prior to site entry in accordance with the site specific HASP. 6. Use stakes, flagging, or cones to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. Underground utility clearance should be performed in accordance with KEMRON's utility clearance SOP 12-UTL-00, and utility clearance should always be confirmed before beginning work. ***Sampling:*** Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augered hole and the core extracted. When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils". The following procedures are used for collecting soil samples with a split spoon: 1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top. 2. Place the sampler in a perpendicular position on the sample material. 3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result. 4. Record in the site log book or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth. 5. Withdraw the sampler, and open by unscrewing the bit and head and splitting

the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 ½ inch diameters. A larger barrel may be necessary to obtain the required sample volume.

6. Without disturbing the core, use the Encore or equivalent to collect the soil sample for VOC analysis. Otherwise, transfer portion for VOC analyses to appropriate labeled sample container(s) and seal tightly.
7. Place the remaining soil into a stainless steel bowl, homogenize, and transfer portions for remaining analyses to appropriate sample container(s) and seal tightly.
8. Place in cooler and cool to 4° Celsius (C) with wet ice.
9. If another sample is to be collected in the same hole, but at a greater depth, reattach a (decontaminated / clean) split spoon sampler to the drill rod, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
10. Abandon the hole according to applicable state regulations or install monitoring well if applicable in accordance with the site work plan and KEMRON SOP 12-MWC-00.

Post-Sampling Activities:

Several activities need to be completed and documented once soil sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.
- Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

7. FIELD RECORDS

7.1 Field Logbook

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific soil sampling documentation include the following:

a. Site Name;
b. Date and time of arrival;
c. Weather conditions, including ambient air temperature and/or miscellaneous observations;
d. Location;
e. Date and time sampling;
f. Sample location and boring ID;
g. Sample depth;
h. Sample sequence number;
i. Sample Equipment ID;
j. Method of sampling;
k. Field parameters (pH, conductivity, and temperature);
l. Fractions sampled and preservation method(s);
m. Signature of Sampler and Date;
n. Physical characteristics, to include the general soil type (sand, silt, clay, and organic/other matter such as wood chips, as determined using the Unified Soil Classification System (USCS)), approximate grain size (fine, medium, coarse), presence of observable biota, odor, color, layers, depth of layer changes;
o. Pertinent field observations and comments;
p. Site drawing showing sample locations, dimensions, and prominent features.

7.2 Chain-of-Custody

Completed chain-of-custody (COC) forms must be maintained for all samples. Sample labels should be checked against the completed COC for accuracy. An example COC form is included as Attachment A. The original form will be sent to the laboratory with the samples and signed by the lab upon receipt. Original completed COC forms will be returned to KEMRON with the analytical report. A copy of the COC will be retained by the field crew (sampler) and kept with field records.

8. DECONTAMINATION

All reusable equipment taken to the field will be clean. The following procedures, or that specified in the site-specific documents, are completed during a sampling event for multiple uses of equipment, at the conclusion of equipment use at the site and if visual evidence indicates a need at the onset of equipment use.

1. Hand wash, or flush/pump through automatic samplers/equipment, with a non-phosphate laboratory detergent dissolved in tap water;

2. Rinse with tap water;
3. Rinse thoroughly with deionized water;
4. Allow to air dry;
5. Wrap in aluminum foil and a plastic bag, if needed, and store in a clean, dry area.

Decontamination solvent such as isopropanol, acetone or hexane may be used for equipment exposed to high levels of organics (e.g., NAPL). Use of specific decontamination solvents should be addressed in project-specific planning documents and the waste solvents managed and disposed in accordance with applicable regulations.

9. SAMPLE SHIPMENT

Equipment decontamination chemicals may need to be shipped to the site and samples may need to be shipped to the laboratory. All shipping is completed in accordance with the current federal and international regulations (DOT, EPA, and IATA). Shipping stipulations are to be included in the project documents, but should you have any questions regarding shipping of any materials to and from the site, contact the authorized shipping representative in your home office.

10. ASSOCIATED SOPs

Field Log Book 07-DOC-00

Well Installation 12-MWC-00

Utility Clearance 12-UTL-00

11. REFERENCES

ASTM D 1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils"

USEPA Environmental Response Team, Feb 2000. Standard Operating Procedure (SOP) #2012, Soil Sampling.

USEPA, July 2002. Draft Revision 1, Method 5035A, Appendix A, The Collection and Preservation of Aqueous and Solid Samples for Volatile Organic Compound (VOC) Analysis.

ATTACHMENT A –EXAMPLE CHAIN OF CUSTODY FORM

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

KEMRON ENVIRONMENTAL SERVICES

Phone: 740-373-4071
Fax: 740-373-4835

CHAIN-OF-CUSTODY RECORD

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project #:	Project Name:
Sampler (print):	Signature:

Program
- ☐ NPDES
- ☐ AFCEE
- ☐ RCRA
- ☐ USACE
- ☐ Other ________

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF CONTAINERS	Hold																	ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)
Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp in °C	Remarks:

*Homogenize all composite samples prior to analysis

Page ______ of ______

Document Control #: 12-MWI-01

STANDARD OPERATING PROCEDURE FOR MONITIORING WELL INSTALLATION OVERSITE

Issue Date: November 3, 2005
Revision 01

Last Review/Implementation Date: October 2010

Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date

John Dwyer, Executive Vice President — 10/11/2010 Date

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1. POLICY

KEMRON personnel will follow procedures established in the "SOP for Monitoring Well Installation" unless specified in a site-specific work plan (WP) or Field Assignment Form (FAF). Personnel will follow all Health and Safety procedures as directed by the Corporate Health and Safety Plan and the Site Specific Health and Safety Plan.

2. PURPOSE

The purpose of this SOP is to provide steps ensuring that the oversight of MW installation or MW installed by hand-auger are completed as stated in this SOP.

3. SCOPE

This procedure provides a method for oversight for temporary and permanent monitoring well installation. MW installation by hand augering is also included in this SOP. This procedure does not address MW design / construction.

4. RESPONSIBILITIES

4.1 Project Manager Responsibilities

The KEMRON Project Manager provides the site-specific MW design and ensures MW installation specifications are provided in a WP or FAF.

4.2 Field Staff Responsibilities

KEMRON Field Staff are required to follow this SOP unless a written site-specific WP or FAF is provided by the Project Manager. Deviations from this SOP require prior written approval from the KEMRON Project Manager or his designated alternate where such deviations can be the WP or FAF. Field staff properly document the MW installation.

5. DEFINITIONS

5.1 Annular Space

The space between two concentric tubes or casings, or between the casing and the borehole wall. This would include the space(s) between multiple strings of tubing/casings in a borehole installed either concentrically or multi-cased adjacent to each other.

5.2 Borehole

A circular open or uncased subsurface hole created by drilling.

5.3 Bridge

An obstruction within the annulus which may prevent circulation or proper placement of annular materials.

5.4 Casing

A pipe, finished in sections with either threaded connections or beveled edges to be field welded, which is installed temporarily or permanently to counteract caving, to advance the borehole, and/or to isolate the zone being monitored.

5.5 Casing, Protective

A section of larger diameter pipe that is placed over the upper end of a smaller diameter monitoring well riser or casing to provide structural protection to the well and restrict unauthorized access into the well.

5.6 Grout

A low permeability material placed in the annulus between the well casing or riser pipe and the borehole wall (in a single-cased monitoring well), or between the riser and casing (in a multi-cased monitoring well), to maintain the alignment of the casing and riser and to prevent movement of groundwater or surface water within the annular space.

5.7 Multi-cased well

A well constructed by successively smaller diameter casings with depth.

5.8 Filter Pack

A clean silica sand or sand and gravel mixture of selected grain size and gradation that is installed in the annular space between the borehole wall and the well screen, extending an appropriate distance above the screen, for the purpose of retaining and stabilizing the particles from the adjacent strata.

5.9 Permanent Monitoring Well

Permanent monitoring wells are wells used for routine collection of groundwater samples and/or other data.

5.10 Riser

The pipe extending from the well screen to or above the ground surface.

5.11 Soil plug

A plug which naturally forms at the bottom of the hollow-stem auger while drilling. This soil plug must be removed when sampling or installing well casings. Plug removal can occur by washing out the plug using a side discharge rotary bit, or augering out the plug with solid-stem auger bit sized to fit into the hollow-stem auger.

5.12 Tamper

A heavy cylindrical metal section of tubing that is operated on a wire rope or cable. It slips over the riser and fits inside the casing or borehole annulus. It is generally used to tamp annular sealants or filter pack materials into place and prevent bridging.

5.13 Temporary Monitoring Well

Temporary monitoring wells (TMW's) are wells used for collecting groundwater samples for screening purposes.

5.14 Well Development

Well development serves to remove the finer grained material from the well screen and filter pack that may otherwise interfere with water quality analysis, restore groundwater properties disturbed during the drilling process and to improve the hydraulic characteristics of the filter pack and hydraulic communication between the well and the hydrologic unit adjacent to the well screen.

5.15 Well Screen

A filtering device used to retain the primary or natural filter pack; usually a cylindrical pipe with openings of a uniform width, orientation, and spacing.

5.16 Tremie Pipe

A pipe used to deliver filter pack or grout from the surface to specific depths. Use of the tremie pipe is recommended when there is a bridge potential.

6. PLANNING

6.1 Work Plan (WP) or Field Assignment Form (FAF)

The Project Manager prepares the WP or FAF to include the following:

a. Known or suspected geology at site
b. Known or suspected depth to water (DTW) at well locations
c. Proposed well locations
d. Proposed well construction details
   i. Total depth of well
   ii. Borehole diameter
   iii. Screen length
   iv. Diameter of well (and other casing, if applicable)
   v. Well screen material (PVC, SS, etc.) and construction
   vi. Filter pack size
   vii. Screen slot size
   viii. Filter pack seal (fine sand, bentonite, etc.)
   ix. Depth of filter pack
   x. Depth of seal
   xi. Method of grouting
   xii. Depth of outer casing if applicable
   xiii. Surface completion (flush-mount, stick-up, etc.)

e. Surface conditions at proposed well locations (concrete, asphalt, gravel, etc.)
f. Provision for field adjustment of proposed well locations

g. Method of well installation
h. Permits required
i. Traffic conditions at site
j. Health and Safety concerns (vapors, traffic)
k. Special circumstances such as sands, bedrock, perched water, confining layer, etc.

6.2 Pre-mobilize Planning

6.2.1 Field staff review and understand WP

6.2.2 Field staff or PM to contact drillers
The drilling company is contacted to arrange for meeting times and to provide drilling specifications such as borehole diameter and approximate depth, casing diameters, screen slot size, and well head or manhole cover requirements.

6.2.3 Utility Clearance implemented (per KEMRON SOP)

6.2.4 Site owner is informed of the work date

6.2.5 HASP is prepared

6.2.6 Mobilization equipment, supplies, etc are gathered
This may include field book, site map, directions to the site, HASP, surveying equipment, IP, supplies for well development, soil/groundwater sampling supplies (see appropriate SOP), markers, measuring tape or wheel, safety cones, and tools.

7. MW INSTALLATION AND OVER-SITE PROCEDURE

The tables in this section provide MW installation or over-site procedures for permanent and temporary wells. Any site specific requirements will be included in the project WP or FAF. See Attachment A for well diagrams.

TABLE 1: PERMANENT MW INSTALLATION OVERSITE (via HOLLOW-STEM AUGER, SOLID STEM AUGER, ROTARY DRILLING, SONIC DRILLING, etc.)

Item	Description
Pre-Installation Procedure	• Check-in with site owner / personnel • Verify utility markings (per utility clearance SOP) • Mark off proposed well locations per WP or FAF • Posthole dig each well location (per Utility Clearance SOP) • H&S tailgate meeting with driller and HASP signing • Check drill rig for safety issues (missing bolts, damaged cables, etc.) and environmental issues (leaking oil, equipment not cleaned from previous sites, etc.) • Brief drillers on location; equipment decon; and excluding the use of oils, petroleum based lubricants, solvents and PVC cement / glue; and IDW containment
MW Installation Procedure	• Per WP or FAF observe / ensure… o generation of a stable and straight borehole of specified diameter o measurement of depth to water to be sure screened area will bracket the water table o plug removal (hollow-stem augering only) o screen, riser, and casing are correct construction and specification o filter pack installation o assembly of the correct lengths of screens and risers o perpendicular installation o assembly does not float o riser extends to a correct height relative to the ground surface o riser is finished evenly so groundwater measurements are consistent o placement of a temporary cap for the remainder of the installation o filling and tamping of annular space with filter pack material o addition of any hydrating material o depth to filter pack meets installation specifications o installation of a seal above the filter pack o seal material meets the installation specifications o application and tremieing of grout mixture and that materials meet specifications o installation of multicased wells, if specified o leak-proof installation of protective casing and manhole/vault o installation of a sloping concrete pad at or below the ground level and installation of the outer protective casing o cuttings and purge fluids are properly contained • Mark the name of the well in the wet concrete or on the well vault • Lock the outer protective casing • Specified time intervals between hydrating bentonite, grout placement, and well development are followed.

	• Installation of weep holes in stick-up surface completion, if applicable.
Well development	• Develop the well as stated in the WP or FAF. See the Groundwater Sampling SOP.
Documentation	• Document the date, drilling company, drillers names, KEMRON employees on site, weather conditions, initial groundcover per well location, document observations / drillers comments during each drilling event (i.e., competency, blow counts, circulation, etc.), borehole diameter, screen and riser lengths, depth to filter pack, depth, surface completion details and thickness of seal

TABLE 2: TEMPORARY MW INSTALLATION (via HAND AUGERING OR DIRECT PUSH)

Item	Description
Pre-Installation Procedure	• Check-in with site owner / personnel • Verify utility markings (per utility clearance SOP) • Mark off proposed well locations per WP or FAF • Posthole dig each well location (per Utility Clearance SOP) • H&S tailgate meeting with driller and HASP signing • Brief drillers on location; equipment decon; and excluding the use of oils, petroleum based lubricants, solvents and PVC cement / glue; and IDW containment
MW Installation Procedure	• Per WP or FAF… o Ensure/generate a stable and straight borehole o measure DTW to be sure screened area will bracket the water table o install filter pack installation o assemble the correct lengths of screens and risers o ensure perpendicular installation o ensure assembly does not float o ensure riser extends to a correct height relative to the ground surface o place a temporary cap for the remainder of the installation o fill and tamp annular space with filter pack material o add any hydrating material o ensure depth to filter pack meets installation specifications o install a seal above the filter pack o ensure seal material meets the installation specifications o If specified, apply grout mixture where materials meet specifications o Install a temporary well cover o Cuttings and purge fluids are properly contained • Lock the outer protective casing
Well development	See Table 1
Documentation	See Table 1

8. FIELD RECORDS

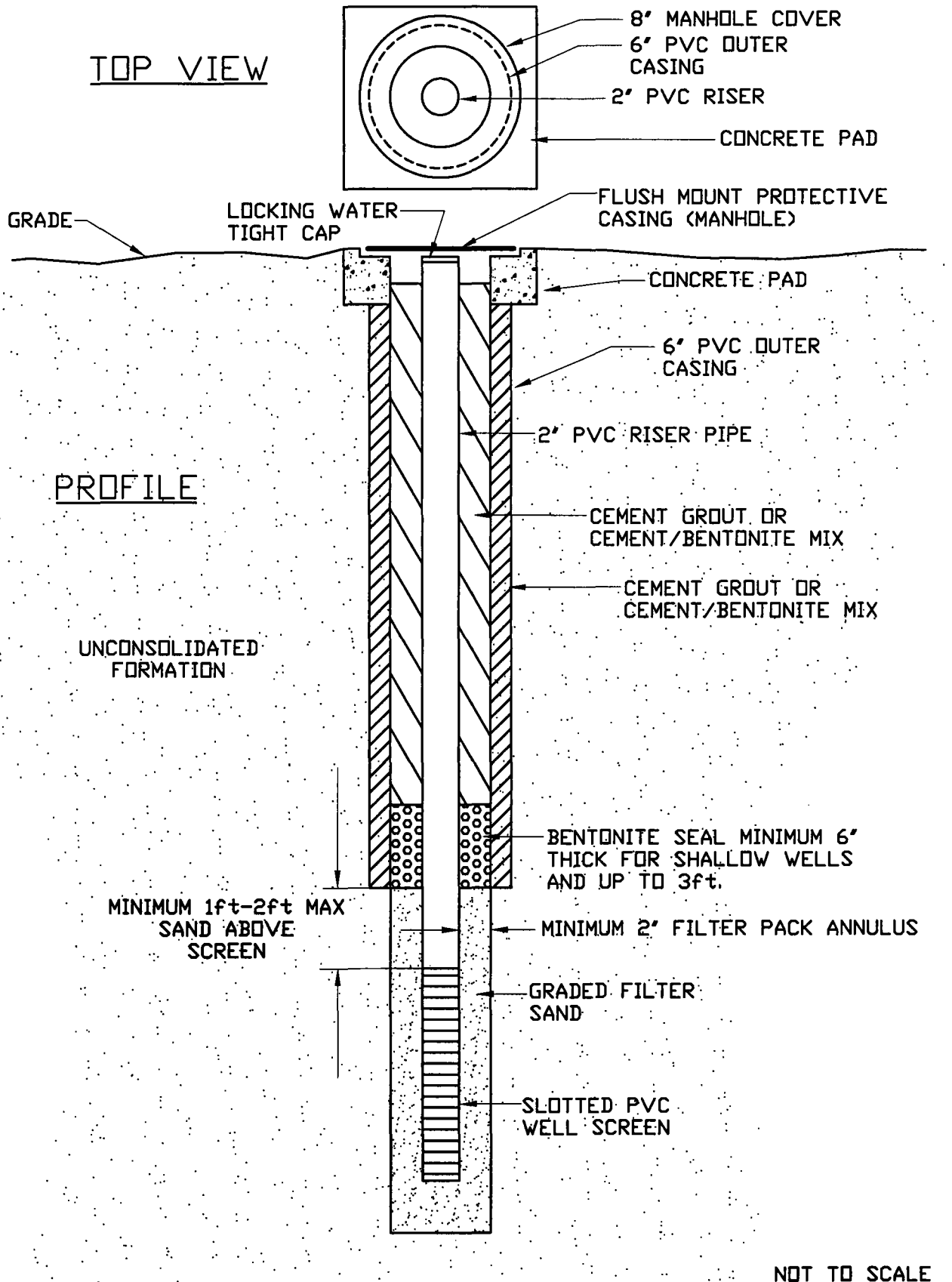
8.1 *Field Logbook*

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific well installation documentation include the following:

xiv. Site Name;
xv. Date and time of arrival;
xvi. Weather conditions, including ambient air temperature and/or miscellaneous observations;
xvii. Location;
xviii. Date and time drilling;
xix. Drilling location and boring ID;
xx. Well depth;
xxi. Physical characteristics, to include the general soil type (sand, silt, clay, and organic/other matter such as wood chips, as determined using the Unified Soil Classification System (USCS)), approximate grain size (fine, medium, coarse), presence of observable biota, odor, color, layers, depth of layer changes;
xxii. Plan and section view sketches of well construction;
xxiii. Pertinent field observations and comments.

ATTACHMENT A –MONITORING WELL INSTALLATION DIAGRAMS

TYPICAL DOUBLE-CASED PVC MONITORING WELL CONSTRUCTION DIAGRAM



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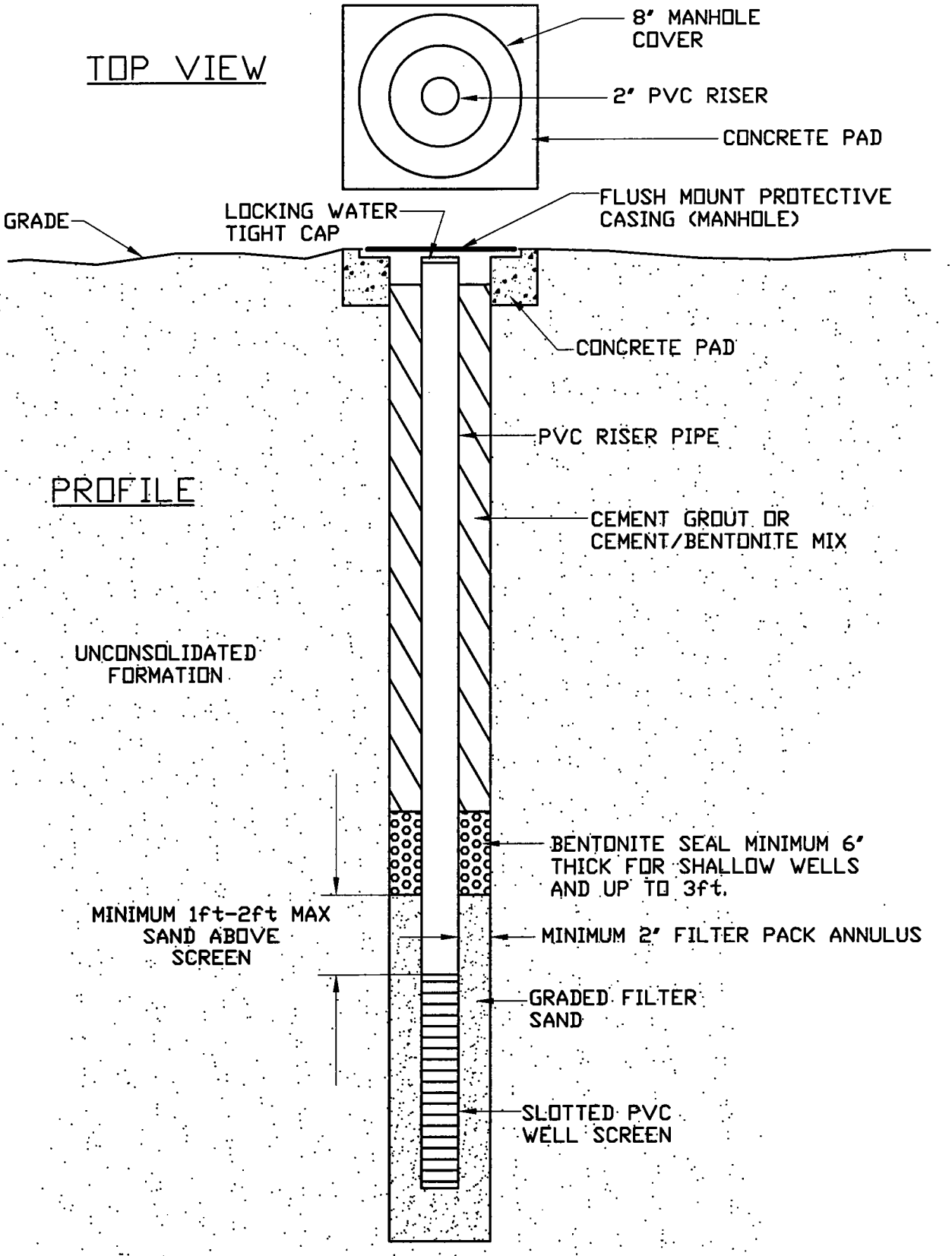
TYPICAL PVC MONITORING WELL CONSTRUCTION DIAGRAM



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Document Control #: 12-MWC-00

STANDARD OPERATING PROCEDURE FOR MONITORING WELL CONSTRUCTION

Issue Date: November 8, 2004
Revision 00

Last Review/Implementation Date: November 2004

KEMRON Environmental Services

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Marilyn K. Zumbro

11/8/04

Marilyn Zumbro, Regional Manager — Date

11/8/04

John Dwyer, Vice President — Date

Kelly McClemens

11/8/04

Kelly Clemons, Corporate Quality Assurance Manager — Date

The following persons have read and understand this SOP:

Signature	Date

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1. PURPOSE

The purpose of this procedure is to provide a monitoring well construction procedure. Personnel will follow all Health and Safety procedures as directed by the Corporate Health and Safety Plan and the Site Specific Health and Safety Plan.

2. SCOPE

This procedure addresses general steps for monitoring well construction. Related drilling activities fall outside the scope of this procedure.

3. RESPONSIBILITIES

3.1 *Project Manager Responsibilities*

The Project Manager instructs the KEMRON field staff on the monitoring well construction activities and provides the well construction plan to the field staff.

3.2 *Field Staff Responsibilities*

The field staff is responsible for assisting the Project Manager when needed to ensure all materials and supplies are available and ready for construction and to follow the plan for construction in the field. Any deviations are communicated and approved by the Project Manager prior to execution of the changes.

4. DEFINITIONS

4.1 *Annular Space*

The space between two concentric tubes or casings, or between the casing and the borehole wall. This would include the space(s) between multiple strings of tubing/casings in a borehole installed either concentrically or multi-cased adjacent to each other.

4.2 *Permanent Monitoring Well*

Permanent monitoring wells are wells used for routine collection of groundwater samples and/or other data.

4.3 *Temporary Monitoring Well*

Temporary monitoring wells (TMW's) are wells used for collecting groundwater samples for screening purposes.

5. GENERAL

5.1 All wells will be installed by a properly licensed driller or well installation contractor, where applicable.

5.2 All necessary state and local permits must be obtained prior to beginning the work.

5.3 Equipment, tools, materials, etc. will be properly decontaminated, prior to site arrival and prior to each well installation, to prevent possible introduction of contaminants in the wells during construction. If the area is known, or believed, to be contaminated, a decontamination solution appropriate for the contaminant of concern will be used. All decontamination waste will be properly managed.

5.4 In most cases, hollow stem augers should be used for the installation of permanent or temporary monitoring wells. However in some cases it may be necessary to use air or mud rotary or direct push technology (DPT) or hand auger to install the wells.

5.5 The monitoring wells will be properly developed. After the grout has been allowed to cure, the development of the wells can be completed by surging, over-pumping, surge-blocking, or some other approved method. The monitoring wells should be developed until it yields water that is free of sediment..

5.6 All development water and/or drill cuttings impacted by a contaminant must be properly managed to prevent further spread of contamination.

5.7 The completed monitoring wells shall be surveyed at the top of the casing at marked location and concrete pad with reference to either the National Geodetic Vertical Datum (N.G.V.D) or local bench mark.

5.8 Document well construction. Typical Well Construction Diagrams are attached.

6. PERMANENT MONITORING WELL CONSTRUCTION SPECIFICATIONS

6.1 Prior to well installation, under ground utilities will be identified as stated in the KEMRON SOP - Subsurface Utility Clearance.

6.2 Before the drilling of each well is initiated, hand augering or post hole must be initiated for the top four (4) feet below the land surface, in order to determine if utilities are present at the location of the drilling.

6.3 The outer diameter of the hollow stem auger will be at least six (6) inches in diameter if a 4 inch casing is to be installed, and at least four (4) inches in diameter if a 2 inch casing is to be installed. A minimum 2-inch filter pack annulus is required for both cases.

6.4 The monitoring well(s) will be two (2) inch or four (4) inch diameter with threaded joints. The monitoring well casing will be constructed of stainless steel, P.V.C. (schedule 40 minimum) or approved equal.

6.5 Monitoring wells typically have a well screen that is two (2) inch or four (4) inch diameter with a preferred slot size of 0.010-inch; if a slot size of 0.020-inch or higher is installed, a coarser sand pack must be used. The American Society for Testing and Materials (ASTM) D 5092-90 recommends that the slot size should retain at least 90%

(and preferable 99%) of the sand pack. The top of the well screen should typically be set at least two (2) feet above the normal annual high water table and the bottom at least three (3) feet below the annual low water level.

6.6 The screened interval should be determined based on lithology and site specific information such concentrations detected at the shallow monitoring wells. For double-cased wells, a minimum of 2-inch annular space between casings is recommended and the top of the well screen must be a minimum of 2 feet below the outer casing.

6.7 Solvents or P.V.C. glue will not be used. P.V.C. threaded/slip couplings may be secured using stainless steel screws or rivets. Care should be exercised to ensure that the stainless screws do not protrude inside the well casings.

6.8 The monitoring well casing will be installed true vertical in the drilled bore hole.

6.9 A clean washed sand pack will occupy the annular space from the bottom of the bore hole to least one (1) foot above the well screen. The ASTM D 5092-90 recommends the use of sand with a grain size that is about four to ten times greater than the d-30 (30% finer) grain size of the hydrologic unit being filtered. Use of a filter sock in lieu of a sand pack is not acceptable.

6.10 Hydrated bentonite will be used to create a seal (minimum 6-inch thick) above the annular space sand pack material to avoid grout migration into the sand pack. The remaining annular space between the casing and in the bore hole will be grouted with neat cement or with a cement/bentonite mix.

6.11 Permanent monitoring wells will be constructed in such a way that they are not prone to surface water infiltration or located in low areas that experience repeated flooding.

6.12 Construct permanent monitoring wells so that the well heads are protected. Protection may include metal covers set in a concrete pad sloping away from the well or protective containment such as standup metal well casing.

6.13 The top of the permanent monitoring wells will be sealed with a water tight locking cap. The well will be kept locked at all times to prevent tampering.

6.14 All permanent monitoring wells will be adequately labeled to distinguish them as monitoring wells and will be properly secured to prevent vandalism and/or the accidental introduction of contaminants.

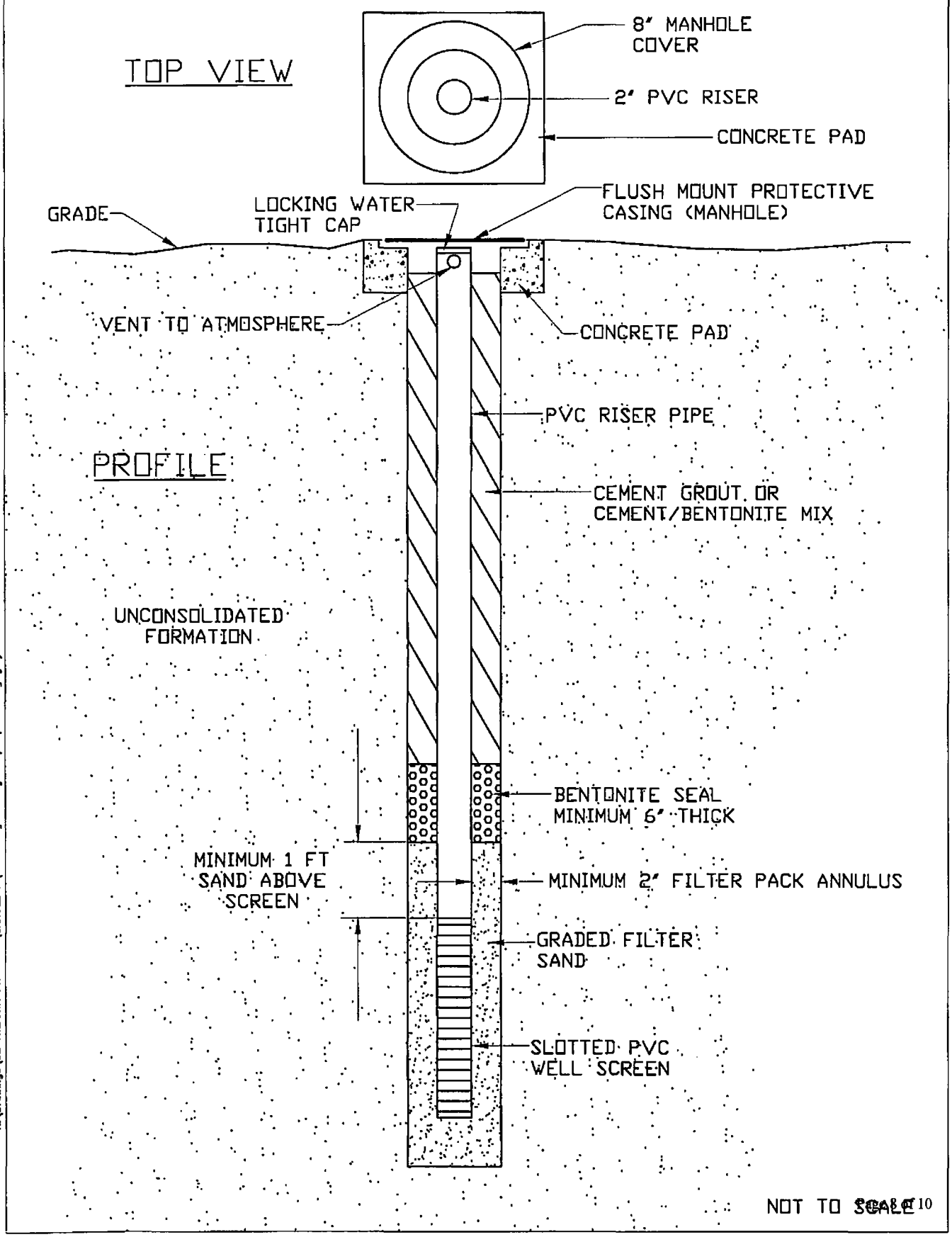
7. TEMPORARY MONITORING WELL CONSTRUCTION SPECIFICATIONS

7.1 Prior to installation of a temporary well (TMW), under ground utilities will be identified as stated in the KEMRON SOP – Subsurface Utility Clearance.

7.2 TMWs can be installed using a hollow stem auger or direct push technology. A clean washed sand pack will occupy the annular space from the bottom of the bore hole to one (1) foot above the well screen. A bentonite seal, cement grout and manhole cover may not be necessary for TMWs; but a water tight locking cap is required. See the work plan for the site specific requirements since there are a variety of methods for temporary well construction.

APPENDIX A - WELL CONSTRUCTION DIAGRAM

TYPICAL PVC MONITORING WELL CONSTRUCTION DIAGRAM



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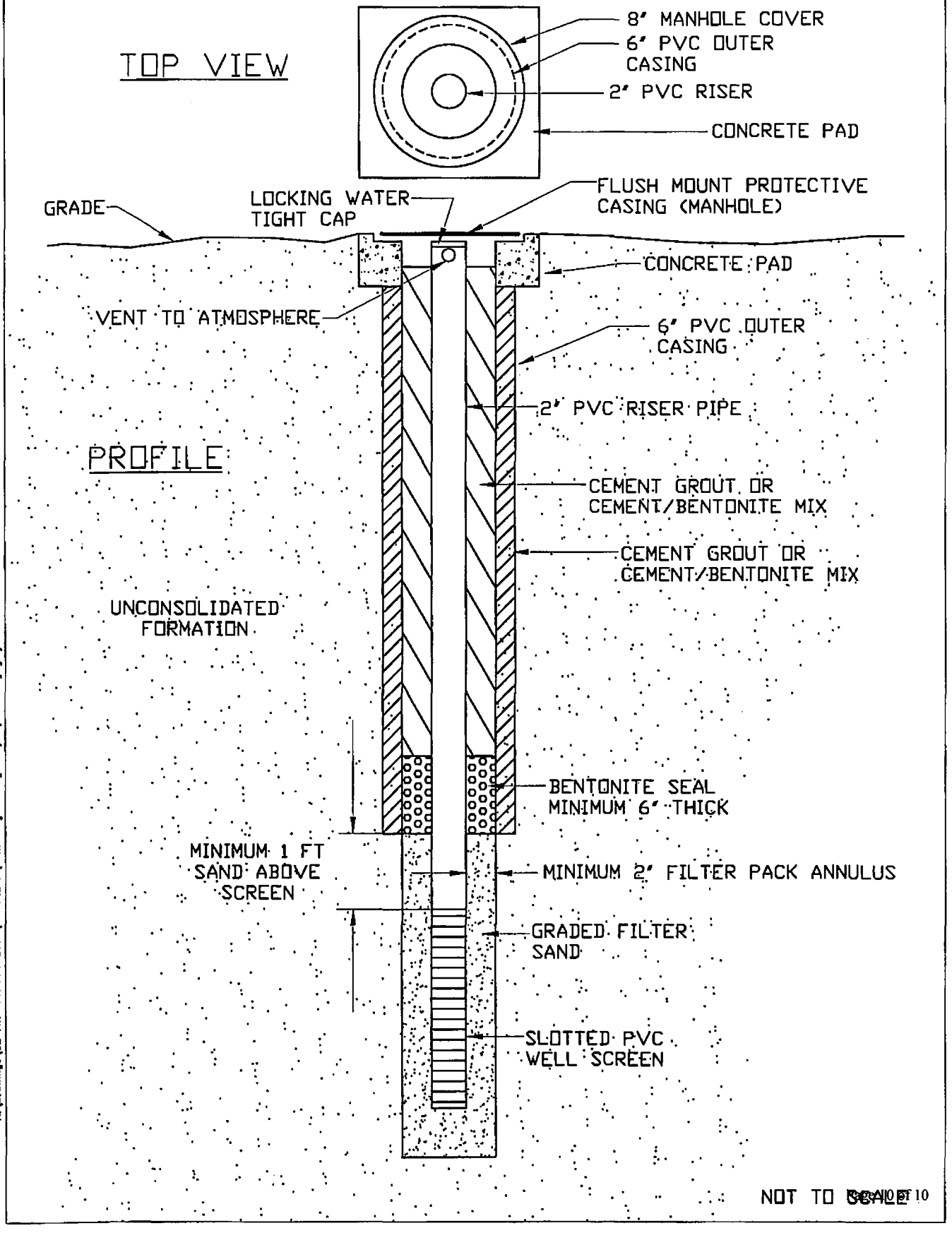
APPENDIX B – DOUBLE-CASED WELL CONSTRUCTION DIAGRAM

TYPICAL DOUBLE-CASED PVC MONITORING WELL CONSTRUCTION DIAGRAM



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Document Control #: 12-GWS-01

STANDARD OPERATING PROCEDURE FOR GROUNDWATER SAMPLING

Issue Date: November 8, 2004

Revision 01
Last Review/Implementation Date: October 11, 2010

Approved by:

Lou Ehrhard, Corporate Quality Assurance Manager — 10/11/2010 Date

John Dwyer, Executive Vice President — 10/11/2010 Date

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1. PURPOSE

The objectives of this procedure are to ensure valid and representative samples of groundwater are collected from monitoring wells that meet project requirements and to prevent deterioration and contamination of samples before laboratory analysis. By complying with procedures specified in this Standard Operating Procedure (SOP), KEMRON will ensure that the following are successfully completed:

- Proper well purging
- Proper sampling
- Prevention of contamination or cross-contamination of groundwater samples during collection, containerization, transit and storage
- Obtain and record accurate and complete sampling data
- Preparation of complete and correct sample labels
- Preparation and filing of sample chain of custody
- Communication of adequate instruction to the laboratory

2. SCOPE

This SOP provides technical guidance on groundwater sampling including decontamination procedures, types, and uses of sampling equipment. This SOP applies to KEMRON personnel and subcontractors collecting groundwater samples for KEMRON projects. Client, federal, or state requirements, or project-specific requirements may dictate specific types of equipment or procedures to be used when applying this SOP to a particular project. Deviations from this SOP to accommodate site-specific requirements shall be documented in project planning documents and approved by the KEMRON Project Manager and Project Quality Assurance (QA) Manager, if applicable, prior to the performance of work.

3. RESPONSIBILITIES

Project Manager Responsibilities
The KEMRON Project Manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan. The KEMRON Project Manager ensures all field staff have reviewed and signed off on this SOP prior to beginning field work. A sign-off sheet covering all SOPs related to the project will be prepared and filed with project documents.

Field Staff Responsibilities
The KEMRON field staff prepares, organizes, and inspects all equipment required for the sampling event prior to the initiation of sampling activities. The field sampler and shipper are responsible for initiating, maintaining, and transferring chain-of-custody. Upon completion of the sampling event, the field staff returns all equipment to the designated storage area ensuring that the equipment is decontaminated and in a condition ready for reuse. KEMRON field staff

are required to follow procedures specified in this SOP, or approved project-specific SOP, prior to initiating the sampling event. Field staff are required to sign off that they have read and understand this SOP prior to beginning field work.

4. DEFINITIONS

Bailer: A bailer is used to sample wells. A bailer may be of stainless steel, Teflon, polyethylene or other appropriate construction and may be composed of a reel system equipped with a Teflon-coated stainless steel suspension cord, a disposable monofilament line or disposable braided filament line. A bailer can be dedicated or non-dedicated to a specific well and disposable or reusable.

Chain-of-Custody: An unbroken trail of accountability that ensures the physical security of samples, data, and records. The chain-of-custody documentation shows times, dates, and names of the individuals relinquishing and receiving the samples identified on the record.

Custody: Physical control of an object, in this case of an environmental sample.

Dedicated: Equipment (bailer or pump) dedicated to a particular well which remains associated only with the assigned well until the work/project is completed.

Depth to Water: The depth to water is the depth from the top of the well casing to the surface of the groundwater. An established point (typically the surveyed top of the well casing) is utilized as a reference point for measuring the depth to water.

Evacuated Well: Removal of standing volume of water in a well by pumping or bailing.

Field Blank: A field blank sample undergoes the full handling and shipping process of an actual sample. It is designed to detect sample contamination that can occur during field operation or during shipment. Field blanks are prepares using certified clean water preserved in the same manner as the other collected samples, and then submitted to the laboratory for analysis.

Flow Rate: A measurement taken of the water flowing from the well when a pump is used. The flow rate is in units of volume per time.

Low-Flow Purge: Per EPA guidance, low-flow purging is purging at rate of 200-500 mL/min to have minimal drawdown of the water table and minimize disruption of the water column. Low-flow purging is accomplished using low-flow purge equipment.

Non-Dedicated: Equipment (bailer or pump) not dedicated to a particular well. Non-dedicated equipment is decontaminated between use or if for one-time use (bailers) then properly disposed of after use.

Purging: The process of removing stagnant water from a monitoring well (water in the screen, well casing, and saturated annulus) immediately prior to sampling, causing its replacement by groundwater from the adjacent formation, which is representative of actual aquifer conditions.

Recharged Well: A well is considered recharged when the water level recovers to at least 80% of the static water level.

Slow Recovery Wells: Slow recovery wells are wells that require more than 6 hours to recharge after purging.

Stabilized Well: Well stabilization is achieved following well purging when water level and field measurements are observed to be consistent between measurements and within project work plan requirements.

Trip Blank: Unlike field blanks, the trip blanks are used for VOCs analysis only. In addition, trip blanks are prepared prior to going into the field by filling containers (VOC vials) with clean water. The samples are kept closed, handled, transported to the field, and then returned to the laboratory in the same manner as the other samples. Trip blanks are used to evaluate error associated with shipping and handling, and analytical procedures. They are used in conjuncture with field blanks to isolate sources of sample contamination already noted in previous field blanks.

Unrecoverable Wells: Wells that have not sufficiently recovered within 24 hours.

Water Column Height: Well depth minus the depth to water.

Water Level Measurement Reference: The point on the well head where water levels are measured (typically the high point of the casing). This point is marked and surveyed as soon as practical after well installation to ensure consistent measurement.

Water Quality Measurements: Groundwater chemistry measurements used to establish the quality of groundwater. Measurements may include: temperature, pH, conductivity, dissolved oxygen (DO), oxidation reduction potential (ORP), and turbidity.

Well Stability: Stability is achieved as specified in the project work plan. The following are provided as a reference.

- Change in groundwater chemistry measurements is within a specified range (indicating stability) by the end of the third well volume purged; or
- Stabilization is considered complete after five well volumes are purged even if field parameters have not met specified range (range established to indicate stability); or
- Stabilization is complete when well is fully evacuated. Using these criteria, measurements are taken to verify the evacuation. Allow well to recharge prior to sampling.

Well Volume Calculation: The standing water volume for typical well diameters is below:

1" Casing = .041 gal/ft
2" Casing = .163 gal/ft
4" Casing = .653 gal/ft
6" Casing = 1.469 gal/ft

or can be calculated using the following formula:

$$Volume\ (gallons) = (3.14)\, r^2\, h \times (7.48 \frac{gal}{ft^3})$$

r = inside well casing radius (in feet)
h = height (in feet) of the water column in the well

5. PLANNING

The Project Manager provides written instructions of any project-specific information for the sampling crew. The written instructions can be as simple as a sampling requirements report or as detailed as a Sampling and Analysis Plan (SAP). Items in written sampling instructions may include the following:

a) Purging method;
b) Criteria for a complete purge;
c) Criteria for stabilized well;
d) Wells to sample;
e) Method of sampling;
f) Analytical parameters;
g) Applicable regulations for handling purged water;
h) Volume to purge;
i) Sample containers to be used for each analysis;
j) Preservative for each analysis; and
k) Instructions for samples to be field filtered or lab filtered for dissolved metals, if appropriate.

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific SAP/Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blank, and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the SAP/QAPP.

Information regarding the sample containers, preservation techniques and holding times for groundwater samples will be described in the project specific SAP/QAPP.

Groundwater sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards. These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project health and safety officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

6. SAMPLE COLLECTION EQUIPMENT AND PROCEDURES

The tables in this section provide a description of equipment and sampling procedures for different groundwater sampling methods. The project work plan will specify the method and any site-specific requirements. Once the groundwater is sampled, the sample will be aliquoted in the following sequence or as stated in the project-specific planning documents: Field Parameters, Volatile Organic Compounds (VOCs); Semi-volatile Organic Compounds (SVOCs), Pesticides, Herbicides, PolyChlorinated Biphenyls (PCBs), Metals, and Wet chemistry parameters.

Groundwater sampling objectives may vary significantly between projects. Project objectives should be defined within the project-specific planning documents. The list of required materials below identifies the types of equipment which may be used for a range of groundwater sampling applications. The various types of sampling equipment which may be used include:

Well Purging Equipment

- Bailers
- Bladder pumps
- Submersible pumps
- Peristaltic pumps
- Centrifugal Pumps
- Waterra™ pumps

Field Instruments

- Individual or multi-parameter meter(s) to measure temperature, pH, specific conductance, DO, ORP, and/or turbidity
- Flow through cell
- Water level measuring device
- Interface probe or product detection paste

Sampling Equipment

- Reusable or disposable bailers
- Bladder Pump
- Electric Submersible Pump
- Suction Lift Pumps (Peristaltic)

Sample Preparation Equipment

- Filtration equipment
- Intermediate containers
- Sample kit (i.e., bottles, labels, preservatives, custody records, cooler)

General Equipment

- Well keys
- Project-specific sampling plans (SAP, QAPP, HASP) including map of wells to be sampled
- Sample collection records
- Field notebook/pen
- Waterproof marker pens
- Deionized water dispenser bottler
- Sample cup
- Buckets
- Coolers, or sample shuttles
- Instrument calibration solutions
- Power source (e.g. generator and/or battery)
- Equipment decontamination supplies
- Health and safety supplies (PPE, etc)
- First-Aid kit
- Tool box (e.g. bolt cutter, turkey baster, etc.)
- 55-gallon drum for collecting purge water
- Graduated cylinder (determine flow)
- Flow controller for submersible pump

Expendable Materials

- Deionized water supply
- Disposable bailer string (nylon or polypropylene)
- 0.45 micron filters
- Paper towels
- Plastic sheeting
- Ice/blue ice for sample preservation
- Disposable latex powder-free glove liners
- Disposable nitrile gloves
- Plastic trash bags
- Ziploc® bags

This equipment list was developed to aid in field organization and should be used in preparation for each sampling event. Depending on the site-specific sampling plan, additional material and equipment may be necessary and should be determined before the scheduled sampling event. Similarly, not all of the items shown in this list may be necessary for any one sampling event.

TABLE 1: Purging and Sampling by Bailer

Purging & Sampling Equipment:	The Bailer system can be dedicated or non-dedicated. a) Bailer: pre-cleaned closed-top Teflon® or stainless steel bailer or a disposable polyethylene bailer. b) Either of the following: (1) New braided nylon or polypropylene cords, one per well (disposable). (2) Reusable lanyards (monofilament, stainless steel, or Teflon-coated) for bailer systems.
Sampling Procedure:	Prior to sampling, obtain sample containers from laboratory (or laboratories). Ensure presence of preservatives in bottles as appropriate. Before opening the well, a clean working surface shall be set up around the well head using a clean plastic sheet. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment. ***Preliminary Measurements:*** Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark). To obtain a water level measurement, lower the probe of a water level measuring device into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to nearest 0.01 feet) by repeatedly raising and lowering the tape to converge on the exact measurement. Obtain the reading from the top of casing (TOC) measuring point. The water level measurement should be entered on the Groundwater Sample Collection Record or in the field log book. The measurement device shall be decontaminated immediately after use with a non-phosphatic detergent and rinsed with distilled water. Generally, only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface or allowed to become kinked. Measuring devices, including interface probes, which come into contact with free product, will likely require more thorough decontamination. Calculate the water level (water column height) and volume of water in casing. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface probe as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. Wells containing free product are generally not used for groundwater sampling, since the concentration of contaminants present in the free product can adversely affect the quality of the water sample, lending to a non-representative water sample. ***Purge Well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in the work plan) will be collected from the decontaminated / cleaned purge equipment. a) Slowly and gently, lower the bailer into the top of the standing water column. Keeping the bailer at the top of the water column is crucial throughout purging especially at the end of purging. b) Allow bailer to fill and remove from the well. Bailer rope should never touch the ground surface at any time during the purge routine. c) Collect purged water in a graduated bucket, etc. to determine the number of well volumes purged. d) Following each well volume purged, take water level and field parameter measurements. e) Containerize the purged water unless specified otherwise in the project work plan.

Purging a well to dryness may occur under some low-yield conditions. When the well recovers, a cascading effect may occur within the screened zone which can volatilize some organic compounds. This may be considered inappropriate by regulatory agencies when VOCs are the target analyte of interest. Purging a well to dryness, then sampling after it has recovered may be acceptable for other target analytes, however. Under low yield conditions, low-flow sampling pumps such as bladder pumps may be required for VOC sample collection.

Stabilize well: Measure and record water level until 80% static water level is achieved (well fully recharged). Groundwater chemistry stability is achieved as specified in the project work plan.

Sampling

a) Sample wells:
   - Within six hours of purging unless it is a slow recovery well or
   - As soon as sufficient recharge water is available or up to 24 hours after purging for slow-recovery wells or wells that purge completely dry.

b) Use one pre-cleaned or disposable bailer per well.
c) Attach new nylon rope or other appropriate cord to the bailer.
d) Slowly immerse the bailer into the top of the water column to reduce agitation and splashing.
e) Fill bailer and promptly remove from well.
f) Record sample observations in field log book.

Aliquot Sample: Aliquot the sample into the containers necessary for both laboratory analysis and any further field measurements. For laboratory analysis, include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. Place in cooler and cool to 4° Celsius (C) with wet ice.

Groundwater samples collected for total dissolved metals analyses will be field filtered prior to being placed in sample containers and properly preserved. Groundwater filtration will be performed using a peristaltic pump and a 0.45-micron in-line water filter. Disposable filters are commonly available in 0.45-micron size. Low-capacity or high-capacity cartridges are available and may be selectively used based on sample turbidity.

The filtration of groundwater samples shall be performed either directly from the pump discharge line or from laboratory-supplied intermediate containers. In either case, well purging shall be performed first. Fresh groundwater shall then be filtered directly into sample containers.

Post-Sampling Activities: Several activities need to be completed and documented once ground-water sampling has been completed. These activities include, but are not limited to:

- Ensuring that all field equipment has been decontaminated and returned to proper storage location. Once the individual field equipment has been decontaminated, tag it with date of cleaning, site name, and name of individual responsible.
- Processing all sample paperwork, including copies provided to laboratory.
- Compiling all field data for site records.
- Verifying all analytical data processed by the analytical laboratory against field sheets and chain-of-custody to ensure all appropriate sample analyses has been returned to sampler.

TABLE 2: Pump Assisted Purging and Bailer Sampling

Purge Equipment:	Motor-driven centrifugal lift pump
Sampling Equipment:	The bailer system can be dedicated or non-dedicated. a) Bailer: pre-cleaned closed-top Teflon® or stainless steel bailer or a disposable polyethylene bailer. b) Either of the following: (1) New braided nylon or polypropylene cords, one per well (disposable); (2) Reusable lanyards (monofilament, stainless steel, or Teflon-coated) for dedicated bailer systems.
Sampling Procedure:	Before opening the well, a clean working surface shall be set up around the well head using a clean plastic sheet. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment. ***Preliminary Measurements:*** Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark) and calculate the water level (water column height) and volume of water in casing. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. Ensure that water level meter / interface probes are probes are decontaminated after each use. If multiple wells are to be sampled, work from cleanest to most contaminated based on analytical data and/or field observations made during soil boring / well installation. ***Purge well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in work plan) will be collected from the decontaminated / cleaned purge equipment. Operate all petroleum fueled power sources or related equipment downwind from the sampling location. The well should not be pumped dry during low-flow purging. 1. Lower the pump / hose assembly into the top of the standing water column. Keep assembly towards top of water column so that the purging will evacuate water from the formation into the screened area of the well and up through the casing to remove the entire static volume. 2. Begin purging the water and calculate flow rate. 3. Determine the time necessary to purge one-half the well screen volume or that specified in the work plan. Take field parameters, depth to water, and flow-rate measurements every five to ten minutes (or more frequently depending on the time necessary to purge one-half the well screen volume). 4. Stop purging when necessary well volume has been removed and parameters have stabilized. 5. Containerize the purged water unless specified otherwise in the project work plan. ***Stabilize well:*** Measure and record water level until 80% static water level is achieved (well fully recharged). Measure and record field parameters until groundwater chemistry stability is achieved as specified in the project work plan. See Appendix A for reference information. ***Sampling*** 1. Sample wells: a) Within six hours of purging unless it is a slow recovery well or b) As soon as sufficient recharge water is available or up to 24 hours after purging for slow-recovery wells or wells that purge completely dry.

	2. Use one pre-cleaned or disposable bailer per well. 3. Attach new nylon rope or other appropriate cord to the bailer. 4. Slowly immerse the bailer into the top of the water column to reduce agitation and splashing. 5. Fill bailer and promptly remove from well. 6. Record sample observations in field log book. ***Aliquot Sample:*** Aliquot the sample into the containers necessary for both laboratory analysis (see Tables 4, 5, 6 and 7) and for any further field measurements. For laboratory analysis, include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. Place in cooler and cool to 4° Celsius (C) with wet ice. ***Decontamination:*** After sampling a well, decontaminate all reusable equipment and samplers prior to future use. See Section 7 for field measurements.

TABLE 3: Low Flow Purge and Sample Collection

Purging and Sampling Strategy:	Low-Flow Purge Collection Method with pump
Purge and Sampling Equipment:	Dedicated or non-dedicated equipment may be used. Low-Flow Purge System includes submersible or non-submersible pump, tubing, and suspension cable.
Sampling Procedure:	***Pump Installation and Set-up***: (1) Open the well and allow water level to equilibrate before taking a water level measurement. (2) Measure the depth to water at the pre-marked well casing reference point (notch or other visible mark) and calculate the water level (water column height) and volume of water in casing. (3) Set submersible pump or pump tubing within the screen interval or slightly above screened interval of the well. (4) Place the pump inlet or submersible tubing just below the surface of the water column. (5) Set-up associated equipment (generator, compressor, tubing, etc.) ***Preliminary Measurements:*** Ensure well is fully recharged after pump / tubing installation by taking a water level measurement. Note: Following water-level measurement, if free phase product is suspected, measure the product thickness with an interface probe if and as specified in the work plan. If measurable quantities of product are encountered do not sample and notify the Project Manager immediately. ***Purge and Stabilize Well:*** Note: If non-dedicated equipment is used for purging, at least one equipment blank (or as specified in work plan) will be collected from the decontaminated / cleaned purge equipment. Operate all petroleum fueled power sources or related equipment downwind from the sampling location. The well should not be pumped dry during low-flow purging. The intent of low flow purging is to minimize drawdown. 1. Lower the pump inlet as the well is purged. 2. Calculate Flow Rate of purged groundwater (range 200 - 500 ml per minute). 3. Flow rate may be adjusted at this point but flow-rate should not exceed the recharge rate of the aquifer. Monitor by measuring the top of the water column with a water level recorder or similar device while purging. Continue to measure the water level flow-rate until stability is achieved. 4. A maximum of a 0.3 foot draw down produced by the pump is allowed. 5. Take field parameters, depth to water, and flow-rate measurements every 5–10 minutes (or more frequently depending on the time necessary to purge one-half the well screen volume). 6. Containerize the purged water unless specified otherwise in the project work plan. 7. Sampling occurs after the water level and groundwater chemistry parameters are stable as specified in this SOP or in project work plans. ***Sampling:*** 1. Reduce the flow to a low level to minimize sample disturbance, if necessary, and collect sample directly from the discharge tubing connected to the pump. Discharge tubing should not contact the inside of the sample containers. 2. Aliquot samples according to the number and analysis type as specified in the work plan and include initials, date and time of sample collection, sample ID, preservative and analysis required on sample container label. 3. Place in cooler and cool to 4° Celsius (C) with wet ice. 4. Record sample observations in field log book.

	Decontamination: After sampling, decontaminate the low-flow purge equipment prior to further use. See Section 7 for field measurements.

7. FIELD MEASUREMENTS

Unless specified otherwise in the project documents, the following measurements will be taken and evaluated for acceptability:

Parameter	Acceptable Range for establishing stability*
Temperature	10% of reading
pH	±0.2 standard units
Conductivity	±3% of readings
Dissolved oxygen	±10% of reading
Redox potential(ORP or Eh)	±20 mV
Turbidity*	≤5 NTU or ±10% reading, whichever is greater

* Three consecutive acceptable measurements typically establish water quality.

8. FIELD RECORDS

8.1 Field Logbook

See the Field Documentation SOP, 07-DOC-00, for generic documentation items. Examples of specific groundwater sampling documentation include the following:

a. Facility name;
b. Date and time of arrival;
c. Weather conditions, including ambient air temperature and/or miscellaneous observations;
d. Location;
e. Well designation;
f. Well sequence number;
g. Static water level;
h. Total depth of well;
i. Depth to water;
j. Casing diameter;
k. Well volume or one-half well screen volume;
l. Method of purging;
m. Start time of purging;
n. Total volume of water evacuated from well;
o. Evacuation completion time;
p. Sampling strategy (bailer manual, bailer automated, low-flow);
q. Flow rate recorded (if applicable);
r. Field equipment calibration check;

s. Water quality measurements;
t. Water level at time of sample collection;
u. Date and time sample collected;
v. Sample sequence number;
w. Sample Equipment ID;
x. Other pertinent observations of water samples (color, turbidity, odor, etc.);
y. Fractions sampled and preservation method;
z. Signature of sampler(s) and date; and
aa. Miscellaneous information, i.e. well was purged dry prior to taking 3 evacuation volumes.

8.2 Chain-of-Custody

Completed chain-of-custody (COC) forms are maintained for all samples. A sample COC form is included as Attachment A. The original form is sent to the laboratory with the samples and signed by the lab upon receipt. Original completed COC forms are returned to KEMRON with the analytical report. A copy of the COC will be retained by the field crew and kept with field records.

9. DECONTAMINATION

All equipment taken to the field will be clean. Decontamination of equipment is performed:

- During a sampling event for multiple uses of equipment,
- At the conclusion of equipment use at the site, or
- If visual evidence indicates a need for decontamination at the onset of equipment use. This does not apply for dedicated equipment.

The following procedures, or those specified in the site-specific documents, are completed for reusable non-dedicated equipment:

1. Hand wash, or flush / pump through automatic samplers/equipment, with a non-phosphate laboratory detergent dissolved in tap water;
2. Rinse with tap water;
3. Rinse thoroughly with deionized water;
4. Allow to air dry;
5. Wrap in aluminum foil and a plastic bag, if needed, and store in a clean, dry area;

Decontamination solvent such as isopropanol, acetone or hexane may be used for equipment exposed to high levels of organics (e.g., NAPL). Use of specific decontamination solvents should be addressed in project-specific planning documents and the waste solvents managed and disposed in accordance with applicable regulations.

10. SHIPPING

Equipment decontamination chemicals may need to be shipped to the site and samples may need to be shipped to the laboratory. All shipping is completed in accordance with the current federal and international regulations (DOT, EPA, IATA). Shipping stipulations are to be included in the

project documents, but should you have any questions regarding shipping of any materials to and from the site, contact the authorized shipping representative in your home office.

11. ASSOCIATED SOPs

Field Log Book 07-DOC-00
Well Installation 12-MWC-00

12. REFERENCES

USEPA, Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers, EPA 542-S-02-001, May 2002.

USEPA, Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedure, EPA 540-S-95-504, April 1996.

USEPA, RCRA Groundwater Monitoring, Draft Technical Guidance, November 1992.

USEPA, Potential Source of Error in Groundwater Sampling at Hazardous Waste Sites, EPA 540-S-92-019, August 1992.

ATTACHMENT A – EXAMPLE CHAIN-OF-CUSTODY

COC No. A 39472

109 Starlite Park
Marietta, OH 45750

KEMRON ENVIRONMENTAL SERVICES

CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project #:	Project Name:
Sampler (print):	Signature:

Program: ☐ NPDES ☐ AFCEE ☐ RCRA ☐ USACE ☐ Other ______

ADDITIONAL REQUIREMENTS

Sample I.D. No.	Comp*	Grab	Date	Time	Protocol CWA	Protocol SW846	NUMBER OF CONTAINERS	Hold																ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)

Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Cooler Temp. in °C	Remarks:

*Homogenize all composite samples prior to analysis.

Page ______ of ______

ATTACHMENT B – EXAMPLE GROUNDWATER SAMPLE COLLECTION FORM

Well Number _____ Project Name:
Sample No. _____ Project Number:
Date _____ Collectors:

Well Information

Monument Condition ☐Good ☐Needs Repair_____
Well Cap Condition ☐Good ☐Locked ☐Replaced ☐Needs Replacement
Elevation Mark ☐ Yes ☐ Added ☐other
Well Diameter ☐ 2-inch ☐4-inch ☐6-inch ☐Other
☐Odor Comments _____

Purge Data

Total Well Depth _____ ft ☐ Clean Bottom ☐Muddy Bottom ☐Not Measured
Depth to Water _____ ft
Casing Volume _____ ft (H20) X _____ gpf = _____ X 3 = _____ gallons
3/4 " = 0.02 gpf 1"= 0.04 2" = 0.16 gpf 4"=0.65 gpf 6"=1.47 gpf

Purge Method

Pump Type: Tubing: Total volume purged _____
Sample Intake Depth _____ Purge Rate _____
Purge Start Time _____ Purge Stop Time _____ Sample Rate _____

Field Parameters

Time	Gallons	pH	Turbidity (NTU)	Temperature (C)	Conductivity (mS/cm)	D.O. (mg/L)	Comments

Sampling Device

Filter Type: Size:

Sample Containers Collection Time_____

Tag No.	Type		Preservative	Analytical Method	QA Remarks
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			
	☐Amber	☐Poly			

APPENDIX D

Air Monitoring Field Documentation Sheets

FORM E
REAL TIME AIR MONITORING LOG

Date	Time	Work Area	Location	PID Serial No.	PID Reading (ppm)	PDR Serial No.	PDR Reading (mg/m^3)		Comments	Name
							Conc.	TWA		

FORM F
AIR SAMPLE RECORD

Company Name:	**Analytical Lab:**
Project Name:	
Project Contact:	
Phone No:	**Contact:**
Fax No:	**Phone No:**

Sample Location	Pump ID	Filter No	Tube No	Date		Time		Run Time (min)	Pump Flow Rate (L/min)			Total Volume (L)	Sample ID	Analysis	NIOSH Method
				Start	Stop	Start	Stop		Start	Stop	Average				

Sampled by: (Print/Sign) _____

Special Request: _____

Comments:

Date	Time	Temp.					
_____ Weather @	_____	_____°C / Humidity	_____% / Bar.	_____ mm Hg / Wind from	_____ @	_____ mph
_____ Weather @	_____	_____°C / Humidity	_____% / Bar.	_____ mm Hg / Wind from	_____ @	_____ mph

CompanyName: Project Contact: Project Name: Location:	TAT: Phone No. Project # PO #

Sampling ID	DESCRIPTION	Sampling Period Time (min)		Total (min)	Pump ID	Pump Flow Rate (L/min)	Total Volume (L)
		Start	Stop				

Sampled By: (Print/Sign): _____

Special Requests: _____

APPENDIX E

Laboratory Analytical Standard Operating Procedures

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STANDARD OPERATING PROCEDURES FOR GASOLINE RANGE ORGANICS SW846 8015

Issue/ Implementation Date: 15 March 2010

Last Review Date: 15 April 2012

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Michael D. Albertson, Volatiles Supervisor — Date: 4/1/12

Leslie S. Bucina, Quality Assurance Officer — Date: 4-10-12

David E. Vandenberg, Managing Director — Date: 4/10/12

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1.0 SCOPE AND APPLICATION

1.1 Microbac Standard Operating Procedure GCV05 pertains to the determination of Gasoline Range Organics alkane range C_6 to C_{10} in solid and liquid matrices using purge-and-trap Gas Chromatography. Per client request, the GRO alkane range may be broadened to C_{12}. The Ohio BUSTR regulation has defined a GRO carbon range of C6 – C12.

1.2 This method references USEPA Methods 8000B/8015B/5030B (12/96), 8000C (03/03), 8015C (02/07), 8015D (06/03), 5030C (05/03), 5035A (07/02), and AFCEE QAPP's 1998 (3.0), 2001 (3.1), and 2005 (4.0).

1.3 Microbac SOP GCV05 applies to all volatile organic GC analyses except where a client's specific Quality Assurance Project Plan (QAPP) supercedes this document.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCV	Continuing Calibration Verification
DI water	Deionized water
FID	Flame Ionization Detector
GC	Gas Chromatography
GRO	Gasoline Range Organics
ICAL	Initial Calibration
ICV	Initial Calibration Verification
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MSDS	Material Safety Data Sheets
NCR	Nonconformance Report
PID	Photo Ionization Detector
PPE	Personal Protective Equipment
QA	Quality Assurance
QC	Quality Control
RL	Reporting Limit

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RT	Retention Time
SOP	Standard Operating Procedure
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Gloves must be worn while handling any chemicals, standards, or samples. Other required PPE include lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following VOC's have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene	carbon tetrachloride
chloroform	vinyl chloride

2.3 The toxicity or carcinogenicity of the other reagents and analytes used in this method have not been precisely defined, therefore, each chemical should be treated as a potential health hazard. Exposure to the compounds should be reduced to the lowest possible level. Procedures involving concentrated samples and primary standard preparation must be performed in a fume hood.

2.4 Broken glassware hazards exist when handling glassware. Glassware must be inspected prior to use. Broken glassware must be discarded immediately in the broken glassware receptacle located in the laboratory.

2.5 MSDS are available to all employees for each analyte and reagent used within the laboratory. MSDS should be consulted prior to handling chemicals.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Pre-cleaned 40 mL glass VOA vials with Teflon-faced silicone septum must be used for water samples. Pre-cleaned 120 mL glass screw cap vials with Teflon caps or Encore (or equivalent) containers must be used for solid/soil matrices.

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3.2 Water samples for VOA analysis should be preserved with concentrated HCl (pH ≤ 2) and stored at ≤ 6° C, thus achieving a 14 day holding time from date of collection. Non-preserved water samples have a holding time of 7 days and should be stored at ≤ 6° C. Waste, soil, sludge, and oil samples do not require any preservative other than being stored at ≤ 6° C and have a 14 day holding time from date of collection.

3.3 Soil preservation varies with the state or program, and may include freezing, collection in methanol, addition of sodium bisulfate, or simply storage at ≤ 6° C. For samples utilizing method 5035A, refer to Microbac SOP PAT01 for preservation and storage information.

3.4 Upon completion of sample analysis and hold time expiration, samples are removed from storage units and returned to sample archive. Samples requiring internal chain-of-custody are returned to the sample custodian to complete the chain.

3.5 Sample hold-time is defined as time elapsed from collection date and time to sample analysis date and time.

3.6 At a minimum, 40 mL of water and 5.00 g of soil are required for analysis. Smaller sample volumes dictate dilution analysis.

4.0 METHOD PERFORMANCE

4.1 Table 1 summarizes the performance data for water and soil/solid waste analysis. This table includes the ranges for accuracy and precision, current laboratory MDLs, nominal laboratory RLs, and True values.

4.2 The laboratory performed an initial assessment of the MDLs using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 1 and were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project RLs may be higher.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS

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to assess precision/accuracy and to annually evaluate the associated control limits.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Samples for Volatile Organics analyses are susceptible to laboratory contaminants. To eliminate the potential for interferences from other areas of the laboratory, the Volatiles laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Soil analysis may result in low surrogate standard recovery due to the poor purging efficiencies of some soil matrices. In the event that this occurs, reanalysis of the sample is performed to confirm matrix effects.

5.3 Prior to sample analysis, a system blank is analyzed to demonstrate the system is free of contaminants and interferences.

5.4 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. Contamination by carryover during a non-monitored run may require the re-analysis of the affected sample(s).

5.5 Samples may become contaminated by diffusion of volatile organics through the septum into the sample during shipment and storage. A trip blank prepared from organic-free reagent water is carried through the sampling and handling process and serves as a check on such contamination.

5.6 Storage blanks are placed in sample refrigerators to monitor potential cross-contamination. Refer to Microbac SOP MSV01 for storage blanks procedure.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph (GC): Hewlett-Packard (HP) 5890 Series II equipped with O.I. Analytical FID; Agilent 6890 GC equipped with Agilent FID; Agilent 7890 equipped with O.I. Anaytical FID/PID

6.2 Liquid sample concentrator (LSC): Tekmar LSC 2000, 3000, Stratum

6.3 Autosampler: Varian Archon

6.4 Columns: Restek, RTX 502.2, 60m, 0.53 mm I.D, 3.0 um; RTX1, 60 m, 0.53 mm ID, 3.0 um; Restek RTX 502.2, 105 m, 3.0 mm.

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6.5 Trap: J, K, M traps (Supelco)

6.6 Balance (top loading): Ohaus Navigator, Mettler PE600

6.7 Volumetric Flasks: 1 mL to 20 mL class "A"

6.8 VOA vials: 40 mL glass (Eagle Pitcher, ESS)

6.9 Syringe: 5 mL gas-tight with Luer lock tip (Hamilton)

6.10 Micro-syringes: 5 uL, 10 uL, 25 uL, 50 uL, 100 uL, 500 uL, 1000 uL (Hamilton)

6.11 Mininert vials: 2 mL, 5 mL, 10 mL, with septum valve

6.12 Spatulas: Stainless steel and wooden

6.13 Pasteur pipets: 1 mL, 10 mL disposable (Kimble)

6.14 pH paper: range 0-14, 0-2.5 (Macherey Nagel, EMD)

6.15 Equivalent equipment and supplies may be used.

6.16 Refer to Table 2 for Instrument operating parameters

6.17 Computer, software, hardware:

Instrument	Operation System	Computer Name	Connection Type	Instrument Software
HP3	Windows 2000 Professional	C10036	1 Gbps	Enviroquant Chemstation B.01.00
HP5	Windows XP Professional	C10010	1 Gbps	Enviroquant Chemstation C.00.00
HP19	Windows 7 Professional	HP19	1 Gbps (2)	Enviroquant Chemstation E.02.02.1431

7.0 STANDARDS AND REAGENTS

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All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 GRO Primary standards (unleaded gasoline):

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
Initial calibration	Ultra Scientific	RGO – 601	5,000 ug/mL
ICV/CCV	CPI	4401-020246-06	5,000 ug/mL

7.2 Primary surrogate standard:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
chlorobenzene	Accustandard	AS-E0006	1000 ug/mL

7.3 Primary GRO retention time markers standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
2-methylpentane	Chem-Service	F2186S	100 ug/mL
1,2,4-trimethylbenzene	Chem-Service	F832S	100 ug/mL
n-dodecane	Chem-Service	F2000S	100 ug/mL

7.4 GRO intermediate standards:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ug/mL)
Initial calibration	5000	900	5	900
ICV/CCV	5000	900	5	900

7.5 GRO intermediate retention time markers standards:

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PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ug/mL)
2-methylpentane	100	1000	3	33.3
1,2,4-trimethylbenzene	100	1000	3	33.3
n-dodecane	100	1000	3	33.3

7.6 GRO Intermediate surrogate

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ug/mL)
Primary stock surrogate	1000	150	10	15

7.7 Working GRO retention time markers standard:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL DI water)	FINAL CONCENTRATION (ug/L)
WATER ANALYSIS				
Intermediate retention time standard	33.3	50	50	33.3
SOIL ANALYSIS				
Intermediate retention time standard	33.3	5	5	33.3

7.8 Sodium bisulfate: J.T. Baker (granular or monohydrate)

7.9 Methanol: purge and trap grade (J.T. Baker)

7.10 Purified sand: J.T. Baker

7.11 DI water: On demand UV-treated

7.12 Standards stored at < 0°C or per manufacturer's instructions. Standards stored in glass vials with Teflon-lined lids and/or mininert vials. Expiration dates for primary standards are per manufacturer's instructions, intermediate standards have a 30 day expiration date from the preparation date (standards tracked via solution numbers).

7.13 Equivalent standards and reagents may be used.

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7.14 Working standards prepared per the following:

Working Standards Preparation

INTERMEDIATE SURROGATE VOLUME (15 ug/mL)	INTERMEDIATE GRO VOLUME (900 ug/mL)	FINAL VOLUME (DI water)	SURROGATE WORKING STANDARD CONCENTRATION	GRO WORKING STANDARD CONCENTRATION
WATER				
10 uL	5 uL	50 mL	3 ug/L	90 ug/L
50 uL	20 uL	50 mL	15 ug/L	360 ug/L
100 uL	50 uL	50 mL	30 ug/L	900 ug/L *
150 uL	100 uL	50 mL	45 ug/L	1800 ug/L **
200 uL	200 uL	50 mL	60 ug/L	3600 ug/L
250 uL	400 uL	50 mL	75 ug/L	7200 ug/L
SOIL				
10 uL	5 uL	50 mL	3 ug/Kg	90 ug/Kg
5 uL	2 uL	5 mL	15 ug/Kg	360 ug/Kg
10 uL	5 uL	5 mL	30 ug/Kg	900 ug/Kg *
15 uL	10 uL	5 mL	45 ug/Kg	1800 ug/Kg **
20 uL	20 uL	5 mL	60 ug/Kg	3600 ug/Kg
25 uL	40 uL	5 mL	75 ug/Kg	7200 ug/Kg

* Denotes LCS/MS/MSD/LCSD
** Denotes CCV/ICV

NOTE: Soil standards are prepared with 5.00 g (±0.05 g) of reagent sand.

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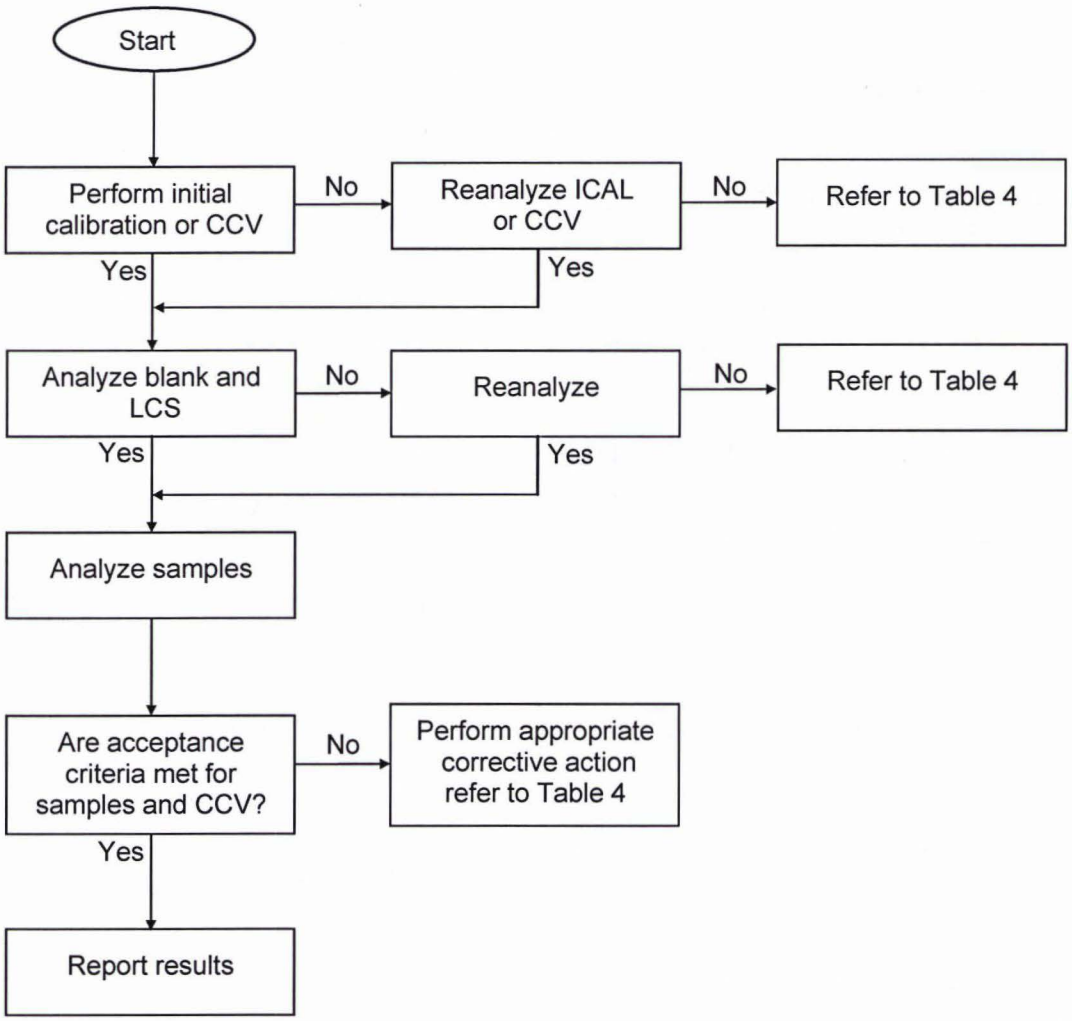
8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



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9.0 SAMPLE PREPARATION

9.1 Refer to purge-and-trap procedures found in Microbac SOP PAT01.

10.0 CALIBRATION PROCEDURES

10.1 Prior to standards and samples, a window defining RT standard is analyzed. This standard contains 2-methyl pentane (C_6) and 1,2,4-trimethylbenzene (C_{10}) used to define the GRO integration range (n-dodecane used for C_{12}).

10.2 The initial calibration is matrix specific and contains a minimum of five calibration levels (the lowest standard concentration is at or below the RL). All analyses are performed using parameters in Table 2.

10.3 Upon completion of initial calibration standards analyses calibration factors (CF), the average CF ($\overline{CF}$), and the percent relative standard deviation (% RSD) of the CF is calculated for GRO and surrogate.

10.4 The % RSD for all target analytes and surrogates must be ≤ 20 %. If the % RSD exceeds 20 % one of the following may be employed.

10.4.1 High and/or low points for poor responding or saturating analytes may be removed provided the ICAL still has a minimum of 5 levels and the low standard is at or below the RL.

10.4.2 Linear regression curve fitting provided the correlation of coefficient (r) ≥ 0.995 for the particular analytes(s).

10.4.3 Quadratic regression curve fitting provided the coefficient of determination (r^2) ≥ 0.990 and six (6) calibration levels are used for the particular analyte(s).

10.5 Following the analysis of an initial calibration, an ICV standard is analyzed. Acceptance criteria is ± 15% of the expected value (% drift).

10.6 Prior to analyzing samples, after every 10 samples, and at the end of a sequence, a CCV is analyzed. Acceptance criteria is ± 15% drift for 8015B and ± 20% drift for 8015C/D.

10.7 Refer to Section 13.0 for corrective action regarding out of control situations.

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11.0 ANALYTICAL PROCEDURE

11.1 RT standard and ICAL or CCV analyzed per Section 10.0.

11.2 Once calibration requirements are met, a matrix specific method blank is analyzed to verify that the analytical system is free of interferences.

11.3 Following the analysis of a method blank a matrix specific LCS is analyzed.

11.4 Refer to Section 13.0 for blank and LCS preparation and analysis.

11.5 After blank and LCS criteria are met samples may be analyzed. Samples are analyzed until 10 analyses are performed (blank, LCSs, and MS/MSDs count as analyses, rinses do not). After 10 analyses a CCV is analyzed. Sample analysis may continue as long as the CCV standard meets criteria in Section 8.0. The final analysis of each analytical batch must be a CCV standard.

The following illustrates an example analytical batch sequence:

Analytical batch sequence:

RT standard
ICAL or CCV
Method blank
LCS, LCSD
7 samples (including MS/MSD, sample/sample duplicate)
CCV standard
10 samples
CCV standard
etc.
end of run: CCV standard

NOTE: If the number of samples exceeds 20 in the analytical batch, a blank and LCS must be analyzed.

11.6 Water sample preparation: Allow sample to warm to ambient. Place vial in auto-sampler tray. Record sample pH after analysis.

11.7 Soil sample preparation (refer to Microbac SOP PAT01).

11.8 After sample analysis, the data system produces a quantitation report listing the concentration found in the sample. GRO is identified based on the retention times of 2-methylpentane(C_6) and 1,2,4-trimethylbenzene(C_{10}) in the window

defining standard. RT windows are needed to determine the GRO range. To calculate the windows the analyst is required to perform statistical operations on chromatograms from three consecutive days. The width of the RT windows for 2-methylpentane, surrogate chlorobenzene and 1,2,4-trimethylbenzene are calculated by multiplying the standard deviation of their peaks by three (3). The RT of GRO begins with the lower RT window limit for 2-methylpentane and ends with the upper RT limit for 1,2,4-trimethylbenzene. The GRO window is adjusted daily based on the retention times from the window defining standard. For GRO range C_6 to C_{12}, n-dodecane is used in place of 1,2,4-trimethylbenzene.

11.9 If the response for GRO exceeds the initial calibration range, the sample must be diluted. Dilutions should be prepared so that GRO is near the midpoint of the calibration. Water and mid-level soil dilutions are prepared by using syringes or pipets to transfer aliquots of sample into a volumetric flask containing DI water. Examples of water dilutions are presented below.

DILUTION	SAMPLE VOLUME (mL)	DI WATER VOLUME (mL)	FINAL DILUTION VOLUME (mL)
10x	5	45	50
50x	1	49	50
100x	0.5	49.5	50
1000x	0.05	49.95	50

Low-level soil dilutions are prepared by weighing an aliquot less than 5.00 g in a 40 mL VOA vial. Examples of low-level soil dilutions:

DILUTION	SAMPLE AMOUNT (g)	DI WATER VOLUME (mL)
2x	2.5	5
2.5x	2.0	5
5x	1.0	5

11.9.1 Low level soils collected via 5035A must utilize the mid-level aliquot for dilutions.

Mid-level soil dilutions are prepared by diluting an aliquot of the methanol extract in a volumetric flask. Examples of mid-level soil dilutions:

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DILUTION	METHANOL EXTRACT VOLUME (mL)	DI WATER VOLUME (mL)	FINAL VOLUME (mL)
50x	1	49	50
100x	0.5	49.5	50
500x	0.1	49.9	50
1000x	0.05	49.95	50

11.10 After the raw data is processed, it is uploaded into the LIMS.

11.11 The data is reviewed two times by the data review team; once for completeness and a second time for data validation.

Analyses performed using parameters in Table 2.

12.0 DETAILS OF CALCULATIONS

12.1 Calibration factor (CF) using external standard calibration:

$$CF = \frac{A_s}{C_s}$$

where:

A_s = peak area for the analyte or surrogate in the external standard
C_s = concentration of the external standard (ug/L)

NOTE: Surrogate response subtracted from GRO response

12.2 Mean calibration factor $(\overline{CF})$:

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

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12.3 Standard deviation(s):

$$s = \sqrt{\frac{\sum\left(x - \bar{x}\right)^2}{n-1}}$$

where:

x = CF

12.4 Percent relative standard deviation (%RSD):

$$\%RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:

s = standard deviation
$\bar{x}$ = mean calibration factor

12.5 Percent drift (% drift):

The percent (%) difference or drift is calculated using the formula:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = true concentration of the analyte or surrogate in the standard
C_x = measured concentration of analyte or surrogate in the standard

12.6 Concentration of analyte *(C_x)* in a sample:

$$C_x = \frac{(A_x)(D)}{(\overline{CF})}$$

where:

A_x = peak area for the analyte or surrogate
D = dilution factor for the samples (1/10 = 10)

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$\overline{CF}$ = mean CF for the analyte or surrogate from calibration curve
C_x = calculated concentration of the analyte or surrogate (ug/L)

12.7 Correction of soil wet weight concentration to dry weight (C_d):

$$C_d = \frac{(C_x)(100)}{(\%solids)}$$

where:

C_x = the uncorrected wet weight concentration
% solids = the percent solids content of the sample

12.8 Surrogate and LCS accuracy (% Recovery):

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration
$\%R$ = percent recovery

12.9 MS/MSD, LCS/LCSD, and sample/sample duplicate accuracy (%R) and precision (RPD):

%R for MS/MSD, LCS/LCSD:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration
$\%R$ = percent recovery

Relative percent difference (RPD):

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$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2} \right] 100$$

where:

C_1 = concentration of the first sample
C_2 = concentration of the second sample

12.10 Linear calibration calculations (external standard method)

12.10.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where,

y = response (area) = R
x = concentration = C_i
m and b = the slope and intercept from the regression equation

NOTE: For a given response ratio we can solve for C_i:

$$C_i = [R - b]/m$$

12.11 Quadratic calibration calculations (external standard method)

12.11.1 The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

$$y = ax^2 + bx + c$$

OR

$$ax^2 + bx + (c - y) = 0$$

12.11.2 Solving for x using the quadratic equation:

$$x = \frac{b \pm \sqrt{(b^2 - 4a(c - y))}}{2a}$$

where,

y = response (area) = R
x = concentration = C_i
a,b and c = the constants from the regression equation

NOTE: Use equations 12.11 or 12.12 to calculate the sample concentration, C_x, in 12.7.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 This section cites the procedures and analyses required to fully document the quality of data generated by the method. The required components of the laboratory's QA program and specific QC analyses are described in this section. For each QC analysis, the complete analytical procedure, the frequency of required analyses, and interpretation of results are specified.

13.2 Workgroups are analytical batches that contain RT standards calibration standards (ICAL, ICV, CCV), QC samples, and client samples.

13.3 Workgroups are comprised of:

- RT standards: used to define $C_6 - C_{10}$ range.
- ICAL/CCV standards: used to calibrate instrument or verify accuracy of the calibration curve
- ICV: standard from an alternate source used to verify accuracy of the calibration curve
- Method blank: verify system is free of contaminants and interferences
- LCS, LCSD verify accuracy of system
- MS/MSD: measure matrix effect of environmental sample on target analytes; measure precision
- Sample/sample duplicate: dual analysis of environmental system to measure precision
- Environmental sample: samples submitted for analysis

13.4 Following an initial calibration an ICV is analyzed. ICV results must be within ±15% drift. Failure of the ICV warrants corrective action including, but not limited to:

- reanalysis of ICV standard
- reanalysis of calibration curve
- preparation of new standards

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- instrument maintenance
- qualifying analytical results
- addressing issue(s) in a case narrative

13.5 Following an ICV a CCV is analyzed unless the analytical sequence is continued immediately following the ICAL. CCV results must be within ± 15% drift for 8015B and ± 20% drift for 8015C/D. Failure of the CCV warrants corrective action including, but not limited to:

- reanalysis of CCV standard
- reanalysis of calibration curve
- preparation of new standards
- instrument maintenance
- qualifying analytical results
- addressing issue(s) in a case narrative
- reanalysis of samples associated with non-compliant CCV

13.6 Following the CCV a matrix specific method blank is analyzed. GRO must be below the RL.

- Water blank preparation: 40 mL VOA vial is filled with UV-treated DI water. The vial is placed in the tray of the auto-sampler.
- Soil blank preparation: 5.00 g (±0.05 g) of reagent sand is weighed in a tared 40 mL VOA vial. 5 mL of DI water is added to the vial. The vial is placed on the auto-sampler and 5 mL of UV-treated DI water containing surrogate standard is added by the auto-sampler.
- Middle-level extraction blank: 5.00 g (±0.05 g) of sand is weighed into a tared vial. 10 mL of methanol is added and the vial is shaken. The contents are allowed to settle. A 50x dilution is performed and the extract is loaded on the auto-sampler.

13.7 Following the method blank a matrix specific LCS is analyzed. GRO % recovery must be within acceptance criteria set in Table 4. LCS results may exceed upper advisory limit providing GRO in associated samples is not detected (ND).

- Water and low-level soil LCS prepared per Section 7.0.
- Middle-level extraction LCS preparation: 5.00 g (± 0.05 g) of sand is weighed in a tared 40 ML VOA vial. 9.5 mL of methanol and 500 uL of LCS stock are added and the vial is shaken. The contents are allowed to settle. A 50x dilution is performed and the extract is loaded on the auto-sampler.

Corrective action for LCS/LCSD outlier include but are not limited to:

- reanalysis of calibration curve
- preparation of new standards
- instrument maintenance
- qualifying analytical results
- addressing issue(s) in a case narrative
- reanalysis of samples

13.8 MS/MSD analyses are performed every 20 samples provided a designated sample is provided else an LCS/LCS duplicate is analyzed.

13.9 MS/MSD preparation: Refer to Section 7.0 substituting sample for DI water and Table 4 for acceptance criteria.

13.10 Sample/sample duplicate: two samples are prepared and analyzed.

13.11 All data is scrutinized by the analysts for method specific compliance. Check lists are utilized and accompany each data batch (Figure 1).

13.12 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.12.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 4, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 4.

All data is scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)

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- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.12.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RAC, corrections, corrective action(s) and evidence of effectiveness.

13.12.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that may be invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.13 Daily QC forms are prepared with each analytical run and accompany all data packages. Computerized control charts are prepared from these daily summary forms which graphically illustrate Microbac's Quality Assurance Program.

13.14 Surrogate is added to all standards, QC samples, and environmental samples. Table 3 lists surrogate acceptance limits.

13.15 All QC samples are treated in the same manner as samples (ie: undergo same preparation and analytical procedures).

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13.16 RT windows are calculated as ± 3 standard deviations. RT Windows are centered each day using the CCV. If there is not enough data collected to generate RT Windows, the default window of .03 set by the EPA Method SW846 (8000B Sec. 7.6) is used. The default window is also used when the standard deviation of the three replicate retention time measurements for a compound = 0.000.

13.17 GRO is identified as a group of peaks within the carbon range C_6 to C_{10}. RT markers 2-methylpentane and 1,2,4-trimethylbenzene are used to delineate the range for GRO (n-dodecane C_{12}). RT windows for the markers are calculated (annually) by determining the standard deviation (SD) of the retention times obtained over a 72 hour period and multiplying the SD by three. The beginning of the RT window for 2-methylpentane and the ending of the RT window for 1,2,4-trimethylbenzene designated the chromatographic range of GRO. The RT windows are adjusted daily by the injection of the working RT standards. Quantitation of all standards, QC samples, and environmental samples utilizes the sum of the areas of the chromatographic peaks (minus the surrogate) within the RT windows.

13.18 Refer to Table 4 for QC criteria and corrective action.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review:

14.1.1 All data undergoes a 100 % primary review to ensure method and project specific compliance, reduce the data into reportable results, and generate appropriate QC forms. All items in Figure 1 (data review checklist) are reviewed and results are uploaded to the LIMS.

14.1.2 Following the primary review the data undergoes a 100 % peer review. All items in 14.1.1 are repeated by the peer. The peer review is performed by the supervisor or designee.

14.2 Data reporting:

14.2.1 Following peer review all uploaded results are reviewed, verified, and qualified.

Default reporting units:

- water sample: "ug/L"
- soil/sediment sample: "ug/Kg"

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- oil sample: "ug/Kg"

NOTE: Project specific QAPP may supercede the default reporting units. Soil/sediments reported on a dry weight basis.

14.2.3 All uploaded results are uploaded to a maximum number of significant figures dictated by the LIMS. The number of significant figures in the final report may vary per project requirements.

14.2.4 Dilution and sample matrix confirmation analyses are uploaded into the LIMS and per the client's request may be reported as separate analyses or combined (concatenated) into one set of results.

14.3 Quantitative results are generated by Hewlett-Packard Enviroquant software. A correction is utilized to subtract the area of the surrogate standard from the total GRO area.

14.4 Data may be reviewed by an analyst other than the primary analyst provided the reviewing analyst's initials are recorded on the "Data Checklist".

15.0 PREVENTIVE MAINTENANCE

15.1 The pressure of all gases is checked daily. Other maintenance performed as needed.

15.2 Maintenance is recorded in the instrument maintenance log book.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start

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date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 The waste streams are as follows:

- non-halogenated solvents, solid waste

16.3 Laboratory policies and procedures for management of hazardous waste are found in SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 *"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846"*, 3rd Edition:

5030B	December, 1996
5030C	May, 2003
5035A	July, 2002
8000B	December, 1996
8000C	March, 2003
8015B	December, 1996
8015C	February, 2007
8015D	June, 2003

17.2 AFCEE QAPP, Version 3.0, March 1998

17.3 AFCEE QAPP, Version 3.1, August 2001

17.4 AFCEE QAPP, Version 4.0, February 2005

17.5 Microbac SOP PAT01 "Methods 5030B and 5035 Purge and Trap for Volatile Organics"

17.6 Microbac SOP 45 "Method Validation Procedures"

17.7 Microbac SOP 41 "Manual Integration of Chromatographic Peaks"

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17.8 Microbac SOP 33 "Laboratory Waste Management"

17.9 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.10 Microbac SOP GC-CAPA "Corrective Action / Preventative Action: Initiating, Tracking, and Monitoring"

17.11 Microbac SOP GP-RCA "Root Cause Analysis"

17.12 "Leaking Underground Fuel Tank (LUFT) Field manual", State Water Resources Control Board, State of California, Sacramento, CA, May 1998

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Appendix I
California Gas Chromatograph Method

1. For any samples from California or North Carolina, the California Gas Chromatograph Method, Eisenberg, D. M. and others, 1985, Guidelines for addressing fuel leaks will be followed.

2. A 10% acceptance criteria for continuing calibration is used rather than 15%.

Appendix II
Navy Clean

For LCS % recovery on any Navy Clean project, Microbac will adhere to the 100% acceptance limits requirements for the specified compounds of interest (short list) as per the Navy IRCDQM Appendix C 1999.

Appendix III
South Carolina Requirements

The following apply to all samples analyzed via 8015C for the state of South Carolina.

All soil samples received in the laboratory from South Carolina require analysis using 5035. Client notification will be performed if soil samples are not collected accordingly. South Carolina permits the freezing of soils samples in lieu of sodium bisulfate preservation for 5035 provided this occurs within 48 hours of collection. Frozen soils have a fourteen day hold time.

Retention time windows are determined with each initial calibration and are not adjusted daily by the working retention time standard. The daily retention time markers must meet retention time windows determined at the time of the initial calibration or an initial calibration must be performed.

The following table lists LCS control limits for analyses associated with South Carolina samples:

PARAMETER	WATER LCS	SOIL LCS	MID-SOIL LCS
GRO	83-124	78-118	80-125

When evaluating calibration curves, the following acceptance criteria will only be used to ascertain linearity:

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1) Average response factor for GRO if %RSD < 20%
2) If GRO %RSD > 20%, linear regression used providing r > 0.995

NOTE: Non-linear curve fitting (i.e.: quadratic regression) will not be used.

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Table 1
QA Objectives for Method 8015B/C/D

MATRIX	ACCURACY (% RECOVERY)*	PRECISON (% RPD)*	MDL***	REPORTING LIMIT**	LCS, MS/MSD TRUE VALUE
Water	85 - 120	20	45 ug/L	100 ug/L	900 ug/L
Soil	85 – 120	30	45 ug/Kg	100 ug/Kg	900 ug/Kg

* Evaluated annually from laboratory control samples (actual control limits may vary)
** Nominal reporting limit (project specific reporting limits may vary)
*** Denotes observed MDL (verified annually); MDL study performed per 40CFR, Part 136.

Table 2
Recommended Parameters *

LSC	
purge	40 mL/min. for 9 minutes
dry purge	1 minute
desorb preheat	245° C
desorb	250° C for 1 minute
bake	260° C for 12 minutes
valve, lines, mount	120° C
bake gas bypass (BGB)	OFF
GC	
injection port temperature	N/A
detector temperature	250° C
oven program: initial temperature	50° C
hold (initial)	9 minutes
ramp #1	10° C/minute to 230° C hold for 2 minutes
AUTOSAMPLER	
soil probe, transfer line	110° C
upper soil valve	95° C
purge preheat (soil only)	40° C for 2 minutes
purge temperature (soil only)	40° C
soil purge time	9 minutes

* Recommended parameters; may be adjusted to improve performance.

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Table 3
Surrogate Standard Recovery Limits

SURROGATE	8015B WATER % RECOVERY	AFCEE WATER % RECOVERY	8015B SOIL % RECOVERY	AFCEE SOIL % RECOVERY
chlorobenzene	74-138	74-138	64-148	64-148

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Table 4
Quality Control Criteria
Gasoline Range Organics (GRO Analysis)
Method 8015B/C/D

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration (ICAL)	Initially and upon failure of two consecutive CCV's	% RSD ≤ 20%	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1)
Initial Calibration Verification (ICV)	After each initial calibration	± 15% of expected value or project specific criteria	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing Calibration Verification (CCV)	Initially, every 10 samples, and at end of sequence	8015B: ± 15% of expected value 8015C/D: ± 20% of expected value	Re-analyze CCV; upon second failure, repeat initial calibration (1)
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	GRO < ½ reporting limit	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative (1)
Laboratory Control Sample/Laboratory Control Sample duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch.	Table 1 (1,2)	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative (3)
Matrix Spikes/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Table 1 (1,2)	Qualify data and/or address in narrative.
Sample/Sample Duplicate	One per matrix/batch; maximum of 20 samples per batch.	Table 1 (1,2)	Qualify data and/or address in narrative.
Surrogate spike	Every sample, standard, and quality control sample	Table 1 (1)	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative (3)

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

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Figure 1

Checklist ID: 28099

Microbac Laboratories Inc.

Data Checklist

Date: 02-MAY-2008
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Run Log ID:	
Calibration/Linearity	
ICV/CCV	
ICB/CCB	
ICSA/ICSAB	
CRI	
Blank/LCS	
MS/MSD	
Post Spike/Serial Dilution	
Upload Results	
Data Qualifiers	
Generate PDF Instrument Data	
Sign/Annotate PDF Data	
Upload Curve Data	
Workgroup Forms	
Case Narrative	
Client Forms	
Level X	
Level 3	
Level 4	
Peer Reviewer	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Check the reasonableness of the results	

Primary Reviewer: Secondary Reviewer:

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Figure 2
VOA Preservation and Extraction Log

Microbac Laboratories Inc.
VOA Preparation/Preservation/Extraction Log

Workgroup (AAB#):WG270339
Method:8260
Reagent ID:RGT10001

Analyst:DGB
Run Date:06/05/2008 09:50

SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total Wt	Sample Wt	Water	MeOH	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.86			30.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	B	04/20/08 11:25	06/05/08 09:51	77.08	5.12	30.1	24.98	5		5	5
L08020355-09	C	04/20/08 11:25	06/05/08 09:52	77.08			25.08		10	15.748816	5

Comments:
1 = improperly sealed cap
2 = preserved out of hold
3 = effervesced
4 = preserved with NaHSO4
5 = preserved by freezing
6 = preserved in field

Analyst: Douglas Butcher

VOA_EXT - Modified 05/22/2008
PDF ID: 1108658
Report generated: 06/05/2008 09:53

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STANDARD OPERATING PROCEDURE
ORGANIC ANALYSIS OF PESTICIDES
METHOD 8081A/8081B

Issue/Implementation Date: 15 October 2010

Last Review Date: 15 October 2010

Microbac Laboratories, Inc.
Ohio Valley Division
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Approved By:

Michael D. Cochran, Semi-Volatile Supervisor — Date 10/5/10

Leslie S. Bucina, Quality Assurance Officer — Date 10/5/10

David E. Vandenberg, Managing Director — Date 10/6/10

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1.0 SCOPE AND APPLICATION

1.1 This method describes the procedures used to analyze chlorinated pesticides in a variety of sample matrices. This method uses fused silica columns in order to separate target compounds. Tables 1 and 2 lists the compounds that may be determined by this method. This method follows the procedures outlined in SW-846 Methods 8081A or 8081B. LIMS products will be used to designate the specific method to be followed for each sample.

2.0 SAFETY PRECAUTION

Analysts using this method must be aware of certain hazards that are associated with performing the analysis.

2.1 Chemical Hazards

2.1.1 Several organic solvents are used for the preparation of standards and for the dilution of samples. These solvents include hexane, methylene chloride, methanol, benzene, and toluene. Each of these solvents are either carcinogenics, suspected carcinogenics or attack organs of the human body. Extreme care must be exercised when these compounds are being used. Gloves must be worn and proper ventilation must be in place before using any of the above mentioned solvents. All work with these solvents must be performed in a fume hood that has had the face velocity verified within the past year.

2.1.2 Concentrated organic standards are used to calibrate the instruments. Many of these chemicals have been found to cause cancer and must be handled with extreme care. Gloves must be worn while these compounds are being used. These compounds must be handled under a fume hood to prevent personnel exposure.

2.2 Thermal Hazards

Several zones on the GC are heated to high temperatures. Care must be exercised when working around these areas to avoid severe burns to the skin.

2.3 Broken Glassware Hazards

All dilutions that require the use of glassware must be made with care to reduce the possibility of cuts from broken glass. All broken or defective glassware must be disposed of in the broken glass container located in the laboratory.

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2.4 Radioactive Hazards

Electron capture detectors are a sealed source radioactive ionization detector. Because it is a sealed source, the radioactive Ni 63 is not exposed to the environment and therefore poses little danger to the analyst. Wipe tests are performed on each detector on a semi-annual basis to check for Ni 63 contamination on the outside of the detector housing. These samples are sent away to an independent laboratory where they are analyzed for Ni 63 contamination. If detectors pass these wipe tests then the detectors will remain in service provided they are still operating within specifications. If the detectors have been leaking during the previous six months, then the detector must be removed from service immediately, and returned to Agilent detector exchange program as soon as possible. Extreme care must be taken when handling a leaking detector to prevent personal exposure to radioactive materials.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Sample size requirements are determined by the matrix type of the sample.

3.1.1 Water samples require a volume of three (3) liters per sample. This ensures the extraction laboratory has sufficient sample for the extraction of a matrix spike, and a matrix spike duplicate for the extraction batch. This amount also allows for the sample to be re-extracted should the need arise.

3.1.2 Soil extractions require at least 90 grams of sample. This ensures the extraction laboratory has sufficient sample for the extraction of a matrix spike, and matrix spike duplicate for the extraction batch. This amount also allows for the sample to be re-extracted should the need arise.

3.2 Sample collection must be performed according to the outline set forth in SW-846. This gives the laboratory a sample representative of the sampling site.

3.3 After the samples have been extracted, they must be stored at 4° C ± 2° C until the instrumental analysis can proceed. The samples must be analyzed within 40 days from the day of the extraction.

4.0 METHOD PERFORMANCE

4.1 Table 1 summarizes the performance data for water analysis; Table 2 summarizes performance data for soil/solid waste analysis; These tables include the analyte

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list, ranges for accuracy and precision, nominal laboratory reporting limits (RL), and the current verified detection limit(s).

4.2 The laboratory performed an initial assessment of the method detection limits (MDL) using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The limit(s) of detection (LOD), or verified MDL, are presented in Tables 1 and 2 and were established using verification procedures outlined in SOP 45.

4.4 The limit(s) of quantitation (LOQ) are the nominal laboratory reporting limit(s) (RL) and were established as per SOP 45.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. Going forward, the laboratory will use results from laboratory control samples (LCS) to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 All possible measures are taken to eliminate interferences; however some samples have high levels of material that co-extract and make it virtually impossible to completely remove all interferences from the extracts. All samples, blanks, LCS, matrix spikes, and matrix spike duplicates in a given batch undergo identical clean-up procedures.

5.2 Analysis of the MS/MSD samples for each batch is used in determining the extent of sample matrix interference only. These samples are not used as laboratory control to determine when a batch of samples need to be re-extracted and re-analyzed. If the recoveries for any one target compound in the MS or

MSD is outside the statistically calculated limits shown in Tables 1 and 2 then the analyst must mention the possibility of sample matrix interference in the case narrative and what effect if any these interferences may have on the data.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph system

6.1.1 Gas chromatograph system: Agilent 6890 series

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- splitless injection port system
- electronic pressure control
- HP 7673 or 6890 autosampler and controller
- capillary column fittings and reducing nuts

6.1.2 Restek RTX – CLPesticides megabore column 30 m x 0.53 um I.D. and 0.5 um film thickness.

6.1.3 Restek RTX – CLPesticides II megabore column 30 m x 0.53 um I.D. and 0.42 um film thickness.

6.1.4 Data system: Windows-based Agilent MS Chemstation

- Agilent Environquant software, Version C.00.02 or newer

6.2 Syringe - 10 uL (standard 5 uL autosampler), 100 uL, 500 uL, 1000 uL Hamilton Syringes or equivalent

6.3 Volumetric flasks, Class A - 10 mL to 1000 mL

6.4 Sample vials - glass with Teflon-lined screw caps or crimp tops. (2 mL)

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Solvents: Hexane, Pesticide grade or higher quality

7.2 Pesticide Stock Standards - A 20 ug/mL stock standard is prepared from Accustandard M-8081-SC or equivalent. This ampule contains 1.0 ml of 1000 ug/mL of all of the pesticides compounds except toxaphene. Exactly 1.0 ml of this standard is transferred to a 50 ml volumetric flask containing approximately 40 ml of pesticide grade hexane. Dilute the stock to the mark with pesticide grade hexane and invert three times to ensure proper mixing. The stock standard is now ready for use to prepare daily analytical standards. Secondary stock standards are prepared in an identical manner to that described above except that the high level source is Ultra Scientific standard PPM-808C or equivalent. All standards have a 6-month expiration date. All standards are stored in amber bottles at ± -10° C.

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7.3 Surrogate Stock Standard - A 20 ug/mL surrogate stock standard is prepared from Accustandard # CLP-032-R or equivalent. This ampule contains 1.0 ml of 200 ug/mL of each of the two surrogate compounds. Exactly 1.0 ml of this standard is transferred to a 10 ml volumetric flask containing approximately 8 ml of pesticide grade hexane. Dilute the stock to the mark with pesticide grade hexane and invert three times to ensure proper mixing. The stock surrogate standard is now ready for use to prepare daily analytical standards. Secondary surrogate stock standards are prepared in an identical manner to that described above except that the high level source is Supelco standard # 4-8460 or equivalent.

7.4 Working Pesticide Standard - The working pesticide standard is prepared at 200 ug/L by transferring 1.0 ml of the stock standard and 1.0 ml of stock surrogate standard into a 100 ml volumetric flask containing ~80 ml of pesticide grade hexane. Hexane is added to the mark and the flask is inverted three times to mix properly. This yields a concentration of 200 ug/L pesticides and 200 ug/L surrogates.

7.5 Toxaphene Stock Standard:

The toxaphene stock is prepared from 1000 ug/mL high level standard solution purchased from Supelco (4-8103) or equivalent. The stock is prepared at a level of 10 ug/mL by transferring 1.0 ml of the high level standard into a 100 ml volumetric flask, which contains approximately 80 ml of pesticide grade hexane. Fill the flask to the mark with hexane and invert three times to ensure proper mixing. This stock is now ready for use to prepare daily calibration standards.

Daily check standard stocks are prepared in a similar manner except that the high level source is Accustandard (P-093S-H-10X) or equivalent source.

7.6 Calibration Standards – The lowest calibration standard is at a concentration at or below the reporting limit.

7.6.1 Pesticides - The pesticide calibration standards are prepared from stock standards at 6 different levels. These are made by diluting the working standard using the following scheme:

<u>Pesticides Calibration Standards</u>

#6 - 200 ug/L (transfer working solution)
#5 - 100 ug/L (300 ul stock: 300 ul hexane)
#4 - 20 ug/L (100 ul stock: 900 ul hexane)
#3 - 10 ug/L (50 ul stock: 950 ul hexane)
#2 - 4 ug/L (200 ul standard #3: 300 ul hexane)
#1 – 1 ug/L (100 ul std #3: 900 ul hexane)

7.6.2 Toxaphene - The toxaphene calibration standards are prepared from a 10,000 ug/L stock solution at the following levels by diluting the stock solution and standard #4 using the following scheme:

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Toxaphene Calibration Standards

#5 - 2.0 ug/mL (200 ul stock: 800 ul hexane)
#4 - 1.0 ug/mL (100 ul stock: 900 ul hexane)
#3 - 0.5 ug/mL (50 ul stock: 950 ul hexane)
#2 - 0.25 ug/mL (100 ul standard #4: 300 ul hexane)
#1 - 0.1 ug/mL (100 ul standard #4: 900 ul hexane)

The secondary source standards are prepared at Level #3 of the initial calibration curve.

7.7 Endrin/DDT breakdown standard: A 500 ug/mL Endrin/DDT mix is purchased (Supelco 48282) and 1 mL is diluted in a 100 mL volumetric flask with hexane to yield a 5 ug/mL intermediate standard. 1 mL of the intermediate standard is diluted with hexane in a 100 mL volumetric flask to yield a working concentration of 0.05 ug/mL.

8.0 CALIBRATION PROCEDURE

8.1 Recommended Gas Chromatograph Conditions

Columns -	See Section 6.1
Temp Prog -	110° C for 1.0 min
	25° C/min to 150° C
	then 12° C/min to 260° C
	then 15° C/min to 320° C for 5 min
Carrier Gas -	Helium at 5.4 ml/min
Ionizing Gas -Ar/Me	@ 15 & 25 ml/min
ECD Temp -	325° C
Injector Temp -	225° C
Injection Amount -	1 ul

8.2 Run the following series of calibration standards after a hexane blank, used to assess system cleanliness:

Endrin/DDT breakdown check

#6 Pesticide Standard
#5 Pesticide Standard
#4 Pesticide Standard
#3 Pesticide Standard
#2 Pesticide Standard
#1 Pesticide Standard
2nd Source Calibration verification Standard for Pesticides

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2000 ug/L Toxaphene Standard
1000 ug/L Toxaphene Standard
500 ug/L Toxaphene Standard
250 ug/L Toxaphene Standard
100 ug/L Toxaphene Standard
2nd Source Calibration Verification Standard for Toxaphene

8.2.1 This sequence must be run whenever any major changes are made to the instrument. The curve must have a %RSD of less than or equal to 20, using the equations in Section 12.5. If the % RSD is greater than 20 the analyst may quantitate samples by plotting against the 6 point curve using linear regression (y=mx+b) provided the coefficient of correlation is 0.995 or greater (Section 12.6). If the initial calibration fails to meet criteria, the GC must be inspected for problems. After any problems have been identified and corrected, the initial calibration is rerun.

8.3 DDT and Endrin Breakdown

8.3.1 Every 12 hours the individual channels must be monitored for elevated active sites. Any active sites within the injector ports will cause these compounds to breakdown into similar compounds. DDT will breakdown into DDE or DDD. Endrin will breakdown into endrin aldehyde and endrin ketone. The % breakdown for each channel is calculated by comparing areas for Endrin and DDT with their corresponding breakdown products.

% DDT breakdown =

$$\left[\frac{area(DDE)+area(DDD)}{area(DDT)+area(DDE)+area(DDD)}\right]100$$

% Endrin breakdown =

$$\left[\frac{area(endrin)+area(endrinketone)}{area(endrin)+area(endrinald)+area(endrinketone)}\right]100$$

If the breakdown for either compound is greater then 15% then corrective action must be taken to get the breakdown under 15% before the analyst may proceed. Corrective action may take the form of changing the glass liners, or in more extreme cases of degradation the metal adapter may be changed to alleviate the breakdown. The breakdown must be brought under 15 % in order to continue with the analysis.

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8.4 Calibration Verification

8.4.1 Initial calibration verification (ICV): The second source standards must have a % difference or % drift less than 20, using the equation in Section 12.4. If the standard fails, a second ICV from the same source may be analyzed. Upon the second failure, instrument maintenance and/or recalibration must be performed.

8.4.2 Continuing calibration verification (CCV): A continuing calibration check standard must be run for each analyte of interest initially and after a series of 10 samples have been run or every 12 hours, whichever is more frequent, to ensure the instrument response has not changed. Using the calculation from Section 12.4, the percent difference for each compound of interest must be less than 20 percent for the quantitation column and less than 20 percent for confirmation of a tentatively identified compound. If a CCV fails this criteria, a second CCV from the same source may be analyzed. Upon the second failure, instrument maintenance and/or recalibration must be performed and any samples bracketed by a failing CCV must be reanalyzed. See also Section 13.7.4.

8.4.3 Certain projects (e.g. Ohio VAP) may require the use of Method 8081A; if this is the case, the ICV and CCV standards must have a % D ≤15.

8.4.4 After the endrin/DDT breakdown check, initial calibration and/or CCV, the sequence of the sample run proceeds as follows:

10 samples
Pesticide CCV @ 20 ug/L
Toxaphene CCV @ 250 ug/L
10 samples
Endrin/DDT @ 50 ug/L
Pesticide CCV @ 10 ug/L
Toxaphene CCV @ 500 ug/L
10 samples – etc.

Batch QC samples are to be included in the count of ten (10) injections between CCV standards, but solvent blanks injected as contamination checks do not need to be counted.

8.5 All of the initial calibration, ICV and CCV criteria must also be met for compounds reported from the confirmation column.

9.0 SAMPLE PREPARATIONS

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9.1 All samples submitted to the laboratory for analysis under this method must first be extracted before the analysis can begin. Methods found in SW-846 detail extraction procedures suitable for a wide variety of sample matrices.

9.2 Soil samples submitted for analysis must undergo solvent extraction by sonication. This procedure is detailed in SOP EXPO2. This SOP is found under separate cover.

9.3 Water samples submitted for analysis must undergo solvent extraction by separatory funnel. This procedure is detailed in SOP EXPO1. This SOP is found under separate cover.

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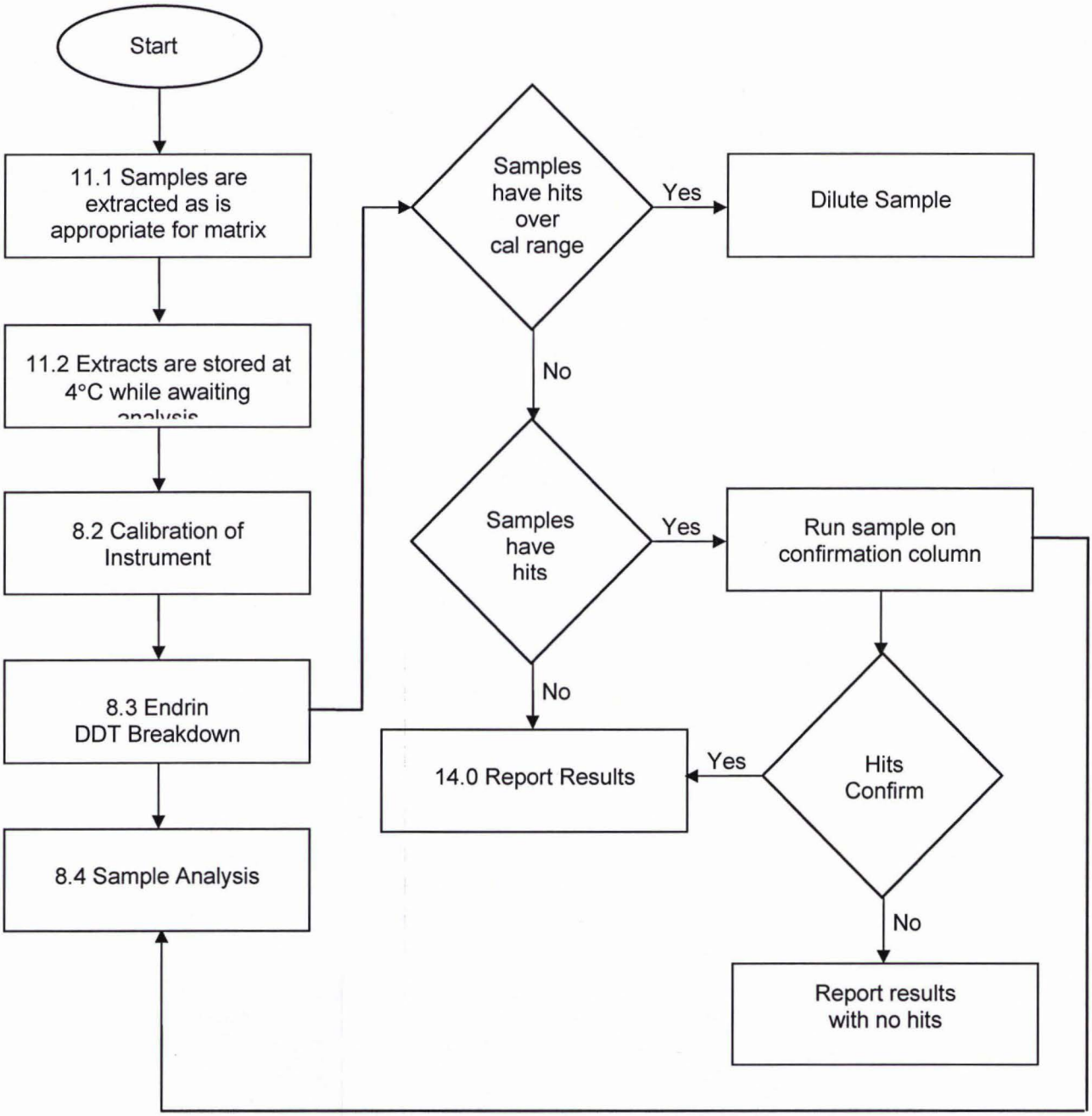
10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



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11.0 ANALYTICAL PROCEDURES

11.1 The instrument(s) are calibrated and the analytical sequence proceeds as in Section 8.0 of this document.

11.2 A report of ND (not detected) is a form of quantitation and must therefore adhere to these rules. The list of compounds requested by the client will constitute the target compounds to be analyzed. Calibration standards which fail criteria for compounds not on the target compound list will not be considered when interpreting the check standard. Only target compounds as defined by the clients request will be reported.

11.3 A compound is considered tentatively identified if a peak is found within the calculated daily retention time window. A compound is considered positively identified if a peak is found within the calculated daily retention time window on the confirmation column also.

Multi-response compounds such as toxaphene will have retention time windows calculated for the selected target peaks, however, the identification of these compounds is based primarily on pattern recognition.

11.4 Quantitation of multi-response compounds such as toxaphene is performed by selecting at least three (3) major peaks and treating them as single components, using baseline-to-valley integration. Each peak must pass all calibration criteria listed in Sections 8.2 and 8.4 of this document. Quantitation of an unknown sample is the average of the concentrations of each of the major peaks selected. A major peak is one that is at least 25% of the largest peak in the multiresponder.

11.5 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Dilutions are prepared by using syringes to transfer aliquots of extract into appropriate amounts of solvent in autosampler vials. Examples are presented below:

Dilution	Amount sample extract	Amount Hexane	Final dilution volume
2x	200 uL	200 uL	400 uL
5x	100 uL	400 uL	500 uL
10x	100 uL	900 uL	1000 uL
20x	50 uL	950 uL	1000 uL

Higher dilutions are prepared by performing serial dilutions, e.g. for a 100x dilution, a 10x dilution is diluted again by a factor of 10.

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11.6 The raw data is processed using the Chemstation software and the data is uploaded into the LIMS.

11.7 The data is reviewed two times by the data review team; once for completeness and a second time for data validation.

12.0 DETAILS OF CALCULATION

12.1 Calibration factor (CF) from external standard calibration:

$$CF = \frac{A_s}{C_s}$$

where:

A_s = Peak area for the analyte or surrogate in the external standard
C_s = Concentration of the external standard (ug/L)

12.2 Mean calibration factor (CF) = $\overline{CF}$

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

12.3 Calculation of standard deviation. The standard deviation is calculated using the formula:

$$s = \sqrt{\frac{\sum \left(x - \bar{x}\right)^2}{n-1}}$$

12.4 The % difference or drift is calculated using the formula:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

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12.5 The % RSD is calculated using the formula:

$$RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:

s = Standard deviation
x = Average

12.6 Coefficient of correlation

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - (\sum X)^2 / n\right)\left(\sum Y^2 - (\sum Y)^2 / n\right)}}$$

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.7 The LCS recovery is calculated as follows:

$$\%R = \left(\frac{C_x}{C_T}\right)100$$

where:

C_x = the concentration of the analyte in the LCS.
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.8 Calculation of % Recovery

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

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C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.9 Calculation of RPD

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2} \right] 100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.10 Calculation of concentration of analyte using calibration factor

$$C_x = \frac{(A_x)(D)}{(CF)}$$

where:

A_x = Peak area for the analyte or surrogate
D = Dilution factor for the samples (1/10 = 10)
CF = Mean CF for the analyte or surrogate from
C_x = Calculated concentration of the analyte or surrogate (ug/L)

12.11 Linear calibration calculations (external standard method)

12.11.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response(area) = R
x = Concentration = C_i

And m and b are the slope and intercept from the regression equation
For a given response ratio we can solve for C_i:

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12.11.2 $C_i = [R - b]/m$

Use equations 12.4 or 12.5 to calculate the sample concentration, C_x.

12.12 Solving for the concentration in water samples using linear regression.

For a given concentration, compute the unknown, C_x

$$C_x = (C_i)\left(V_f / V_i\right)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
D = dilution factor
C_x = concentration in the sample (ug/L)

12.13 Solving for the concentration in soil samples using linear regression:

$$C_x = (C_i)\left(V_f / W_i\right)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = initial sample weight (g)
D = dilution factor
C_x = concentration of the sample (ug/kg) (as received)

13.0 QUALITY CONTROL (QC) REQUIREMENTS

The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), laboratory control samples (LCS), matrix spikes (MS), matrix spike duplicates (MSD), laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in Section 8.0 for initial calibration, initial calibration verification (ICV) using a second source, and continuing calibration verification (CCV), and are included in the overall review

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process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 4.

13.1 A batch is defined as a group of samples which are extracted together. A batch contains a maximum of 20 samples. With each batch of samples extracted, a laboratory control sample (LCS) and a method blank must also be extracted. Each batch should contain a matrix spike (MS) and matrix spike duplicate (MSD). All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch. The LCS, MS and MSD are spiked so as to yield a concentration that fall near the mid point of the curve.

(**NOTE**: Some projects require an LCS duplicate (LCSD). Consult the project QAPP for guidance).

13.2 Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Table 1 and Table 2 are used in the absence of a project QAPP, or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare a nonconformance report (NCR), and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare a (NCR) to be reviewed by the department supervisor. Corrective action will consist of re-extraction and re-analysis of the affected samples for, at a minimum, the COC for which a result was derived outside control limits, unless the client's representative and the quality assurance officer (QAO) approve of another course of action.

13.3 The method blank cannot contain amounts of any target analytes which are over the reporting limits (RL). If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL for the presents of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch. Some projects require that blanks are evaluated to a concentration of ½ the RL, consult the specific QAPP for guidance.

13.4 In order to monitor the extraction efficiency in each sample, a surrogate solution containing 2,4,5,6-tetrachloro-m-xylene and decachlorobiphenyl is added to each sample in the extraction batch. The recoveries for at least one of these surrogates must fall within the limits given in Table 1 and Table 2. If any individual sample has

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both surrogates outside the given limits, or less than 10% recovery for either surrogate, then the sample must be re-extracted.

(**NOTE**: Ohio VAP requires that all surrogates be within the acceptable limits for batch QC samples and field samples, except in cases of obvious matrix interference, and the run may not be reported if the method blank or LCS have any surrogates out.)

13.5 Compounds are identified by their peak retention time on the GC. Retention time windows are needed to evaluate samples in order to determine if a given peak represents an actual pesticide. Retention time windows are performed at method setup and repeated whenever major maintenance is performed. To calculate the window the analyst is required to perform statistical operations on chromatograms from 3 consecutive days. The retention time window is calculated by multiplying the standard deviation of each peak by 3. This gives the range that a particular peak may be identified as a pesticide. Furthermore because the laboratory is confirming all pesticide analysis on a second column of different polarity, the pesticide must also be identified on the second column. All multiresponders (toxaphene and chlordane) do not need to have a 2nd column confirmation, pattern recognition is sufficient. If the % difference of the results between the two columns is more than 40%, report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-affected result and flag the data accordingly. If the % difference is < 40%, report the higher of the two results, unless the project QAPP specifies otherwise.

The second column must meet all batch QC criteria for compounds requiring confirmation.

13.6 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of SOP LQAP (Laboratory Quality Assurance Program), SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and SOP GP-RCA (Root Cause Analysis).

13.6.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 4 does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Table 4.

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All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1).

A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.6.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per SOP GP-CAPA. The corrective action process includes a root cause analysis as per SOP GP-RCA, corrections, corrective action (s) and evidence of effectiveness.

13.6.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

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14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Prior to data entry into the LIMS (either manual or automatic), all data must undergo two (2) levels of review in the department. The primary review is performed by the analyst and the secondary review is performed by either the department supervisor (or a designee) or another qualified analyst.

14.2 Data Reporting

The reporting requirements depend upon the need of the client. Microbac offers four levels of data reporting which are described in some detail below.

14.2.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.2.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.2.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.2.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

14.2.5 Results for water samples are reported in ug/L to three significant figures.

14.2.6 Results for soil samples are reported in ug/kg, or mg/kg, on a dry weight basis to three significant figures.

15.0 PREVENTIVE MAINTENANCE

In order to minimize the downtime of the instrumentation, preventive maintenance is performed on a routine basis. The injection port liners and septa are changed regularly. Additionally, from time to time when the peak shape of the standards in the chromatograph is deformed, the front portion of the analytical column is clipped to improve performance. The ECD's are changed out when the calibration of the instruments becomes increasingly difficult.

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16.0 WASTE MANAGEMENT AND POLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutant Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule.

17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8081A Revision 1, December 1996.

17.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8081B, February 2007.

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17.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000B, Revision 2, December 1996.

17.5 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000C, Revision 3, March 2003.

17.6 Microbac SOP LQAP, "Laboratory Quality Assurance Plan"

17.7 Microbac SOP45, "Standard Operating Procedures for Method Validation Procedures"

17.8 Microbac SOP GP-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.9 Microbac SOP GP-RCA, "Root Cause Analysis"

17.10 Microbac SOP33, "Laboratory Waste Management"

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TABLE 1
MICROBAC's QA OBJECTIVES AND ANALYTICAL METHODS FOR ORGANOCHLORINE PESTICIDES ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	VERIFIED MDL (ug/L)	REPORTING LIMITS (ug/L)
Aldrin	309-00-2	8081A/8081B	42-122	0-30	0.01	0.05
alpha-BHC	319-84-6	8081A/8081B	50-130	0-30	0.01	0.05
beta-BHC	319-85-7	8081A/8081B	50-130	0-30	0.01	0.05
delta-BHC	319-86-8	8081A/8081B	45-140	0-30	0.01	0.05
gamma-BHC (Lindane)	58-89-9	8081A/8081B	40-135	0-30	0.01	0.05
alpha-Chlordane	5103-71-9	8081A/8081B	65-130	0-30	0.01	0.05
gamma-Chlordane	57-74-9	8081A/8081B	60-135	0-30	0.01	0.05
4,4'-DDD	72-54-8	8081A/8081B	50-150	0-30	0.01	0.05
4,4'-DDE	72-55-9	8081A/8081B	50-145	0-30	0.01	0.05
4,4'-DDT	50-29-3	8081A/8081B	45-150	0-30	0.01	0.05
Dieldrin	60-57-1	8081A/8081B	60-140	0-30	0.01	0.05
Endosulfan I	959-98-8	8081A/8081B	45-120	0-30	0.01	0.05
Endosulfan II	33213-65-9	8081A/8081B	30-130	0-30	0.01	0.05
Endosulfan sulfate	1031-07-8	8081A/8081B	55-135	0-30	0.01	0.05
Endrin	72-20-8	8081A/8081B	55-140	0-30	0.01	0.05
Endrin aldehyde	7421-93-4	8081A/8081B	40-135	0-30	0.01	0.05
Endrin ketone	53494-70-5	8081A/8081B	50-140	0-30	0.01	0.05
Heptachlor	76-44-8	8081A/8081B	40-130	0-30	0.01	0.05
Heptachlor epoxide	1024-57-3	8081A/8081B	60-130	0-30	0.01	0.05
Methoxychlor	72-43-5	8081A/8081B	55-150	0-30	0.01	0.05
Toxaphene	8022-04-6	8081A/8081B	NA	NA	0.3	1.0
2,4,5,6-tetrachloro-m-xylene (surr)	877-09-8	8081A/8081B	20-180	--		--
Decachlorobiphenyl (surr)	2051-24-3	8081A/8081B	25-140	--		--

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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TABLE 2
MICROBAC's QA OBJECTIVES AND ANALYTICAL METHODS FOR ORGANOCHLORINE PESTICIDES ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	VERIFIED MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)
Aldrin	309-00-2	8081A/8081B	45-140	0-30	0.33	1.65
alpha-BHC	319-84-6	8081A/8081B	50-145	0-30	0.33	1.65
Beta-BHC	319-85-7	8081A/8081B	55-130	0-30	0.33	1.65
delta-BHC	319-86-8	8081A/8081B	55-130	0-30	0.33	1.65
gamma-BHC (Lindane)	58-89-9	8081A/8081B	50-135	0-30	0.33	1.65
alpha-Chlordane	5103-71-9	8081A/8081B	65-130	0-30	0.33	1.65
gamma-Chlordane	57-74-9	8081A/8081B	55-135	0-30	0.33	1.65
4,4'-DDD	72-54-8	8081A/8081B	40-140	0-30	0.33	1.65
4,4'-DDE	72-55-9	8081A/8081B	70-140	0-30	0.33	1.65
4,4'-DDT	50-29-3	8081A/8081B	50-150	0-30	0.33	1.65
Dieldrin	60-57-1	8081A/8081B	65-125	0-30	0.33	1.65
Endosulfan I	959-98-8	8081A/8081B	30-130	0-30	0.33	1.65
Endosulfan II	33213-65-9	8081A/8081B	35-140	0-30	0.33	1.65
Endosulfan sulfate	1031-07-8	8081A/8081B	55-135	0-30	0.33	1.65
Endrin	72-20-8	8081A/8081B	60-140	0-30	0.33	1.65
Endrin aldehyde	7421-93-4	8081A/8081B	35-145	0-30	0.33	1.65
Endrin ketone	53494-70-5	8081A/8081B	60-130	0-30	0.33	1.65
Heptachlor	76-44-8	8081A/8081B	50-140	0-30	0.33	1.65
Heptachlor epoxide	1024-57-3	8081A/8081B	50-120	0-30	0.33	1.65
Methoxychlor	72-43-5	8081A/8081B	55-145	0-30	0.33	1.65
Toxaphene	8022-04-6	8081A/8081B	25-138	0-30	16.7	33
2,4,5,6-tetrachloro-m-xylene (surr)	877-09-8	8081A/8081B	39-130	--		--
Decachlorobiphenyl (surr)	2051-24-3	8081A/8081B	33-143	--		--

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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TABLE 3
Example Retention Times

Compound Name	Approximate Retention Time	
	RTX-CLP	RTX-CLP II
Aldrin	9.91	10.98
Alpha-BHC	8.06	8.75
Beta-BHC	9.12	9.87
Delta-BHC	9.68	10.32
Gamma-BHC	8.78	9.36
Alpha-Chlordane	11.39	12.31
Beta-Chlordane	11.19	12.23
4,4'-DDD	12.66	13.31
4,4'-DDE	11.75	12.54
4,4'-DDT	13.07	13.75
Dieldrin	11.98	12.77
Endosulfan I	11.49	12.37
Endosulfan II	12.89	13.51
Endosulfan Sulfate	13.52	14.07
Endrin	12.53	13.12
Endrin Aldehyde	13.3	13.67
Endrin Ketone	14.53	14.67
Heptachlor	9.34	10.42
Heptachlor Epoxide	10.87	11.72
Methoxychlor	14.18	14.36
Toxaphene	Pattern Recognition	

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TABLE 4
Quality Control Criteria
Pesticide Method 8081A/8081B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration (ICAL)	Initially and upon failure of Two consecutive CCV's	% RSD ≤ 20 for each analyte. If % RSD >20, linear regression with r≥0.995 for each analyte.	Evaluate cause; Repeat calibration; or Qualify data and discuss in narrative (1)
Second source calibration verification (ICV)	After each initial calibration	≤ 20% drift for each analyte (1) (≤ 15% for 8081A)	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Daily, before sample analysis, every ten samples, and at the end of the analysis sequence	≤ 20% drift for each analyte (1) (≤ 15% for 8081A)	Samples bracketed by an unacceptable CCV must be reanalyzed. Repeat initial calibration after two consecutive failures of the CCV (1).
Retention time window check	Each Sample	Relative retention time (RRT) of analyte within +/- 0.66	Correct problem; Re-analyze affected samples
Method Blank (MB)	One per matrix/batch: Maximum of 20 samples	< project report limit for each target analyte (1)	Notify supervisor and initiate NCR; Investigate; re-extract/ reanalyze samples or qualify data and address in the report narrative (3)
Laboratory Control Sample (LCS)	One per matrix/batch: Maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; Investigate; re-extract/ reanalyze samples or qualify data and address in the report narrative (3)
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated range (1)	Qualify data and/or address in the report narrative
Surrogate Spike	Every sample, standard, and quality control sample	Recoveries within the designated rages; use project QAPP or standard control criteria (1)	Notify supervisor and initiate NCR; investigate; re-extract/ reanalyze sample (s) or qualify data and address in the report narrative (3)

(1) Evaluation criteria are often project specific. Check the project QAPP or program criteria, e.g. OVAP.
(2) Standard criteria are set at three standard deviations from the mean. 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

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Figure 1

Checklist ID: 29956

Microbac Laboratories Inc.

Data Checklist

Date: 07-JUL-2008
Analyst: ECL
Analyst: NA
Method: 8081
Instrument: HP15
Curve Workgroup: NA
Runlog ID: 23093
Analytical Workgroups: L08060886, L08070147, L08070148

ANALYTICAL	
System Performance Check	NA
DFTPP (MS)	NA
Endrin/DDT breakdown (8081/MS)	X
Pentachlorophenol/benzidine tailing (MS)	NA
Eluent check (IC)/system pressure (HPLC)	NA
Window standard (FID)	NA
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	NA
Continuing calibration blank (CCB) (IC)	NA
Special standards	X
Blanks	X
TCL hits	X
Surrogate recoveries	X
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	X
MS/MSD/Sample duplicates	X
Recoveries	X
%RPD	X
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	X
Surrogate recoveries	X
Internal standard areas (MS)	NA
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	NA
Reruns	NA
Manual integrations	X
Project/client specific requirements	X
REPORTING	
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	ECL
SUPERVISORY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer: 08-JUL-2008

Secondary Reviewer: 10-JUL-2008

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Figure 2

Run Log ID: 23093

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HP15 Dataset: 070708
Analyst1: ECL Analyst2: NA
Method: 8081 SOP: GCS09 Rev: 8

Maintenance Log ID:

Column 1 ID: RTX-CLP Column 2 ID: RTX-CLP2
Workgroups: WG276166
Internal STD: NA Surrogate STD: STD26771 Calibration STD

Comments:

Seq.	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	15G15164.F	WG276181-01 ENDRIN/DDT	1	1	STD21936	07/07/08 11:34
2	15G15164.R	WG276181-01 ENDRIN/DDT	1	1	STD21936	07/07/08 12:02
3	15G15165.F	WG276181-02 PEST CCV 20 PPB	1	1	STD26279	07/07/08 12:02
4	15G15165.R	WG276181-02 PEST CCV 20 PPB	1	1	STD26279	07/07/08 12:31
5	15G15166.F	WG276166-01 PEST MRL 1.0 PPB	7	1	STD26279	07/07/08 13:15
6	15G15166.R	WG276166-01 PEST MRL 1.0 PPB	7	1	STD26279	07/07/08 13:43
7	15G15167.F	WG276182-01 TOX CCV 500 PPB	1	1	STD24420	07/07/08 13:43
8	15G15167.R	WG276182-01 TOX CCV 500 PPB	1	1	STD24420	07/07/08 14:12
9	15G15168.F	WG275923-02 BLANK V302 P129	7	1	SOIL	07/07/08 14:12
10	15G15168.R	WG275923-02 BLANK V302 P129	7	1	SOIL	07/07/08 14:40
11	15G15169.F	WG275923-03 LCS V302 P129	7	1	SOIL	07/07/08 14:40
12	15G15169.R	WG275923-03 LCS V302 P129	7	1	SOIL	07/07/08 15:09
13	15G15170.F	WG275923-04 TOX LCS V302 P129	7	1	SOIL	07/07/08 15:09
14	15G15170.R	WG275923-04 TOX LCS V302 P129	7	1	SOIL	07/07/08 15:37
15	15G15171.F	L08060886-07	7	1	SOIL	07/07/08 15:37
16	15G15171.R	L08060886-07	7	1	SOIL	07/07/08 16:05
17	15G15172.F	WG275923-01 L08060886-10 REF	7	1	SOIL	07/07/08 16:05
18	15G15172.R	WG275923-01 L08060886-10 REF	7	1	SOIL	07/07/08 16:34
19	15G15173.F	WG275923-05 L08060886-11 MS	7	1	SOIL	07/07/08 16:34
20	15G15173.R	WG275923-05 L08060886-11 MS	7	1	SOIL	07/07/08 17:02
21	15G15174.F	WG275923-06 L08060886-12 MSD	7	1	SOIL	07/07/08 17:02
22	15G15174.R	WG275923-06 L08060886-12 MSD	7	1	SOIL	07/07/08 17:31
23	15G15175.F	L08060886-16	7	1	SOIL	07/07/08 17:31
24	15G15175.R	L08060886-16	7	1	SOIL	07/07/08 17:59
25	15G15176.F	L08060147-01	7	1	SOIL	07/07/08 17:59
26	15G15176.R	L08060147-01	7	1	SOIL	07/07/08 18:28
27	15G15177.F	L08060148-01	7	1	SOIL	07/07/08 18:28
28	15G15177.R	L08060148-01	7	1	SOIL	07/08/08 08:15
29	15G15178.F	WG276166-02 PEST MRL 1.0 PPB	7	1	STD26279	07/08/08 08:15
30	15G15178.R	WG276166-02 PEST MRL 1.0 PPB	7	1	STD26279	07/08/08 08:43
31	15G15179.F	WG276181-03 PEST CCV 10 PPB	1	1	STD26279	07/08/08 08:43
32	15G15179.R	WG276181-03 PEST CCV 10 PPB	1	1	STD26279	07/08/08 09:12
33	15G15180.F	WG276182-02 TOX CCV 250 PPB	1	1	STD24420	07/08/08 09:12
34	15G15180.R	WG276182-02 TOX CCV 250 PPB	1	1	STD24420	07/08/08 09:40

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Appendix 1
Navy Clean

For samples analyzed under any Navy Clean project, Microbac will utilize 2nd column confirmation for any multi-responding analyte as per the Navy IRCDQM Appendix C 1999.

Appendix II
South Carolina requirements

The LCS recovery limits for samples analyzed for the state of South Carolina are 70-130%.

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Microbac

STANDARD OPERATING PROCEDURE
ANALYSIS OF DIESEL RANGE ORGANICS
METHOD 8015

Issue/Implementation Date: 29 June 2012

Last Review Date: 29 June 2012

Microbac Laboratories, Inc.
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1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine concentrations of Diesel Range Organics in soil and water samples. This corresponds to an alkane range from C_{10} to C_{28} and covers a boiling point range from approximately 170° C - 430° C. This method follows the procedures outlined in SW-846 Update III Method 8015B Revision 2, 8015C Revision 3 and Method 8015D Revision 4.

1.2 The method involves analysis of samples that have been solvent extracted on a Gas Chromatograph with a Flame Ionization Detector. Sample quantitation is based on a forced baseline integration of the entire chromatograph beginning with C_{10} and ending with C_{28}.

1.3 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

COA	Certificate of analysis
COD	Coefficient of determination
CCV	Continuing calibration verification
DRO	Diesel range organics
FID	Flame ionization detector
GC	Gas chromatograph
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LQAP	Laboratory Quality Assurance Program
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report
RL	Reporting limit
RSD	Relative standard deviation
SOP	Standard operating procedure
QC	Quality control

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

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1.4 Updates that effect concentration, vendor choices, reagents, MDLs, RLs and QC limits are subject to change without notice.

2.0 SAFETY PRECAUTIONS

2.1 Analysts using this method must be aware of certain hazards associated with performing the analysis.

2.2 Chemical Hazards

Organic solvents are handled while using this method. These solvents include hexane, methylene chloride, toluene, and benzene. Each of these solvents are either carcinogenics, suspected carcinogenics or attack organs of the human body. Extreme care must be exercised when these compounds are being used. Gloves must be worn and proper ventilation must be in place before using any of the above mentioned solvents. All work with these solvents must be performed in a fume hood that has had the face velocity verified within the past year.

2.2.1 Concentrated organic standards are used to calibrate the instruments. Many of these chemicals have been found to cause cancer and must be handled with extreme care. Gloves must be worn while these compounds are being used.

These compounds must be handled under a fume hood to prevent personnel exposure.

2.3 Thermal Hazards

2.3.1 Injection ports on the GC instrumentation are heated to a very high temperature in excess of 200° C. Contact with these areas must be avoided to prevent severe burns. The analyst should turn the heated zones off and allow them to cool before attempting to work close to them.

2.3.2 FID detectors on the GC instrumentation are heated to a very high temperature in excess of 200° C. Contact with these areas must be avoided to prevent severe burns. The analyst should turn the heated zones off and allow them to cool before attempting to work close to them.

2.3.3 The FID detectors need to burn a mixture of hydrogen and air to operate. Burns to the analyst could occur if these areas are not avoided during operation. The gas supplies to these detectors must be turned off before any attempt is made to work closely with the detectors.

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2.4 Broken glassware hazards exist when handling any glassware. Broken glassware must be discarded immediately in the broken glassware receptacle located in the laboratory.

2.5 Compressed gases are needed to operate the GC instrument. These gases must be handled with care to prevent damage to the gas cylinder. Damaged gas cylinders must be reported to the supervisor immediately. Hydrogen gas is extremely flammable and thorough testing of the system must be made to ensure no leaks are present.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Sample size and collection requirements are beyond the scope of this SOP; refer to Microbac Extraction Lab SOP's and SW-846 for details.

3.2 After the samples have been extracted, they must be stored at ≤ 6° C until the instrumental analysis can proceed. The samples must be analyzed within 40 days from the day of the extraction.

4.0 METHOD PERFORMANCE

4.1 Table 1 summarizes the performance data for water analysis; Table 2 summarizes performance data for soil/solid waste analysis. These tables include the analyte list, ranges for accuracy and precision, nominal laboratory RLs, and the current laboratory MDL.

4.2 The laboratory performed an initial assessment of the MDLs using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 1 and Table 2 and were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established as per Microbac SOP 45.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory usea results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

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4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 All possible measures are taken to eliminate interferences; however some samples have high levels of material that co-extract and make it virtually impossible to completely remove all interferences from the extracts. Samples which contain high levels of co-extractable material in the range of C_{10}-C_{28} will be quantitated as DRO by definition.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatography Apparatus

6.1.1 Agilent 5890 or 6890 Gas Chromatograph equipped with dual FID detector and dual split/splitless injection ports.

6.1.2 Agilent 7673 or 7683 autosampler towers, trays and controllers.

6.1.3 Restek RTX-CLP 0.53mm X 30M Megabore analytical column (or equivalent).

6.1.4 Agilent 3365 or newer Chemstation analytical software.

6.1.5 IBM compatible PC with a 486 or higher microprocessor and hard drive storage.

6.2 Glassware

6.2.1 2 mL autosampler vials

6.2.2 10 mL class "A" volumetric flask for preparation of standards

6.2.3 25 mL class "A" volumetric flask for preparation of standards

6.2.4 Pasteur pipets for the non-quantitative transfer of materials

6.3 Syringes

6.3.1 10 uL autosampler syringes

6.3.2 100 uL gas tight syringe for the preparation of standards

6.3.3 1 mL gas tight syringe for the preparation of standards

6.4 Miscellaneous Supplies

6.4.1 Necessary gas cylinders properly plumbed into the GC

6.4.1.1 Hydrogen gas (zero grade/high purity)

6.4.1.2 Helium carrier gas (zero grade/high purity)

6.4.1.3 Compressed Air (zero grade/high purity)

7.0 STANDARDS AND REAGENTS

7.1 All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Pesticide grade high purity methylene chloride

7.3 DRO stock standard – A 10,000 ug/mL stock standard is prepared from commercially available diesel fuel by transferring 0.5 g of diesel fuel into a 50 mL volumetric flask containing approximately 30 mL of methylene chloride. 5 mL of each surrogate stock standard is added to flask and the flask is filled to the 50 mL mark with methylene chloride, capped and inverted several times to ensure proper mixing. All standards have a six (6) month expiration date. All standards are stored in amber vials at ≤ 10° C.

7.4 Surrogates – o-terphenyl (Chemservice F-1054) is prepared by weighing 100 mg into a 10 mL volumetric flask and diluting with methylene chloride. Octacosane (Chemservice O-2227) is prepared by weighing 100 mg into a 10 mL volumetric flask and diluting with carbon disulfide.

7.5 Calibration standards – The calibration standards are prepared from stock standards at six (6) different levels; the lowest concentration is at or below the RL for the method. These are made by diluting the stock standard using the following scheme:

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Level	Concentration	Standards
#6	10,000 ug/mL	Standard stock
#5	5,000 ug/mL	500 uL stock: 500 uL CH_2Cl_2
#4	2,500 ug/mL	200 uL stock: 600 uL CH_2Cl_2
#3	1,000 ug/mL	100 uL standard #6: 900 uL CH_2Cl_2
#2	500 ug/mL	100 uL standard #5: 900 uL CH_2Cl_2
#1	100 ug/mL	100 uL standard #3: 900 uL CH_2Cl_2

7.5 CCV standard - This standard is used for the daily CCV. The 1000 ug/mL standard is prepared volumetrically using 100 uL of the stock standard in Section 7.2 and 900 uL of methylene chloride.

7.6 A window defining standard (Accustandard DRH-0045-5x) is prepared volumetrically at concentration of 200 ug/mL in methylene chloride. This standard contains even-numbered alkanes C_8 to C_{40} and may be used to establish alternate quantitation windows if required for a project.

7.7 ICV standard is purchased from Chemservice (CSRG0-611-1JS) at 5,000 ppm and diluted to 1000 ug/mL in methylene chloride.

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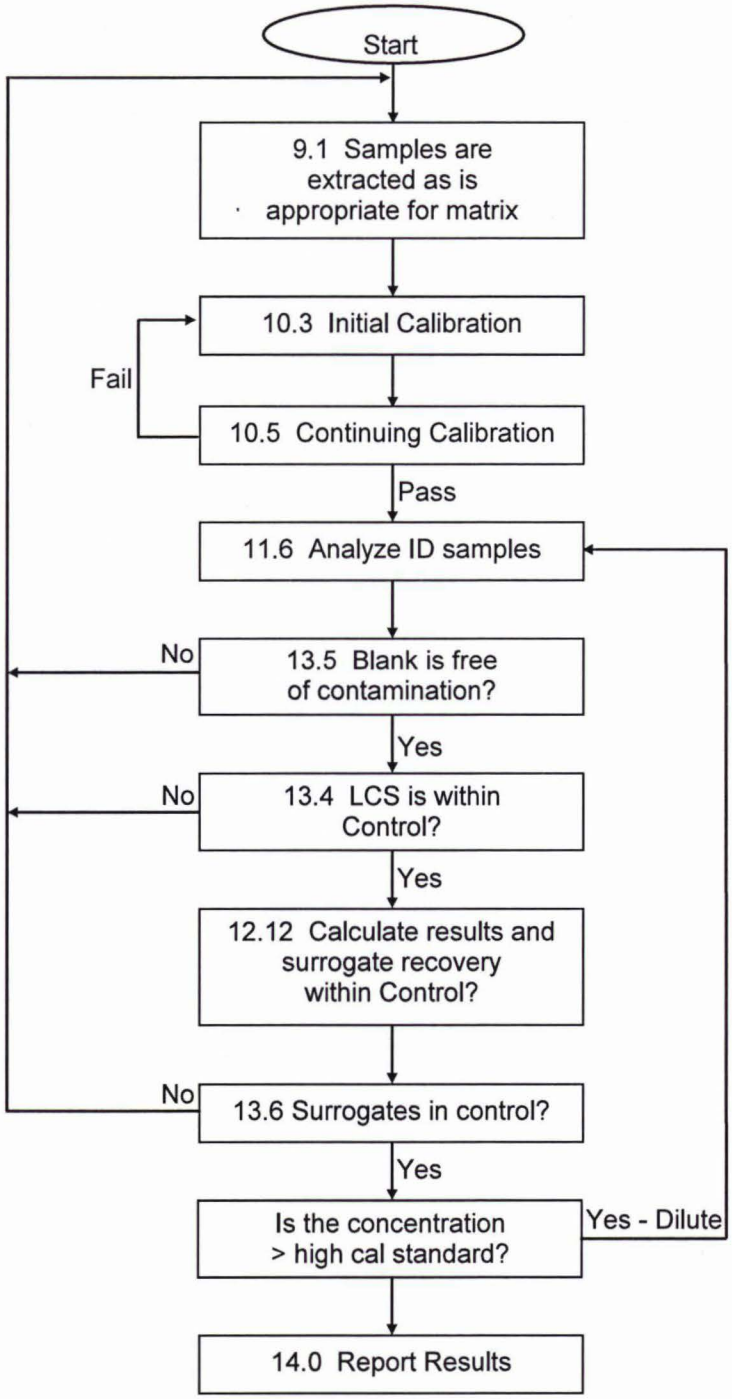
8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



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9.0 SAMPLE PREPARATION

9.1 The preparation of samples is beyond the scope of this SOP. Please refer to Microbac SOP EXD01 for details on sample preparation.

10.0 CALIBRATION PROCEDURES

10.1 Recommended Gas Chromatograph Conditions:

Injector port temperatures:	290° C
Detector temperatures:	310° C
Oven Program:	
Initial temperature:	40° C
Initial hold:	1 minute
Temperature ramp:	15° C /minute
Final temperature:	310° C
Final hold:	6 minutes
Amount injected:	1 uL

10.2 A window defining standard is injected daily prior to sample analysis to determine the integration range for sample analysis; the beginning of the n-decane peak defines C_{10} where integration begins and the end of the n-octacosane peak defines C_{28} where integration ends.

10.3 Initial calibration – 1 uL of each calibration standard is injected onto the instrument and the mean calibration factor and %RSD for the calibration is calculated using the equations from Section 12.0. Peak area responses are tabulated using a forced baseline integration. The RSD should be < 20%; if not, linear regression may be used provided the Coefficient of Determination (COD) R^2 is > 0.99. If the initial calibration fails to meet these criteria, the GC should be inspected for problems.

10.3.1 Using the appropriate markers from the window defining standard, alternate carbon ranges may be quantitated by using the same calibration factor established for C_{10}-C_{28}.

10.4 ICV: Immediately after the analysis of the calibration curve, the 1000 ppm second source standard is analyzed. The ICV must have a %D < 20% when compared to the calibration (Section 12.0). Inability to achieve a %D < 20% indicates a problem and will necessitate instrument maintenance and/or preparation of fresh standards and recalibration.

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10.5 CCV: After initial calibration has been successfully performed, samples may be analyzed by injecting 1 uL of each on the GC. A 1000 ug/mL CCV standard must be injected after every 10 samples or 12 hours, whichever is more frequent (batch QC samples are not to be counted in the ten (10) injections between CCV standards, but solvent blanks injected as contamination checks do not need to be counted). This is done to determine if the response of the analytical system has changed. Details on the calculations for the continuing calibration standard can be found in Section 12.0 of this document. The change in response of the analytical system must be ≤ 20% D.

If a CCV fails to yield CF's with % difference of ± 20 % then the CCV may be reanalyzed. The second CCV analysis must be from the same source as the first. Upon failure of two consecutive CCVs a new curve must be run and the samples, bracketed within the bad CCV, must be re-run.

10.6 If a project requires Method 8015B, the ICV and CCV criteria is < 15% D.

11.0 ANALYTICAL PROCEDURES

11.1 The instrument(s) are calibrated and the analytical sequence proceeds as in Section 10.0 of this document.

11.2 After sample analysis, the data system produces a quantitation report listing target analytes and concentrations found in the sample. Compounds are identified based on retention times in the window defining standard as described in Section 10.2. Retention time windows are needed to determine if a given peak is one of these compounds. To calculate the windows the analyst is required to perform statistical operations on chromatograms from three consecutive days. The width of the retention time windows for n-decane, surrogate o-terphenyl and octacosane are calculated by multiplying the standard deviation of their peaks by three (3). The retention time begins with the lower retention time window limit for n-decane and ends with the upper retention time limit for octacosane. The window is adjusted daily based on the retention times from the window defining standard.

11.3 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions should be prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Dilutions are prepared by using syringes to transfer aliquots of extract into appropriate amounts of solvent in autosampler vials. Examples are presented below:

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Dilution	Amount Sample Extract (uL)	Amount CH_2Cl_2 (uL)	Final Dilution Volume (uL)
2x	200	200	400
5x	100	400	500
10x	100	900	1000
20x	50	950	1000

Higher dilutions are prepared by performing serial dilutions, e.g. for a 100x dilution, a 10x dilution is diluted again by a factor of 10.

11.4 After the raw data is processed, it is uploaded into the LIMS.

11.5 The data is reviewed two times by the data review team; once for completeness and a second time for data validation.

12.0 DETAILS OF CALCULATIONS

12.1 The Calibration factor of each calibration standard must be determined. The total area of the chromatogram between the beginning and ending hydrocarbon markers are given on each quantitation report. The area of the surrogates is subtracted from the total area to determine the corrected total area (CTA) under the chromatogram. This CTA is used to calculate the individual calibration standard CF.

Calibration factor (CF) from external standard calibration is determined using the formula:

$$CF = \frac{A_s}{C_s}$$

where:

A_s = Peak area for the analyte or surrogate in the external standard
C_s = Concentration of the external standard (ug/L)

12.2 The average CF is determined using the formula:

Mean calibration factor (CF) = $\overline{CF}$

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

12.3 The standard deviation is calculated using the formula:

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n-1}}$$

12.4 The %RSD for the curve is calculated by first determining the mean and the standard deviation of the six CFs and the using the formula:

$$RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:

s = Standard deviation
x = Average

12.5 The percent (%) difference or drift is calculated using the formula:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.6 Calculation of concentration of analyte x using calibration factors is determined using the formula:

$$C_x = \frac{(A_x)(D)}{(\overline{CF})}$$

where:

A_x = Peak area for the analyte or surrogate
D = Dilution factor for the samples (1/10 = 10)

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CF = Mean CF for the analyte or surrogate from
C_x = Calculated concentration of the analyte or surrogate (ug/L)

12.7 Linear calibration calculations (external standard method)

The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response (area) = R
x = Concentration = C_i

And m and b are the slope and intercept from the regression equation

For a given response ratio we can solve for C_i:

$$C_i = [R - b]/m$$

Use equations 12.4 or 12.5 to calculate the sample concentration, C_x.

12.8 Coefficient of correlation:

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.9 Solving for the concentration in water sample:

For a given concentration, compute the unknown, C_x

$$C_x = (C_i)(V_f / V_i)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
D = dilution factor
C_x = concentration in the sample (ug/L)

12.10 Solving for the concentration in soil sample:

$$C_x = (C_i)(V_f/W_i)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = initial sample weight (g)
D = dilution factor
C_x = concentration of the sample (ug/kg) (as received)

12.11 Unknown surrogate recovery is determined using the formula:

$$o-terphenly\ and\ octacosane\%\ rec = [SA/Cf\,(surrogate)/surrogate\ added]*100$$

where:

SA = area of the surrogate peak
Cf = average surrogate Cf from the six point. calibration
Surrogate added = The amt. of surrogate in ug/mL added to the samples

12.12 Relative percent difference (RPD) is determined using the formula:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.13 The LCS recovery is calculated as follows:

$$\%R = \left(\frac{C_x}{C_T}\right)100$$

where:

C_x = the concentration of the analyte in the LCS.
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.14 Calculating recovery for matrix spikes (MS/MSD) is determined using the formula:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

13.0 QUALITY CONTROL REQUIREMENTS

13.1 The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), LCS, MS, MSD, laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in Section 8.0 for initial calibration, ICV using a second source, and CCV, and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 3.

13.2 A batch is defined as a group of samples that are extracted together. A batch contains a maximum of 20 samples. With each batch of samples extracted, an LCS and a method blank must also be extracted. Each batch should contain a MS and MSD. All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch. The LCS, MS and MSD are spiked so as to yield a concentration that fall near the mid-point of the curve. The MS/MSD samples may be waived if insufficient sample is available.

NOTE: Some projects require an LCSD. (Consult the project QAPP for guidance).

13.3 Section 10.0 describes the acceptable criteria for the ICV and CCV standards. If the ICV or CCV fails, it may be repeated only once. Samples (up to ten) must be bracketed between passing calibration verification standards.

13.4 The LCS (and LCSD, if included) must be evaluated for the acceptance criteria listed in Tables 1 and 2, and for any project specific criteria which may be more, or less, stringent. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 1 and 2 are used in the absence of a project QAPP, or program specified control limits. If the LCS is outside the control limits, the analyst must stop the analysis and prepare an NCR to be reviewed by the department supervisor. Corrective action will consist of re-extraction and re-analysis of the samples, unless other instructions are provided in the project QAPP.

13.5 The method blank cannot contain amounts of any target analytes that are over the RL. If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL for the presents of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch.

13.6 In order to monitor the extraction efficiency in each sample, a surrogate solution containing o-terphenyl and octacosane, is added to each sample in the extraction batch. The recoveries for at least one of these surrogates must fall within the limits given in Table 1 and Table 2. If any individual sample has both surrogates outside the given limits, then the sample must be re-extracted. Surrogate limits are listed in Table 1 and Table 2.

13.7 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.7.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 3 does not meet acceptance criteria. When nonconforming data

occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 3.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.7.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.7.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that may be invoked. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event. The data may be reported as per the following exceptions.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank is greater than or equal to the RL, but the corresponding sample results are non-detect.
- One or more surrogates are high and the samples are non-detect.
- The continuing calibration verification (bracketing) is biased high and the samples are non-detect.
- There is insufficient sample volume or weight to perform a corrective action.

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- The sample has exceeded holding time.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Prior to data entry into the LIMS (either manual or automatic), all data must undergo two (2) levels of review in the department. The primary review is performed by the analyst and the secondary review is performed by either the department supervisor (or a designee) or another qualified analyst.

14.2 Data Reporting

The reporting requirements depend upon the need of the client. Microbac offers four levels of data reporting which are described in some detail below.

14.2.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.2.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.2.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.2.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

14.2.5 Results for water samples are reported in ug/L to three significant figures.

14.2.6 Results for soil samples are reported in ug/kg, or mg/kg, on a dry weight basis to three significant figures.

15.0 PREVENTIVE MAINTENANCE

15.1 In order to minimize the downtime of the instrumentation, preventive maintenance is performed on a routine basis. The injection port liners and septa are changed regularly. Additionally, from time to time when the peak shape of the standards in

the chromatograph is deformed, the front portion of the analytical column is clipped to improve performance.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

16.4 The waste streams generated by the Semi Volatile Laboratory are as follows:

16.4.1 halogenated solvents

16.4.2 solid waste (autosampler vials).

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17.0 REFERENCES

17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8015B, Revision 2, December 1996.

17.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8015C, Revision 3, February 2007.

17.3 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8015D, Revision 4, June 2003.

17.4 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000B, Revision 2, December 1996.

17.5 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000C, Revision 3, March 2003.

17.6 Microbac SOP45, "Standard Operating Procedures for Method Validation Procedures"

17.7 Microbac SOP EXD01, "Standard Operating Procedures for the Extraction of Diesel Range Organics"

17.8 Microbac SOP LQAP, "Laboratory Quality Assurance Plan"

17.9 Microbac SOP GC-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.10 Microbac SOP GP-RCA, "Root Cause Analysis"

17.11 Microbac SOP33, "Laboratory Waste Management"

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Appendix I
California Gas Chromatograph Method

1. For any samples from California or North Carolina, the California Gas Chromatograph Method, Eisenberg, D. M. and others, 1985, Guidelines for addressing fuel leaks will be followed.

2. A 10% acceptance criteria for continuing calibration is used rather than 15%.

Appendix II
South Carolina Requirements

All standards holding times are six (6) months.

In Section 11.4 the term 'DRO standard' should be diesel fuel standard.

A continuing calibration check standard is to be analyzed every ten (10) samples or every twelve (12) hours whichever is more frequent. In addition, a methylene chloride blank and retention time standard are to be analyzed every twelve (12) hours.

Any samples with surrogate recoveries outside limits will be re-extracted to confirm sample matrix effects.

LCS limits will be 70-130%. If an LCS falls outside these limits we will investigate to see if there is a systematic problem with the method and take any necessary corrective action.

Each analytical sequence will begin with a method blank and LCS immediately after initial calibration or CCV.

Retention time windows are determined with each initial calibration and are not adjusted daily by the working retention time standard. The daily retention time markers must meet retention time windows determined at the time of the initial calibration or an initial calibration must be performed.

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Appendix III
Oil Range Organics

Analysis of Oil Range Organics (ORO) along with DRO or alone may be performed using the following modifications to this method:

1.1 Oil Range Organics correspond to an alkane range from C_{28} to C_{40}. See Tables 4 and 5 for performance data.

1.2 Integration of ORO begins with C_{28} and ends with C_{40}

7.3 DRO/ORO stock standard and calibration standard – prepared the same as DRO stock standard but with the addition of 0.5 g of commercially available 10W30 motor oil to the volumetric flask prior to dilution.

7.7 Initial calibration verification (ICV) standard containing diesel fuel and motor oil is purchased from Ultra Scientific (RGO-730 or equivalent) at a concentration of 50000 ug/mL and diluted to 1000 ug/mL in methylene chloride.

Analysis is otherwise the same as for DRO.

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TABLE 1
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR THE ANALYSES OF DIESEL RANGE ORGANICS
WATER

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% REC)*	PRECISION (% RPD)*	MDL* WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Diesel Range Organics	68334-30-5	8015B/8015C	30-125	0-20	250	500
Surrogates						
o-terphenyl		8015B/8015C	55-140			
octacosane		8015B/8015C	30-145			

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

TABLE 2
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR THE ANALYSES OF DIESEL RANGE ORGANICS
SOIL

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% REC)*	PRECISION (% RPD)*	MDL* SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
Diesel Range Organics	68334-30-5	8015B/8015C	45-105	0-30	2000	10000
Surrogates						
o-terphenyl		8015B/8015C	30-140			
octacosane		8015B/8015C	40-160			

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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TABLE 3
QUALITY CONTROL CRITERIA
DIESEL RANGE ORGANICS METHOD 8015B/8015C

Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	Initially and upon failure of two consecutive CCV's	%RSD <20%. Linear regression with R^2 ≥0.990.	Evaluate cause; Repeat calibration; Or Qualify data and discuss in narrative (1)
Second Source Calibration Verification (ICV)	After each initial calibration	≤ 20% drift for each analyte (1) (≤ 15% 8015B)	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing Calibration Verification (CCV)	Daily, before sample analysis, every ten samples, and at the end of the analysis sequence	≤ 20% drift for each analyte (1) (≤1 5% 8015B)	Samples bracketed by an unacceptable CCV must be reanalyzed. Repeat initial calibration after two consecutive failures of the CCV (1).
Method Blank (MB)	One per matrix/batch: Maximum of 20 samples per batch	< project report limit see Tables 1,2 (1)	See Section 13.5 (2)
Laboratory Control Sample (LCS)	One per matrix/batch: Maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria see Tables 1,2 (1)	See Section 13.4 (2)
Matrix Spikes/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated range see Tables 1,2 (1)	Qualify data and/or address in the report narrative
Surrogate Spike	Every sample, standard, and quality control sample	Recoveries within the designated rages; use project QAPP or standard control criteria see Tables 1,2 (1)	See Section 13.6 (2)

(1) Evaluation criteria are often project specific. Check the project QAPP. Standard criteria are set at three standard deviations from the mean.
(2) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

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TABLE 4
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR THE ANALYSES OF OIL RANGE ORGANICS WATER

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% REC)*	PRECISION (% RPD)*	MDL* WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Oil Range Organics	N/A	8015B/8015C	40-140	0-20	250	500
Surrogates						
o-terphenyl		8015B/8015C	55-140			
octacosane		8015B/8015C	30-145			

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

TABLE 5
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR THE ANALYSES OF OIL RANGE ORGANICS SOIL

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% REC)*	PRECISION (% RPD)*	MDL* SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
Oil Range Organics	N/A	8015B/8015C	40-140	0-30	2000	10000
Surrogates						
o-terphenyl		8015B/8015C	30-140			
octacosane		8015B/8015C	40-160			

* Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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Figure 1

Checklist ID: 27144

Microbac Laboratories Inc.

Data Checklist

Date: 31-MAR-2008
Analyst: HAV
Analyst: NA
Method: 8015B-DRO
Instrument: HP18
Curve Workgroup: NA
Runlog ID: 21389
Analytical Workgroups: L08030612, L08030635, L08030645

ANALYTICAL	
System Performance Check	NA
DFTPP (MS)	NA
Endrin/DDT breakdown (8081/MS)	NA
Pentachlorophenol/benzidine tailing (MS)	NA
Eluent check (IC)/system pressure (HPLC)	NA
Window standard (FID)	X
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	NA
Continuing calibration blank (CCB) (IC)	NA
Special standards	NA
Blanks	X
TCL hits	X
Surrogate recoveries	X
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	X
MS/MSD/Sample duplicates	NA
Recoveries	NA
%RPD	NA
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	NA
Surrogate recoveries	X
Internal standard areas (MS)	NA
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	X
Reruns	X
Manual integrations	NA
Project/client specific requirements	X
REPORTING	
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	HAV
SUPERVISORY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer:
01-APR-2008

Secondary Reviewer:
02-APR-2008

CHECKLIST1 - Modified 03/06/2008
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Figure 2

Run Log ID: 21389

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HP18 Dataset: 033108
Analyst1: HAV Analyst2: NA
Method: 8015B-DRO SOP: GC502 Rev: 5

Maintenance Log ID:

Column 1 ID: RTX-CLP Column 2 ID: NA
Workgroups: WG288996
Internal STD: NA Surrogate STD: STD25255 Calibration STD: STD23177
Comments:

Seq.	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	18G00160	CH2CL2	1	1		03/31/08 14:37
2	18G00161	WIN DEF STD	1	1		03/31/08 15:10
3	18G00162	WG288996-01 DRO CCV @1000ppm	1	1	STD23177	03/31/08 15:43
4	18G00163	WG288646-01 BLANK V296-P147	10	1		03/31/08 16:26
5	18G00164	WG288646-02 LCS V296-P147	10	1		03/31/08 17:01
6	18G00165	WG288646-03 LCS DUP V296-P147	10	1		03/31/08 17:35
7	18G00166	L08030635-01 10x	10	10		03/31/08 18:08
8	18G00167	L08030635-02 10x	10	10		03/31/08 18:42
9	18G00168	L08030635-03 10x	10	10		03/31/08 19:15
10	18G00169	L08030635-04 10x	10	10		03/31/08 19:48
11	18G00170	L08030645-01 10x	10	10		03/31/08 20:22
12	18G00171	L08030645-01 50x	10	50		03/31/08 20:55
13	18G00172	ch2cl2	1	1		03/31/08 21:28
14	18G00173	WG288996-02 DRO CCV @1000ppm	1	1	STD23177	03/31/08 22:01
15	18G00174	L08030612-01 10x	10	10		03/31/08 22:35
16	18G00175	L08030612-02 10x	10	10		03/31/08 23:09
17	18G00176	L08030612-03 10x	10	10		03/31/08 23:42
18	18G00177	L08030635-01 5X	10	5		04/01/08 00:16
19	18G00178	L08030635-03 5X	10	5		04/01/08 00:49
20	18G00179	L08030635-04 5X	10	5		04/01/08 01:22
21	18G00180	ch2cl2	1	1		04/01/08 01:55
22	18G00181	WG288996-03 DRO CCV @1000ppm	1	1	STD23177	04/01/08 02:29
23	18G00182	L08030645-01	10	1		04/01/08 08:43
24	18G00183	ch2cl2	1	1		04/01/08 09:16
25	18G00184	WG288996-04 DRO CCV @1000ppm	1	1	STD23177	04/01/08 11:25

Comments

Seq.	Rerun	Dil.	Reason	Analytes
7				
	Sample was not used, less diluted sample was uploaded.			
9				
	Sample was not used, less diluted sample was uploaded.			
10				
	Sample was not used, less diluted sample was uploaded.			
11				

Page: 1 Approved: 02-APR-08

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STANDARD OPERATING PROCEDURE
ORGANIC ANALYTES
METHOD 8270

Issue/Implementation Date: 15 January 2010

Last Review Date: 15 January 2010

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Michael D. Cochran, Semi-Volatile Supervisor	1/14/10 Date
David L. Bumgarner, Technical Director/QAO	1/14/10 Date
David E. Vandenberg, Managing Director	1/14/10 Date

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1.0 SCOPE AND APPLICATION

1.1 Method 8270 is a capillary column gas chromatography/mass spectroscopy (GC/MS) method utilized to determine the concentration of semivolatile organic compounds in extracts prepared from solid matrices, soil and water. This method follows the procedures outlined in SW-846 Update III Method 8270C Revision 3 and Method 8270D Revision 4. This method is also applicable for toxicity characteristic leaching procedure (TCLP) analytes and wastewater method 625. See Table 3 for the target compound list for each method. See Appendix I for Low level 8270 analysis, Appendix II for Method 625 analysis and Appendix III for 1,4-dioxane analysis.

1.2 Method 8270 can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds that fit this criteria include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

1.3 Several target compounds may require special treatment when being analyzed by this method.

1.3.1 Benzidine can be subject to oxidative losses during solvent concentration, which may contribute to poor chromatographic results. The benzidine data is considered for screening only. Use alternate methods for accurate quantitation.

1.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph and also photochemical decomposition.

1.3.3 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

1.3.4 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.3.5 3-methylphenol and 4-methylphenol are not separable by the conditions in this method and are reported as a total concentration.

1.3.6 Hexachlorophene does not respond well by GC/MS and Kepone has erratic chromatographic performance. These compounds are quantitated against a single calibration point and results are qualified as estimated.

1.3.7 Famphur is subject to erratic performance in the presence of the other compounds in the calibration standards. If this is a compound of concern, it must be calibrated separately; otherwise the results are qualified as estimated.

1.3.8 Aramite is a multiresponder. Quantitation is based on the first eluting peak.

1.4 The reporting limit (RL) of Method 8270 for determining an individual compound is approximately 165 ug/kg (wet weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 5 ug/L for ground water samples. RLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the mass spectrometer. Some compounds have higher reporting limits; see Tables 4 and 5 for details. Projects requiring lower reporting limits may be analyzed using the modifications in Appendix I.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method on the basis of internal audits by laboratory management and senior analysts.

2.0 SAFETY PRECAUTIONS

Analysts using this method must be aware of certain hazards associated with performing the analysis. Concentrated organic standards are used to calibrate the instruments. Many of these chemicals have been found to cause cancer and must be handled with extreme care. Gloves should be worn while these compounds are being used. Several zones on the GC/MS are heated to high temperatures, therefore care must be exercised when working around these areas or severe burns to the skin can occur. All dilutions that require the use of glassware must be made with care to reduce the possibility of cuts from broken glass.

3.0 SAMPLE PRESERVATION & STORAGE

Sample size and collection requirements are beyond the scope of the SOP; refer to extraction lab SOPs and SW-846 for details.

4.0 METHOD PERFORMANCE

4.1 Table 4 summarizes the performance data for water analysis; Table 5 summarizes performance data for soil/solid waste analysis. These tables include

the analyte list, ranges for accuracy and precision, nominal laboratory reporting limits (RL), and the current laboratory method detection limit(s).

4.2 The laboratory performed an initial assessment of the method detection limits (MDL) using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The limit(s) of detection (LOD), or verified MDL, are presented in Tables 4 and 5 and were established using verification procedures outlined in SOP 45.

4.4 The limit(s) of quantitation (LOQ) are the nominal laboratory reporting limit(s) (RL) and were established as per SOP 45.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. Going forward, the laboratory will use results from laboratory control samples (LCS) to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. As a quality control measure, all extraction blanks, are evaluated on all instruments that analyze samples in that extraction batch. Determination as to the source and extent of interference is essential in order to take corrective action to eliminate the problem.

5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Highly concentrated samples can cause interferences in several areas of the gas chromatograph system such as injection port systems, injection port liners, and the capillary column. Refer to the standard operating procedures to remedy these interference areas. To reduce carryover, the sample syringe must be rinsed out between samples with solvent.

Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination. As a final measure to eliminate contamination, a bake-out of the gas chromatograph is effective for most hydrocarbons.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph system: Agilent 6890 series

- splitless injection port system
- electronic pressure control
- HP 7673 or 6890 autosampler and controller
- capillary column fittings and reducing nuts

6.1.2 Column - J & W Scientific DB-5 or Restek RTX-5 Sil-MS or equivalent.
- 30 m x 0.32 mm ID 1 um film thickness

6.1.3 Mass spectrometer: Agilent 5973 Mass Selective Detector

- quadropole analyzer
- electron impact ionization

6.1.4 Data system: Windows-based MS Chemstation

- Agilent Environquant software, Version C.00.02 or newer
- NBS 75 K or newer NIST mass spectral library

6.2 Syringe - 10 uL (standard 5 uL autosampler), 100 uL, 500 uL, 1000 uL Hamilton Syringes or equivalent

6.3 Volumetric flasks, Class A - 10 mL to 1000 mL

6.4 Sample vials - glass with Teflon-lined screw caps or crimp tops. (2 mL)

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Reagent grade or better chemicals and solvents are used in all tests for this method. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The typical cutoff is 95% purity. All standards are stored at 10° C or less, unless otherwise specified by the manufacturer.

7.2 Primary calibration standards are purchased from Restek (equivalent mixtures may be used) and combined in a volumetric flask to prepare an intermediate standard as follows:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHYLENE CHLORIDE)	FINAL CONCENTRATION (ug/mL)
31850 8270 Megamix	1000	1000	5	200
31625 Appendix IX Mix #1	2000	500	5	200
31806 Appendix IX Mix #2	1000	1000	5	200
32419 EPA 8270 Organophosphorous Pesticides Mix	2000	500	5	200
31852 8270 Benzidines Mix #2	2000	500	5	200
31879 Benzoic Acid Mix	2000	500	5	200
31025 Acid Surrogate Mix	2000	500	5	200
31062 B/N Surrogate Standard Mix	5000	200	5	200

7.3 Alternate source (ICV) standards are purchased from NSI and used to prepare two (2) separate intermediate solutions in volumetric flasks.

7.3.1 ICV 1 (8270):

ICV 1 (8270) ALTERNATE SOURCE	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHYLENE CHLORIDE)	FINAL CONCENTRATION (ug/mL)
C-385 PNA Mix	2000	1000	10	200
C-395 Acid Extractables	2000	1000	10	200
C-402 Benzidines	2000	1000	10	200
C-403 Basic Extractables	2000	1000	10	200
C-404 Base/Neutrals 1	2000	1000	10	200
C-405 Base/Neutrals 2	2000	1000	10	200
W-271 Pyridine	5000	400	10	200
W-541 Benzoic Acid	5000	400	10	200
1288 1-Methylnaphthalene	5000	400	10	200

7.3.2 ICV 2 (Appendix IX):

ICV 2 (APPENDIX IX) ALTERNATE SOURCE	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHYLENE CHLORIDE)	FINAL CONCENTRATION (ug/mL)
C-411 Benzidines II	2000	1000	10	200
C-412 Amines	2000	1000	10	200
C-413 PNA II	2000	1000	10	200
C-414 Base/Neutrals III	2000	1000	10	200
C-415 Acid Extractables II	2000	1000	10	200
C-416 Sulfonates	2000	1000	10	200
C-417 Appendix IX OP Pesticides	2000	1000	10	200
C-426 Appendix IX Mix*	2000	1000	10	200
922 Aramite	2000	1000	10	200

*Diallate and Isosafrole are at initial concentrations of 4000 ug/mL and final concentrations of 400 ug/mL.

7.4 Internal standard solution is purchased as a certified mix in methylene chloride (Accustandard Z-014J or equivalent) at a concentration of 400 ug/mL and stored in reaction vials with mininert caps. The standards in this mix are acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene- d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}. Refer to Table 1 for internal standard analyte assignment for quantitation. Each 1 mL of sample extract undergoing analysis must be spiked with 10 uL of the internal standard solution, resulting in a concentration of 40 ng/uL of each internal standard.

7.5 GC/MS tuning standard: a methylene chloride solution containing 1000 ug/mL of decafluorotriphenylphosphine (DFTPP), pentachlorophenol, benzidine and DDT (Accustandard M-625-TS-20X or equivalent) is purchased and is diluted to 50 ug/mL by transferring 1 mL of the stock tuning standard to a 20 mL volumetric flask and diluting to volume with methylene chloride. Store at 4° C or less when not in use.

7.6 Calibration standards: The calibration standards are prepared from the stock standards at seven (7) different levels using the following scheme:

120 ug/mL (600uL stock: 400uL Methylene chloride)
100 ug/mL (500uL stock: 500uL $MeCl_2$)
80 ug/mL (400uL stock: 600uL $MeCl_2$)
50 ug/mL (250uL stock: 750uL $MeCl_2$)
25 ug/mL (75uL stock: 525uL $MeCl_2$)
15 ug/mL (75uL stock: 925uL $MeCl_2$)
3 ug/mL (15uL stock: 985uL $MeCl_2$)

Each calibration standard is spiked with 10 uL of the internal standard solution in the ratio of 10uL internal standard: 1mL of calibration standard. The high calibration point may be dropped if fewer points satisfy different method criteria (8270, AFCEE, etc.). The 3 ug/mL calibration standard is at a concentration below the reporting limit for most compounds (see Tables 4 and 5). The remaining calibration standards correspond to the working range of concentrations found in real samples but do not exceed the working range of the GC/MS system.

7.7 Alternate source standard are prepared by diluting 250 uL of each of the intermediate standards (section 7.3) with 750 uL methylene chloride and adding 10 uL of the internal standard mix.

7.8 If the additional TCL compounds (atrazine, benzaldehyde, 1,1'-biphenyl, caprolactam) are required, a custom mix is purchased from Restek (558465) at a concentration of 2000 ug/mL and a 200 ug/mL intermediate standard is prepared by adding 1000 uL of the standard to a 10 mL volumetric flask and diluting to volume with methylene chloride. Working standards are prepared as in section 7.6.

7.9 The alternate source standard for the additional TCL compounds is purchased from Supelco (47514-U) at a concentration of 2000 ug/mL and a 200 ug/mL intermediate standard is prepared by adding 1000 uL of the standard to a 10

mL volumetric flask and diluting to volume with methylene chloride. The working standard is prepared as in section 7.7.

7.10 Intermediate standards are sealed and stored at ≤ -10°C and protected from light to avoid evaporation and photochemical reaction. Intermediate standard solutions must be replaced every six (6) months or sooner if comparison with quality control check samples indicates a problem.

8.0 CALIBRATION PROCEDURES

8.1 Mass Calibration - Prior to DFTPP analysis, initial calibration, and continuing calibration, the mass spectrometer must be tuned using the system software. This calibration allows for lens polarity and charge intensities to be set for proper mass identification. The tuning need not be repeated on a daily basis unless DFTPP does not pass or maintenance is done on the mass spectrometer.

8.2 Initial calibration

8.2.1 Initial calibration recommended GC/MS conditions:

Mass range	35 - 500 amu
Scan time	1 second/scan or less
Temperature program	40° C, hold for 3 - 5 minutes 15 - 20° C/minute to 180° C 25° C/minute to 320° C, hold until after benzo(g,h,i)perylene elutes
Injector temperature	250 - 280° C
Transfer line temperature	280 - 300° C
Source temperature	230° C
MS Quad temperature	150° C
Injector	Splitless
Split time	0.25 - 1.0 minutes
Sample volume	0.5 uL
Carrier gas	Helium at 40 - 65 cm/sec

8.2.2 DFTPP MS conditions are the same as in Section 8.2 with the GC temperature program modified for a shorter run.

8.2.3 DFTPP hardware tune; prior to any standard or sample analysis, DFTPP must be analyzed. 0.5 uL of the 50 ug/mL DFTPP solution is injected and must result in a mass spectrum, which meets the criteria given in Table 2. These criteria must be

demonstrated at the beginning of each 12-hour shift. Three scans (the apex of the peak, the scan immediately before the apex, and the scan immediately after the apex) are averaged. Background subtraction is allowed only to eliminate extraneous column bleed or background ions. Degradation of DDT to DDE and DDD should not exceed 20%. Pentachlorophenol and benzidine should not have excessive tailing. Tailing factors of 5% for pentachlorophenol and 3% for benzidine are used as a guideline.

8.2.4 The internal standards selected in section 7.3 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. If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4 use m/z 152 for quantitation.)

8.2.5 The initial calibration curve is established by analyzing 0.5 uL of each of the seven (7) calibration standards and calculating response factors (Rfs) for each target compound using the equation in section 12.

8.2.6 Method 8270 requires a minimum of five-point initial calibration; low or high standards may be deleted from the calibration so long as a minimum of five (5) levels are used and the lowest calibration point supports the reporting limit for specific compounds (see Tables 4 and 5). Six (6) calibration points are required when using a quadratic regression fit. Calibration levels cannot be omitted from the middle of the calibration.

8.2.7 Calibration criteria for 8270C

8.2.7.1 A system performance check must be performed to ensure that minimum average RFs are being met. The System Performance Check Compounds (SPCCs) (Table 1) have a minimum acceptable RF of 0.050. These compounds typically have low RFs and tend to decrease in response as the system begins to deteriorate or the standards degrade.

8.2.7.2 The percent relative standard deviation (%RSD=100[SD/RF]) are calculated for each compound. The %RSD should be less than 15% for each compound, and it must be less than 30% for each Continuing Calibration Check (CCC) compound (see Table 1). See section 8.2.9 for calibration options if the %RSD is >15%.

8.2.8 Calibration criteria for 8270D

8.2.8.1 Minimum response factors for the most common analytes are listed in Table 7. Meeting these response factors demonstrates that compounds are behaving as expected and demonstrates the desired sensitivity.

8.2.8.2 The percent relative standard deviation (%RSD=100[SD/RF]) are calculated for each compound. The %RSD should be less than 20% for each compound. See section 8.2.9 for calibration options if the %RSD is >20%.

8.2.9 Calibration options

8.2.9.1 Linear – Using Average RF

The average RF option is always the preferred method of GC/MS calibration, since linearity may be assumed throughout the full calibration range. However, linear and quadratic models may be used under the conditions discussed in the following sections.

8.2.9.2 Linear Regression with Coefficient of Determination (COD) r^2 >0.99

Linear regression is an alternative to average RF, but has the potential for significant bias at the lower concentration levels. It should only be used when re-fitting the lowest calibration standard yields a maximum % drift of 30% (residual test). A weighing factor (1/X or $1/X^2$) may be applied to the calibration to minimize the bias.

8.2.9.3 Quadratic Calibration with COD r^2 >0.99

Several compounds on the 8270 extended lists and the EPA Appendix IX list do not display consistently linear behavior. Quadratic calibration, employing at least six (6) calibration points, may be used to improve accuracy for these analytes, particularly at the lower calibration levels, and is a better alternative than linear regression when linear fails the re-fitting test. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and should not be used for analytes with a previous history of linear performance.

8.2.9.4 Due to the large number of compounds that may be analyzed by this method, some may fail to meet the initial calibration criteria; so long as these compounds are not critical to a project, results may be reported with the data qualified as estimated. If more than 10% of the compounds exceed the RPD

limit and do not meet minimum correlation coefficient criteria for alternate curve fits, the system is too unstable for analysis and corrective action must be taken.

8.3 Initial Calibration Verification (ICV)

After the initial calibration, the 50ug/mL alternate source standard is analyzed and compared to the curve using the %D calculation (from section 12). The %D for all compounds should be ≤30 for all compounds. 90% of the compound list must meet this criteria for the verification to be valid. If there is time remaining in the 12 hour tune time, samples may be analyzed immediately following the ICV.

8.4 Continuing Calibration Verification (CCV)

8.4.1 Every 12 hours during analysis, the GC/MS must be hardware tuned with DFTPP to meet the criteria listed in Table 2. In conjunction with the DFTPP, this solution also contains benzidine, pentachlorophenol and DDT at 50 ug/mL each. The criteria for these analytes is described in section 8.2.3.

8.4.2 A 50 ug/mL continuing calibration verification (CCV) standard containing each compound of interest, surrogates, and internal standards is analyzed after DFTPP passes.

The response factors from this standard are compared with the average response factors from the latest initial calibration curve. This standard must meet SPCC and CCC criteria described in sections 8.3.3 and 8.3.4, respectively. The retention times of all internal standards must not shift more than 30 seconds from that in the mid-point standard of the most recent calibration curve.

8.4.3 Continuing calibration criteria for 8270C

8.4.3.1 System Performance Check Compounds (SPCCs)

SPCC's for method 8270C are identified in Table 1. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCC's is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, column contamination, and active sites in the column or chromatographic system.

8.4.3.2 Calibration Check Compounds (CCC's): after the system performance check is met, CCC's identified in Table 1 are used to check the validity of the initial calibration. The validity is ascertained on the basis of percent drift using the formula from section 12.

If the percent drift for each CCC is less than 20%, the initial calibration is assumed to be valid. If any one CCC has a %D greater than 20%, corrective action must be taken. If no source of trouble can be found, a new initial calibration must be analyzed. If the CCCs are not analytes required by the permit, then all required analytes must meet the 20% drift criterion.

8.4.3.3 Other compounds: the %D of all non-CCC compounds should be ≤40%D. 90% of the compound list must meet this criteria for the CCV to be valid.

8.4.4 Continuing calibration criteria for 8270D

8.4.4.1 Each of the most common analytes must meet the same minimum response factor requirement as the initial calibration (See Table 7).

8.4.4.2 All target compounds of interest must be evaluated using 20% D as a criterion. If the percent difference or percent drift is ≤ 20, the calibration is still valid.

8.4.4.3 Due to the large number of compounds that may be calibrated using this method, some compounds may fail either the minimum response factor or 20%D criteria. These compounds may be reported and qualified as estimated so long as sufficient sensitivity is present and they are not critical to a project. If more than 20% of the calibrated compounds fail the 20% D limit, the system is too unstable for analysis and corrective action must be taken.

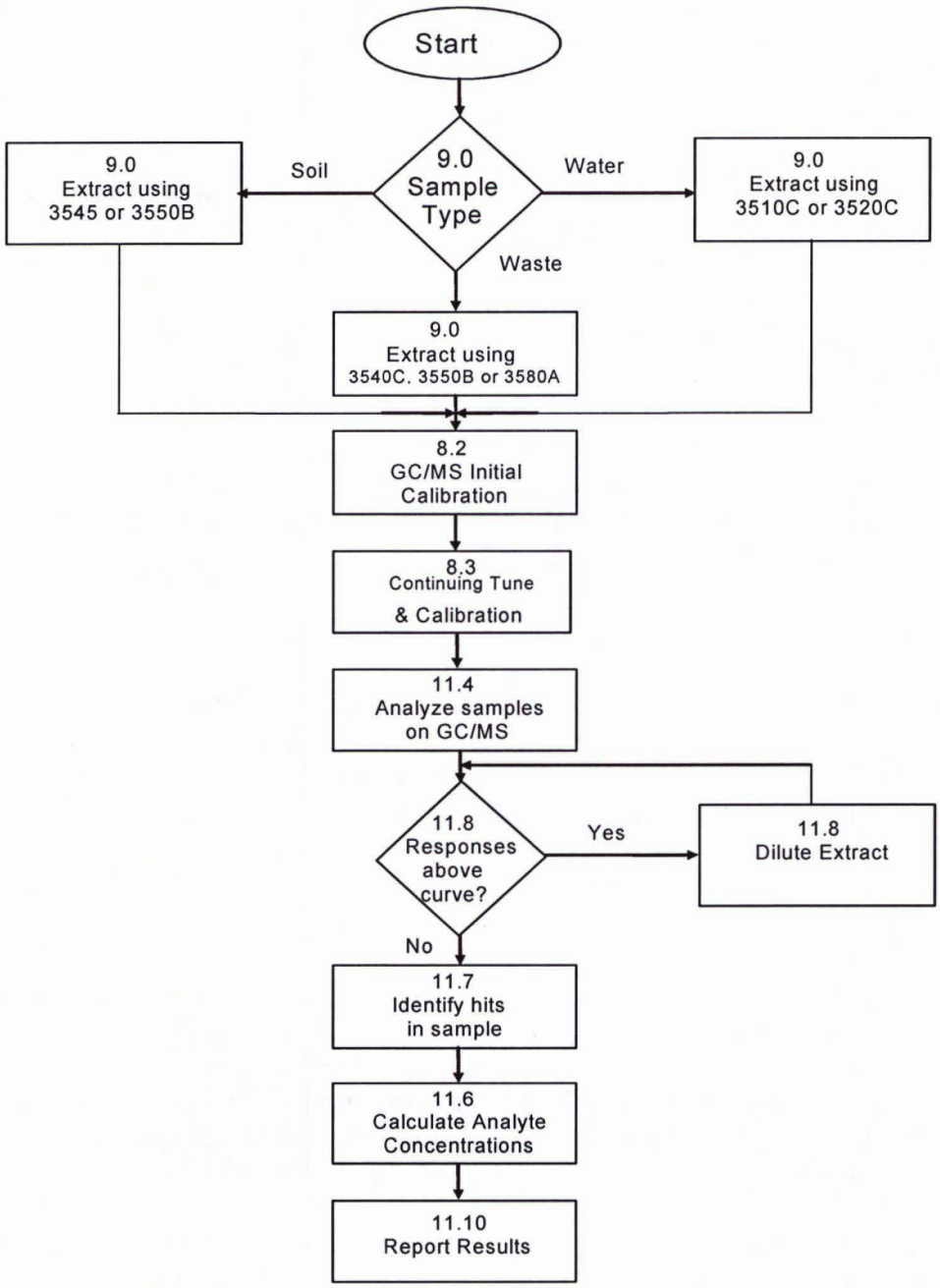
9.0 SAMPLE PREPARATION

Sample preparation - Samples for analysis by M8270 are prepared by the one of the following extraction methods prior to GC/MS analysis. The most current version of the methods will be used.

Matrix	Methods	SOP #
Water	3510C, 3520C	EXB01, EXB08
Soil/Sediment	3545, 3550B	ASE01, EXB02
Waste	3580A	EXB03

9.1 Direct injection - In limited applications direct injection (sample analysis without extraction) of the sample into the GC/MS system with a 10uL syringe may be appropriate. The detection limit is approximately 10,000 ug/L for direct injection; therefore, it is only acceptable where concentrations in excess of 10,000 ug/L are expected.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 The samples are first extracted using the appropriate extraction technique as described in section 9 of this SOP. Sample extracts are stored at 10°C ± 2° C until such time as the instrumental analysis can proceed.

11.2 The instruments are calibrated according to section 8.0 in this SOP.

11.3 Samples are run within the 12 hours tune time, which begins with the injection of DFTPP. At the end of this time, a new DFTPP and continuing calibration standard must be analyzed.

11.4 Prior to injection, a 400uL aliquot of each extract is spiked with 4 uL of internal standard.

11.5 After sample analysis, the computer produces a quantitation report listing all target analytes and concentrations found in the sample. Mass spectra are generated for each analyte found at or above the minimum detection limit and are evaluated for validity. Samples are library searched if necessary.

11.6 Qualitative analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra are obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of the sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

11.6.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.6.2 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of the characteristic ions should agree

within 30% of the relative intensities of these ions in the reference spectrum. Analyst judgment may be used even if these criteria are not met. The positive identification of a hit should not be made based solely on the criteria mentioned above.

11.7 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Dilutions are prepared by using syringes to transfer aliquots of extract into appropriate amounts of solvent in autosampler vials and adding appropriate amounts of internal standard in order to maintain the 40 ug/mL concentration in the extract. Examples are presented below.

Dilution	Amount sample extract	Amount CH2Cl2	Final dilution volume	Amount ISTD
2x	200 uL	200 uL	400 uL	4 uL
5x	100 uL	400 uL	500 uL	5 uL
10x	100 uL	900 uL	1000 uL	10 uL

Higher dilutions are prepared by performing serial dilutions, e.g. for a 100x dilution, a 10x dilution is diluted again by a factor of 10.

11.8 Tentatively identified compounds (TIC): For samples containing components not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. Refer to Section 12 for the determination of TIC concentrations (concentrations are "estimated"). Guidelines for making tentative identification are:

11.8.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

11.8.2 The relative intensities of the major ions should agree within ±20% for TIC's. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% - 70%.)

11.8.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.8.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.8.5 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.

11.9 The raw data is processed using the chem station software and the data is uploaded into the LIMS. The laboratory then performs a primary and secondary review of the raw data and quality control forms.

12.0 DETAILS OF CALCULATIONS

12.1 Response factors are calculated as follows:

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

where:

A_s = peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard
C_s = Concentration of the analyte or surrogate (ug/L)
C_{is} = Concentration of the internal standard (ug/L)

12.2 Accuracy as % difference:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.3 Coefficient of correlation

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.4 The concentration of a target analyte using average response factors is calculated as follows:

$$C_x = \frac{(A_x)(C_{is})(V_f)(D)}{(A_{is})(\overline{RF})(V_i)}$$

where:

A_x = Peak area for the analyte or surrogate
C_{is} = Concentration of the internal standard (ug/mL)
V_f = Final volume of the sample extract (mL)
D = Dilution factor for the samples (1/10 = 10)
A_{is} = Peak area of the internal standard
RF = Mean RF for the analyte or surrogate
V_i = Initial volume of the sample extracted (L)
C_x = Calculated concentration of the analyte or surrogate (ug/L)

12.5 Linear calibration calculations:

12.5.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

y = mx + b

where:

y = Response ratio = Response(x)/Response(istd) = R_x / R_{istd}

x = Concentration ratio = Conc(x)/Conc(istd) = C_x / C_{istd}

And m and b are the slope and intercept from the regression equation

12.5.2 For a given response ratio we can solve for C_x / C_{istd} :

C_x / C_{istd} = [R_x / R_{istd} - b]/m

Use equations 12.4 or 12.5 to calculate the unknown concentration, C_x.

12.6 Quadratic calibration calculations:

12.6.1 The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

12.6.2 $y = ax^2 + bx + c$

or

12.6.3 $ax^2 + bx + (c-y) = 0$

12.6.4 Solving for x using the quadratic equation:

$$x = \frac{b +/- \sqrt{(b^2 - 4a(c-y))}}{2a}$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x / R_{istd}

x = Concentration ratio = Conc(x)/Conc(istd) = C_x / C_{istd}

a,b and c are constants from the regression equation

Use equations 12.4 or 12.5 to calculate the unknown concentration, C_x.

12.7 Solving for the concentration in water sample:

12.7.1 For a given concentration ratio, compute the unknown, C_x

$$C_x = (C_{is})\left(C_x / C_{istd}\right)\left(V_f / V_i\right)(D)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = Initial sample volume (mL)
D = dilution factor
C_x = Concentration of the sample in ug/L

12.8 Solving for the concentration in soil sample:

$$C_x = (C_{is})\left(\frac{C_x}{C_{istd}}\right)\left(\frac{V_f}{W_i}\right)(D)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = Initial sample weight (g)
D = dilution factor
C_x = concentration of the sample (ug/kg) (as received)

12.9 Tentatively identified compounds (TIC) estimated concentration determination:

12.9.1 TIC water (ug/L):

$$ug/L = \frac{(A_x)(I_s)(Vf)}{(A_{is})(\overline{RRF})(Vi)}(D)(1000)$$

where :

A_x = total area of the peak from the total ion chromatogram
I_s = amount of internal standard injected (250ng)
A_{is} = total area of the internal standard from the total ion chromatogram
$\overline{RRF}$ = 1
Vf = final sample (extract) volume (mL)
Vi = initial sample volume (mL)
D = dilution factor

12.9.2 TIC soil/sediment:

$$ug/Kg = \frac{(A_X)(I_S)(Vf)}{(A_{is})(\overline{RRF})(Wi)}(D)(1000)$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, Vf = same as for water
Wi = initial sample weight (g)
D = dilution factor

12.10 The dry weight concentration of a target analyte is further calculated as follows:

$$C_d = \frac{(C_x)(100)}{(\%solids)}$$

where:

C_x = The uncorrected wet weight concentration
% solids = the percent solids content of the sample

12.11 Precision

Precision is expressed as relative standard deviation

$$RSD = \left(\frac{s}{\bar{x}}\right)100$$

12.12 Accuracy as % difference:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.13 Precision of duplicate measurements

Precision of duplicate measurements is expressed as relative percent difference

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.14 The LCS recovery is calculated as follows:

$$\%R = \left(\frac{C_x}{C_T}\right)100$$

where:

C_x = the concentration of the analyte in the LCS.
C_t = the theoretical spike concentration.
%R = percent recovery

12.15 Accuracy as Percent Recovery

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
%R = percent recovery

13.0 QUALITY CONTROL (QC) REQUIREMENTS

The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), laboratory control samples (LCS), matrix spikes (MS), matrix spike duplicates (MSD), laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in section 8 for initial calibration, initial calibration verification (ICV) using a second source, and continuing calibration verification (CCV), and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 6.

13.1 Table 6 describes several control items, their frequency and acceptance criteria, and the corrective action to be taken when the criteria are not met.

13.2 DFTPP must pass at the beginning of each shift, as in Table 2 and Section 8.2.3.

13.3 Continuing Calibration must pass each shift after DFTPP as outlined in Section 8.4. If CCV fails, it can be re-analyzed one time. The second CCV analysis must be from the same source as the first. If it still fails, the instrument must be recalibrated.

13.4 The method blank cannot contain amounts of any target analytes, which are over the reporting limits (RL). If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL

for the presents of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values appear on the QC summary sheet for the batch. Some projects require that blanks are evaluated to a concentration of ½ the RL, consult the specific QAPP for guidance.

13.5 The Laboratory Control Sample (LCS) is spiked by the extraction laboratory with every target analyte at 50 ug/L for water samples and 1665 ug/kg for soil. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 6 and 7 are used in the absence of a project QAPP, or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare a nonconformance report (NCR), and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare an (NCR) to be reviewed by the department supervisor. Corrective action will consist of re-extraction and re-analysis of the affected samples for, at a minimum, the COC for which a result was derived outside control limits, unless the client's representative and the quality assurance officer (QAO) approve of another course of action. If and LCS duplicate (LCSD) is included in the preparation batch, it must meet the sample criteria as the LCS.

13.6 The matrix spike (MS) and matrix spike duplicate (MSD) are spiked by the extraction laboratory for every target analyte at 50 ug/L for water and 1670 ug/Kg for soil. The results are included in the QC Summary Report and the data is used to monitor matrix accuracy and precision.

13.7 Internal standard areas must be between 50 and 200% of the areas from the continuing calibration standard. All samples failing to meet this criteria must be re-analyzed at the same dilution as the initial analysis unless interference can be shown by the sample chromatogram. If a matrix effect is suspected, the sample may be diluted for reanalysis.

13.8 Surrogate recoveries must fall within the limits found in Tables 6 and 7. Samples with surrogates outside these limits must be examined for matrix interference. If none can be proven, the sample must be returned for re-extraction and re-analysis. Samples with surrogate recoveries above the control limits may be approved if no target compounds are found. All surrogate recoveries must be ≥ 10% or a re-extract and re-analysis is warranted unless matrix interference is demonstrated by examination of the chromatogram. Samples failing one acid surrogate and one base neutral surrogate will not be re-extracted or re-analyzed.

Note: For Ohio VAP work all surrogates must be within limits for the method blank, LCS, LCSD, and field samples except in the case of obvious or known matrix interference.

13.9 Hold time: extracts must be analyzed within 40 days of extraction.

13.10 Back Up: All raw data files are automatically saved to the Microbac server on a daily basis.

13.11 Gases: Helium Tanks must be changed when pressure drops to 500 PSI.

13.12 When new standards are made, they must be checked against the existing curve before they can be used. Compare with old standards.

13.7 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13 of SOP LQAP (Laboratory Quality Assurance Program), SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and SOP GP-RCA (Root Cause Analysis).

13.7.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 6 does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Table 6.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 2).

A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.7.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per SOP GP-CAPA. The corrective action process includes a root cause analysis as per SOP GP-RCA, corrections, corrective action (s) and evidence of effectiveness.

13.7.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Prior to data entry into the LIMS (either manual or automatic), all data must undergo two (2) levels of review in the department. The primary review is performed by the analyst and the secondary review is performed by either the department supervisor (or a designee) or another qualified analyst.

14.2 Data Reporting

The reporting requirements depend upon the need of the client. Microbac offers four (4) levels of data reporting which are described in some detail below.

14.2.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.2.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.2.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.2.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

15.0 PREVENTIVE MAINTENANCE

In order to minimize the downtime of the instrumentation preventative maintenance is performed on a routine basis. The injection port liners and septa are changed regularly. The source is cleaned on an "as needed" basis, which is determined when the mass calibration of the instrument becomes increasingly difficult to perform. The front portion of the column may be protected by a guard column to prevent any high boiling contaminants from interfering with the performance of the analytical column. Additionally, from time to time when the peak shape of the standards are deformed, the front portion of the analytical column is clipped to improve performance (approximate 10 cm).

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a

manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Method 8270C, Revision 3, December 1996.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Method 8270D, Revision 4, February 2007.

3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000B, Revision 2, December 1996.

4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000C, Revision 3, March 2003.

5. 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.

6. U.S. EPA Contract Laboratory Program, Statement of Work for Organic Analysis, July 1985, Revision.

7. U.S. EPA Region 5, RCRA QAPP Instructions, Appendix I, April 1998 Revision

8. U.S. EPA 40 CFR Part 264, "Standard for Owners and Operators of Hazardous Waste Treatment, Storage and Disposal Facilities," July 1, 2001.

9. Microbac SOP33, "Laboratory Waste Management"

10. Microbac SOP LQAP, "Laboratory Quality Assurance Plan"

11. Microbac SOP45, "Standard Operating Procedures for Method Validation Procedures"

12. Microbac SOP GC-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

13. Microbac SOP GP-RCA, "Root Cause Analysis"

TABLE 1
TARGET ANALYTES WITH ASSOCIATED INTERNAL STANDARDS AND CHARACTERISTIC IONS

Compound	Primary Ion	Secondary Ion	Tertiary Ion
1,4-Dichlorobenzene-d4 (internal standard)	152	151	150
1,4-Dioxane	88	58	
n-Nitrosodimethylamine	74	42	44
Pyridine	79	52	51
2-Picoline	93	66	92
n-Nitrosomethylethylamine	88	42	43
Methyl Methanesulfonate	80	79	65
2-Fluorophenol (surrogate)	112	64	
n-Nitrosodiethylamine	102	42	44
Ethyl Methanesulfonate	79	109	97
Aniline	93	66	65
Benzaldehyde	105	106	77
Phenol-d5 (surrogate)	99	42	71
Phenol (CCC)	94	65	66
bis(2-Chloroethyl)ether	63	93	95
Pentachloroethane	167	165	117
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	111
1,4-Dichlorobenzene (CCC)	146	148	111
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	111
2-Methylphenol	107	108	77
bis(2-Chloroisopropyl)ether	45	77	121
3-, 4-Methylphenol	107	108	77
n-Nitrosopyrrolidine	100	41	42
n-Nitrosodipropylamine (SPCC)	70	42	101
Acetophenone	105	77	120
n-Nitrosomorpholine	56	116	86
o-Toluidine	106	107	77
Hexachloroethane	117	201	199
Naphthalene-d8 (internal standard)	136	68	
Nitrobenzene-d5 (surrogate)	82	128	54
Nitrobenzene	77	123	65
n-Nitrosopiperidine	114	42	55
Isophorone	82	95	138

TABLE 1 (continued)
TARGET ANALYTES WITH ASSOCIATED INTERNAL STANDARDS AND CHARACTERISTIC IONS

Compound	Primary Ion	Secondary Ion	Tertiary Ion
2-Nitrophenol (CCC)	139	109	65
2,4-Dimethylphenol	122	107	121
0,0,0-Triethyl Phosphorothioate	198	121	97
bis(2-Chloroethoxy)methane	93	95	123
Benzoic Acid	105	122	77
2,4-Dichlorophenol (CCC)	162	164	98
a,a-Dimethylphenethylamine	58	91	134
1,2,4-Trichlorobenzene	180	182	145
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
2,6-Dichlorophenol	162	164	98
Hexachloropropene	213	215	211
Hexachlorobutadiene (CCC)	225	223	227
Caprolactam	55	113	56
n-Nitrosodi-n-Butylamine	84	57	41
p-Phenylenediamine	108	80	107
4-Chloro-3-Methylphenol (CCC)	107	144	142
Safrole	162	104	131
2-Methylnaphthalene	142	141	115
1-Methylnaphthalene	142	141	115
Acenaphthene-d10 (internal standard)	164	162	160
1,2,4,5-Tetrachlorobenzene	216	214	179
Hexachlorocyclopentadiene (SPCC)	237	235	272
2,4,6-Trichlorophenol (CCC)	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (surrogate)	172	171	
Isosafrole	162	131	104
1,1'-Biphenyl	154	153	152
2-Chloronaphthalene	162	127	164
1-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
1,4-Naphthoquinone	158	104	102
Dimethylphthalate	163	194	164
1,3-Dinitrobenzene	168	75	76
2,6-Dinitrotoluene	165	63	89
Acenaphthylene	152	151	153

TABLE 1 (continued)
TARGET ANALYTES WITH ASSOCIATED INTERNAL STANDARDS AND CHARACTERISTIC IONS

Compound	Primary Ion	Secondary Ion	Tertiary Ion
3-Nitroaniline	138	108	92
2,4-Dinitrophenol (SPCC)	184	63	154
Acenaphthene (CCC)	154	153	152
4-Nitrophenol (SPCC)	65	109	139
2,4-Dinitrotoluene	165	63	89
Pentachlorobenzene	250	252	108
Dibenzofuran	168	139	169
2,3,4,6-Tetrachlorophenol	232	131	230
1-Naphthylamine	143	115	116
2-Naphthylamine	143	115	116
Diethylphthalate	149	177	150
Thionazin	107	97	96
Fluorene	166	165	167
4-Chlorophenyl Phenyl Ether	204	206	141
4-Nitroaniline	138	65	108
5-Nitro-o-Toluidine	152	77	106
1,2-Diphenylhydrazine	77	105	182
2,4,6-Tribromophenol (surrogate)	30	332	141
Phenanthrene-d10 (internal standard)	188	94	80
4,6-Dinitro-2-Methylphenol	198	51	105
n-Nitrosodiphenylamine (CCC)	169	168	167
Sulfotepp	322	97	202
Sym-Trinitrobenzene	75	74	213
Diallate	86	234	43
Phenacetin	108	109	179
Phorate	75	121	
4-Bromophenyl Phenyl Ether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	215	173
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	170
Pentachlorophenol (CCC)	266	264	268
Pronamide	173	175	145
Pentachloronitrobenzene	237	249	214
Disulfoton	88	97	89
Phenanthrene	178	179	176

TABLE 1 (continued)
TARGET ANALYTES WITH ASSOCIATED INTERNAL STANDARDS AND CHARACTERISTIC IONS

Compound	Primary Ion	Secondary Ion	Tertiary Ion
Anthracene	178	176	179
Carbazole	167	84	
Parathion Methyl	109	125	263
Di-n-Butyl Phthalate	149	150	104
Parathion Ethyl	97	109	291
4-Nitroquinoline 1-Oxide	190	160	89
Methapyrilene	58	97	71
Isodrin	193	263	195
Fluoranthene (CCC)	202	101	203
Chrysene-d12 (internal standard)	240	120	236
Benzidine	184		
Pyrene	202	200	203
Aramite	185	191	319
p-Terphenyl-d14 (surrogate)	244	122	215
p-(Dimethylamino)azobenzene	225	120	77
Chlorobenzilate	251	139	253
Famphur	218	125	93
Butyl Benzyl Phthalate	149	91	206
3,3-Dimethylbenzidine	212	106	213
Kepone	272	274	237
Hexachlorophene	196	198	209
2-Acetylaminofluorene	181	180	223
bis(2-Ethylhexyl)phthalate	149	167	279
3,3'-Dichlorobenzidine	252	254	126
Benzo[a]anthracene	228	229	226
Chrysene	228	226	229
Perylene-d12 (internal standard)	264	260	265
Di-n-Octyl Phthalate (CCC)	149	167	43
7,12-Dimethylbenz[a]anthracene	256	241	239
Benzo[b]fluoranthene	252	253	125
Benzo[k]fluoranthene	252	253	125
Benzo[a]pyrene (CCC)	252	253	125
3-Methylcholanthrene	268	252	253
Indeno[1,2,3-cd]pyrene	276	138	277
Dibenz[ah]anthracene	278	139	279
Benzo[ghi]perylene	276	138	277

TABLE 2
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

TABLE 3
REPORTABLE ANALYTES FOR MICROBAC GC/MS TESTS

Compound	8270	Appendix IX	TCLP	625	TCL
0,0,0-Triethyl Phosphorothioate		X			
1,1'-Biphenyl					X
1,2,4,5-Tetrachlorobenzene		X			
1,2,4-Trichlorobenzene	X	X		X	
1,2-Dichlorobenzene	X	X			
1,2-Diphenylhydrazine (1)					
1,3-Dichlorobenzene	X	X			
1,3-Dinitrobenzene					
1,4-Dichlorobenzene	X	X	X		
1,4-Dioxane (1)					
1,4-Naphthoquinone		X			
1-Chloronaphthalene (1)					
1-Methylnaphthalene (1)					
1-Naphthylamine		X			
2,3,4,6-Tetrachlorophenol		X			
2,4,5-Trichlorophenol	X	X	X		X
2,4,6-Trichlorophenol	X	X	X	X	X
2,4-Dichlorophenol	X	X		X	X
2,4-Dimethylphenol	X	X		X	X
2,4-Dinitrophenol	X	X		X	X
2,4-Dinitrotoluene	X	X	X	X	X
2,6-Dichlorophenol		X			X
2,6-Dinitrotoluene	X	X		X	
2-Acetylaminofluorene		X			
2-Chloronaphthalene	X	X		X	X
2-Chlorophenol	X	X		X	X
2-Methylnaphthalene	X	X			X
2-Methylphenol	X	X	X		X
2-Naphthylamine		X			
2-Nitroaniline	X	X			X
2-Nitrophenol	X	X		X	X
2-Picoline		X			
3-, 4-Methylphenol	X	X	X		X
3,3'-Dichlorobenzidine	X	X		X	X
3,3-Dimethylbenzidine		X			
3-Methylcholanthrene		X			
3-Nitroaniline	X	X			X

TABLE 3 (continued)
REPORTABLE ANALYTES FOR MICROBAC GC/MS TESTS

Compound	8270	Appendix IX	TCLP	625	TCL
4,6-Dinitro-2-Methylphenol	X	X		X	X
4-Aminobiphenyl		X			
4-Bromophenyl Phenyl Ether	X	X		X	X
4-Chloro-3-Methylphenol	X	X		X	X
4-Chloroaniline	X	X			X
4-Chlorophenyl Phenyl Ether	X	X		X	X
4-Nitroaniline	X	X			X
4-Nitrophenol	X	X		X	X
4-Nitroquinoline 1-Oxide		X			
5-Nitro-o-Toluidine		X			
7,12-Dimethylbenz[a]anthracene		X			
a,a-Dimethylphenethylamine		X			
Acenaphthene	X	X		X	X
Acenaphthylene	X	X		X	X
Acetophenone		X			X
Aniline		X			
Anthracene	X	X		X	X
Aramite		X			
Atrazine					X
Benzaldehyde					X
Benzidine				X	
Benzo[a]anthracene	X	X		X	X
Benzo[a]pyrene	X	X		X	X
Benzo[b]fluoranthene	X	X		X	X
Benzo[ghi]perylene	X	X		X	X
Benzo[k]fluoranthene	X	X		X	X
Benzoic Acid	X				
Benzyl Alcohol	X	X			
bis(2-Chloroethoxy)methane	X	X		X	X
bis(2-Chloroethyl)ether	X	X		X	X
bis(2-Chloroisopropyl)ether	X	X		X	X
bis(2-Ethylhexyl)phthalate	X	X		X	X
Butyl Benzyl Phthalate	X	X		X	X
Caprolactam					X
Carbazole	X				X
Chlorobenzilate		X			
Chrysene	X	X		X	X

TABLE 3 (continued)
REPORTABLE ANALYTES FOR MICROBAC GC/MS TESTS

Compound	8270	Appendix IX	TCLP	625	TCL
Diallate		X			
Dibenz[ah]anthracene	X	X		X	X
Dibenzofuran	X	X			X
Diethylphthalate	X	X		X	X
Dimethoate		X			
Dimethylphthalate	X	X		X	X
Di-n-Butyl Phthalate	X	X		X	X
Di-n-Octyl Phthalate	X	X		X	X
Disulfoton		X			
Ethyl Methanesulfonate		X			
Famphur		X			
Fluoranthene	X	X		X	X
Fluorene	X	X		X	X
Hexachlorobenzene	X	X	X	X	X
Hexachlorobutadiene	X	X	X	X	X
Hexachlorocyclopentadiene	X	X		X	X
Hexachloroethane	X	X	X	X	X
Hexachlorophene		X			
Hexachloropropene		X			
Indeno[1,2,3-cd]pyrene	X	X		X	X
Isodrin		X			
Isophorone	X	X		X	X
Isosafrole		X			
Kepone		X			
Methapyrilene		X			
Methyl Methanesulfonate		X			
Naphthalene	X	X		X	X
Nitrobenzene	X	X	X	X	X
n-Nitrosodiethylamine		X			
n-Nitrosodimethylamine		X		X	
n-Nitrosodi-n-Butylamine		X			
n-Nitrosodiphenylamine	X	X		X	X
n-Nitrosodipropylamine	X	X		X	X
n-Nitrosomethylethylamine		X			
n-Nitrosomorpholine		X			
n-Nitrosopiperidine		X			
n-Nitrosopyrrolidine		X			

TABLE 3 (continued)
REPORTABLE ANALYTES FOR MICROBAC GC/MS TESTS

Compound	8270	Appendix IX	TCLP	625	TCL
o-Toluidine		X			
p-(Dimethylamino)azobenzene		X			
Parathion Ethyl		X			
Parathion Methyl		X			
Pentachlorobenzene		X			
Pentachloroethane		X			
Pentachloronitrobenzene		X			
Pentachlorophenol	X	X	X	X	X
Phenacetin		X			
Phenanthrene	X	X		X	X
Phenol	X	X		X	X
Phorate		X			
p-Phenylenediamine		X			
Pronamide		X			
Pyrene	X	X		X	X
Pyridine		X	X		
Safrole		X			
Sulfotepp		X			
Sym-Trinitrobenzene		X			
Thionazin		X			

(1) Compound available for reporting upon request.

TABLE 4
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF GROUNDWATER BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
0,0,0-Triethyl Phosphorothioate	126-68-1	40-140 (1)	30	2.5	5
1,1'-Biphenyl	92-52-4	40-140 (1)	30	2.5	5
1,2,4,5-Tetrachlorobenzene	95-94-3	40-140 (1)	30	2.5	5
1,2,4-Trichlorobenzene	120-82-1	25-105	30	2.5	5
1,2-Dichlorobenzene	95-50-1	25-110	30	2.5	5
1,2-Diphenylhydrazine	122-66-7	40-110	30	2.5	5
1,3-Dichlorobenzene	541-73-1	25-110	30	2.5	5
1,3-Dinitrobenzene	99-65-0	30-130	30	2.5	5
1,4-Dichlorobenzene	106-46-7	25-110	30	2.5	5
1,4-Dioxane (3)	123-93-1	50-150	30	0.5	10
1,4-Naphthoquinone	130-15-4	40-140 (1)	30	2.5	5
1-Chloronaphthalene	90-13-1	40-140 (1)	30	2.5	5
1-Methylnaphthalene	90-12-0	25-120	30	2.5	5
1-Naphthylamine	134-32-7	25-140 (1)	30	2.5	5
2,3,4,6-Tetrachlorophenol	58-90-2	40-140	30	2.5	5
2,4,5-Trichlorophenol	95-95-4	35-120	30	2.5	5
2,4,6-Trichlorophenol	88-06-2	30-120	30	2.5	5
2,4-Dichlorophenol	120-83-2	20-110	30	2.5	5
2,4-Dimethylphenol	105-67-9	20-120	30	2.5	5
2,4-Dinitrophenol	51-28-5	20-140	30	12.5	25
2,4-Dinitrotoluene	121-14-2	50-139	30	2.5	5
2,6-Dichlorophenol	87-65-0	40-140 (1)	30	2.5	5
2,6-Dinitrotoluene	606-20-2	50-120	30	2.5	5
2-Acetylaminofluorene	53-96-3	40-140 (1)	30	2.5	5
2-Chloronaphthalene	91-58-7	25-120	30	2.5	5
2-Chlorophenol	95-57-8	25-110	30	2.5	5
2-Methylnaphthalene	91-57-6	25-120	30	2.5	5
2-Methylphenol	95-48-7	20-110	30	2.5	5
2-Naphthylamine	91-59-8	40-140 (1)	30	2.5	5
2-Nitroaniline	88-74-4	45-115	30	12.5	25
2-Nitrophenol	88-75-5	20-115	30	2.5	5
2-Picoline	109-06-8	40-140 (1)	30	2.5	5
3-, 4-Methylphenol	108-39-4/106-44-5	20-110	30	2.5	5
3,3'-Dichlorobenzidine	91-94-1	30-140	30	2.5	5
3,3-Dimethylbenzidine	119-93-7	10-120	30	10	25

TABLE 4 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF GROUNDWATER BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
3-Methylcholanthrene	56-49-5	40-140 (1)	30	2.5	5
3-Nitroaniline	99-09-2	40-120	30	12.5	25
4,6-Dinitro-2-Methylphenol	534-52-1	40-145	30	12.5	25
4-Aminobiphenyl	92-67-1	40-140 (1)	30	2.5	5
4-Bromophenyl Phenyl Ether	101-55-3	40-115	30	2.5	5
4-Chloro-3-Methylphenol	59-50-7	25-110	30	2.5	5
4-Chloroaniline	106-47-8	25-120	30	2.5	5
4-Chlorophenyl Phenyl Ether	7005-72-3	35-120	30	2.5	5
4-Nitroaniline	100-01-6	53-135	30	12.5	25
4-Nitrophenol	100-02-7	10-120	30	12.5	25
4-Nitroquinoline 1-Oxide	56-57-5	10-120	30	10	25
5-Nitro-o-Toluidine	99-55-8	10-120 (1)	30	2.5	5
7,12-Dimethylbenz[a]anthracene	57-97-6	40-140 (1)	30	12.5	25
a,a-Dimethylphenethylamine	122-09-8	10-120 (1)	30	12.5	25
Acenaphthene	83-32-9	30-120	30	2.5	5
Acenaphthylene	208-96-8	30-120	30	2.5	5
Acetophenone	98-86-2	25-150	30	2.5	5
Aniline	62-53-3	10-110	30	5	10
Anthracene	120-12-7	55-130	30	2.5	5
Aramite	140-57-8	10-120 (1)	30	2.5	5
Atrazine	1912-24-9	40-140 (1)	30	2.5	5
Benzaldehyde	100-52-7	40-140 (1)	30	2.5	5
Benzidine	92-87-5	0-155	30	12.5	25
Benzo[a]anthracene	56-55-3	60-130	30	2.5	5
Benzo[a]pyrene	50-32-8	55-135	30	2.5	5
Benzo[b]fluoranthene	205-99-2	45-125	30	2.5	5
Benzo[ghi]perylene	191-2-4-2	45-140	30	2.5	5
Benzo[k]fluoranthene	207-08-9	55-140	30	2.5	5
Benzoic Acid	65-85-0	10-100	30	12.5	25
Benzyl Alcohol	100-51-6	20-110	30	2.5	5
bis(2-Chloroethoxy)methane	111-91-1	20-105	30	2.5	5
bis(2-Chloroethyl)ether	111-44-4	25-110	30	2.5	5
bis(2-Chloroisopropyl)ether	108-60-1	20-110	30	2.5	5
bis(2-Ethylhexyl)phthalate	117-81-7	50-150	30	2.5	5
Butyl Benzyl Phthalate	85-68-7	55-150	30	2.5	5
Caprolactam	105-60-2	40-140 (1)	30	2.5	5
Carbazole	86-74-8	50-130	30	2.5	5

TABLE 4 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF GROUNDWATER BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
Chlorobenzilate	510-15-6	40-140 (1)	30	2.5	5
Chrysene	218-01-9	55-130	30	2.5	5
Diallate	2303-16-4	50-200 (1)	30	2.5	5
Dibenz[ah]anthracene	53-70-3	45-125	30	2.5	5
Dibenzofuran	132-64-9	35-115	30	2.5	5
Diethylphthalate	84-66-2	45-120	30	2.5	5
Dimethoate	60-51-5	40-140 (1)	30	2.5	5
Dimethylphthalate	131-11-3	25-112	30	2.5	5
Di-n-Butyl Phthalate	84-74-2	55-118	30	2.5	5
Di-n-Octyl Phthalate	117-84-0	40-146	30	2.5	5
Disulfoton	298-04-4	40-140(1)	30	2.5	5
Ethyl Methanesulfonate	62-50-0	30-130 (1)	30	2.5	5
Famphur (Estimated)	52-85-7	(2)	30	NA	10
Fluoranthene	206-44-0	50-137	30	2.5	5
Fluorene	86-73-7	40-120	30	2.5	5
Hexachlorobenzene	118-74-1	50-130	30	2.5	5
Hexachlorobutadiene	87-68-3	24-105	30	2.5	5
Hexachlorocyclopentadiene	77-47-4	20-143	30	2.5	5
Hexachloroethane	67-72-1	25-95	30	2.5	5
Hexachlorophene (Estimated)	70-30-4	(2)	NA	NA	50
Hexachloropropene	1888-71-7	30-130 (1)	30	2.5	5
Indeno[1,2,3-cd]pyrene	193-39-5	50-135	30	2.5	5
Isodrin	78-59-1	40-140 (1)	30	2.5	5
Isophorone	78-59-1	30-110	30	2.5	5
Isosafrole	120-58-1	10-120 (1)	30	2.5	5
Kepone (Estimated)	143-50-0	(2)	30	NA	20
Methapyrilene	91-80-5	10-120 (1)	30	12.5	25
Methyl Methanesulfonate	66-27-3	40-140 (1)	30	2.5	5
Naphthalene	91-20-3	25-110	30	2.5	5
Nitrobenzene	98-95-3	30-110	30	2.5	5
n-Nitrosodiethylamine	55-18-5	40-140 (1)	30	2.5	5
n-Nitrosodimethylamine	62-75-6	20-110	30	2.5	5
n-Nitrosodi-n-Butylamine	924-16-3	40-140 (1)	30	2.5	5
n-Nitrosodiphenylamine	86-30-6	40-110	30	2.5	5
n-Nitrosodipropylamine	621-64-7	28-120	30	2.5	5
n-Nitrosomethylethylamine	10595-95-6	40-140	30	2.5	5
n-Nitrosomorpholine	59-89-2	40-140 (1)	30	2.5	5

TABLE 4 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF GROUNDWATER BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
n-Nitrosopiperidine	100-75-4	40-140 (1)	30	2.5	5
n-Nitrosopyrrolidine	930-55-2	40-140 (1)	30	2.5	5
o-Toluidine	95-53-4	10-120 (1)	30	2.5	5
p-(Dimethylamino)azobenzene	60-11-7	40-140 (1)	30	2.5	5
Parathion Ethyl	56-38-2	40-140 (1)	30	2.5	5
Parathion Methyl	298-00-0	50-150 (1)	30	2.5	5
Pentachlorobenzene	608-93-5	40-140 (1)	30	2.5	5
Pentachloroethane	76-01-7	20-120 (1)	30	2.5	5
Pentachloronitrobenzene	82-68-8	40-140 (1)	30	2.5	5
Pentachlorophenol	87-86-5	40-140	30	12.5	25
Phenacetin	62-44-2	40-140 (1)	30	2.5	5
Phenanthrene	85-01-8	55-120	30	2.5	5
Phenol	108-95-2	10-120	30	2.5	5
Phorate	298-02-2	40-140 (1)	30	2.5	5
p-Phenylenediamine	106-50-3	10-120 (1)	30	50	100
Pronamide	23950-58-5	40-140 (1)	30	2.5	5
Pyrene	129-00-0	55-130	30	2.5	5
Pyridine	110-86-1	10-120	30	12.5	25
Safrole	94-59-7	40-140 (1)	30	2.5	5
Sulfotepp	3689-24-5	40-140 (1)	30	2.5	5
Sym-Trinitrobenzene	99-35-4	10-120 (1)	30	2.5	5
Thionazin	297-97-2	40-140 (1)	30	2.5	5
SURROGATES					
2-Fluorobiphenyl	321-60-8	43-116	NA		
2-Fluorophenol	367-12-4	21-100	NA		
2,4,6-Tribromophenol	118-79-6	10-123	NA		
Nitrobenzene-d5	4165-60-0	35-114	NA		
Phenol-d5	13127-88-3	10-94	NA		
p-Terphenyl-d14	1718-51-0	33-141	NA		

(1) Compound not routinely spiked into LCS, may be added upon request.

(2) Not spiked and/or evaluated.

(3) See Appendix III

TABLE 4A
MICROBAC'S QA OBJECTIVES FOR LOW LEVEL SEMIVOLATILE ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% RECOVERY)*	PRECISION (%RPD)*	VERIFIED MDL (ug/L)	REPORTING LIMITS (ug/L)
Acenaphthene	83-32-9	54-145	0-40	0.25	0.5
Acenaphthylene	208-96-8	49-137	0-40	0.25	0.5
Anthracene	120-12-7	55-140	0-40	0.25	0.5
Benzo[a]anthracene	56-55-3	60-150	0-40	0.25	0.5
Benzo[a]pyrene	50-32-8	55-146	0-40	0.25	0.5
Benzo[b]fluoranthene	205-99-2	55-142	0-40	0.25	0.5
Benzo[ghi]perylene	191-24-2	47-153	0-40	0.25	0.5
Benzo[k]fluoranthene	207-08-9	55-145	0-40	0.25	0.5
Benzyl Alcohol	100-51-6	36-116	0-40	0.25	0.5
bis(2-Chloroethoxy)methane	111-91-1	40-109	0-40	0.25	0.5
bis(2-Chloroethyl)ether	111-44-4	39-120	0-40	0.25	0.5
bis(2-Chloroisopropyl)ether	108-60-1	37-120	0-40	0.25	0.5
bis(2-Ethylhexyl)phthalate	117-81-7	46-150	0-40	2.5	5.0
4-Bromophenyl Phenyl Ether	101-55-3	53-120	0-40	0.25	0.5
Butyl Benzyl Phthalate	85-68-7	50-150	0-40	0.25	0.5
4-Chloroaniline	106-47-8	36-120	0-40	0.25	0.5
4-Chloro-3-Methylphenol	59-50-7	38-113	0-40	0.25	0.5
2-Chloronaphthalene	91-58-7	60-130	0-40	0.25	0.5
2-Chlorophenol	95-57-8	35-120	0-40	0.25	0.5
4-Chlorophenyl Phenyl Ether	7005-72-3	47-131	0-40	0.25	0.5
Chrysene	218-01-9	60-150	0-40	0.25	0.5
Dibenz[ah]anthracene	53-70-3	50-150	0-40	0.25	0.5
Dibenzofuran	132-64-9	43-129	0-40	0.25	0.5
Di-n-Butyl Phthalate	84-74-2	50-150	0-40	0.25	0.5
1,2-Dichlorobenzene	95-50-1	40-120	0-40	0.25	0.5
1,3-Dichlorobenzene	541-73-1	33-120	0-40	0.25	0.5
1,4-Dichlorobenzene	106-46-7	33-120	0-40	0.25	0.5
3,3'-Dichlorobenzidine	91-94-1	40-150	0-40	0.25	0.5
2,4-Dichlorophenol	120-83-2	44-120	0-40	0.25	0.5
Diethylphthalate	84-66-2	50-138	0-40	0.25	0.5
2,4-Dimethylphenol	105-67-9	40-120	0-40	0.25	0.5

TABLE 4A(continued)
MICROBAC'S QA OBJECTIVES FOR LOW LEVEL SEMIVOLATILE ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% RECOVERY)*	PRECISION (%RPD)*	VERIFIED MDL (ug/L)	REPORTING LIMITS (ug/L)
Dimethylphthalate	131-11-3	40-135	0-40	0.25	0.5
4,6-Dinitro-2-Methylphenol	534-52-1	40-145	0-40	1.0	2.0
2,4-Dinitrophenol	51-28-5	42-150	0-40	1.0	2.0
2,4-Dinitrotoluene	121-14-2	45-140	0-40	0.25	0.5
2,6-Dinitrotoluene	606-20-2	55-150	0-40	0.25	0.5
Di-n-Octyl Phthalate	117-84-0	50-150	0-40	1.0	2.0
Fluoranthene	206-44-0	60-148	0-40	0.25	0.5
Fluorene	86-73-7	59-136	0-40	0.25	0.5
Hexachlorobenzene	118-74-1	50-130	0-40	0.25	0.5
Hexachlorobutadiene	87-68-3	30-120	0-40	0.25	0.5
Hexachlorocyclopentadiene	77-47-4	20-143	0-40	1.0	2.0
Hexachloroethane	67-72-1	40-107	0-40	0.25	0.5
Indeno[1,2,3-cd]pyrene	193-39-5	50-147	0-40	0.25	0.5
Isophorone	78-59-1	31-124	0-40	0.25	0.50
2-Methylnaphthalene	91-57-6	35-120	0-40	0.25	0.50
2-Methylphenol	95-48-7	34-120	0-40	0.25	0.50
4-Methylphenol	106-44-5	25-120	0-40	0.25	0.50
Naphthalene	91-20-3	35-120	0-40	0.25	0.50
2-Nitroaniline	88-74-4	44-131	0-40	1.0	2.0
3-Nitroaniline	99-09-2	42-136	0-40	0.50	1.00
4-Nitroaniline	100-01-6	50-146	0-40	1.0	2.0
Nitrobenzene	98-95-3	42-106	0-40	0.25	0.50
2-Nitrophenol	88-75-5	39-110	0-40	0.25	0.50
4-Nitrophenol	100-02-7	10-132	0-40	1.0	2.0
n-Nitrosodiphenylamine	86-30-6	50-138	0-40	0.25	0.50
n-Nitrosodipropylamine	621-64-7	40-142	0-40	0.25	0.50
Pentachlorophenol	87-86-5	50-149	0-40	1.0	2.0
Phenanthrene	85-01-8	50-133	0-40	0.25	0.50
Phenol	108-95-2	10-120	0-40	0.25	0.50
Pyrene	129-00-0	60-137	0-40	0.25	0.50
1,2,4-Trichlorobenzene	120-82-1	44-100	0-40	0.25	0.50
2,4,5-Trichlorophenol	95-95-4	48-132	0-40	0.25	0.50
2,4,6-Trichlorophenol	88-06-2	50-135	0-40	0.25	0.50

TABLE 4B
MICROBAC'S QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF METHOD 625

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
1,2,4-Trichlorobenzene	120-82-1	44-105	30	2.5	5
2,4,6-Trichlorophenol	88-06-2	37-120	30	2.5	5
2,4-Dichlorophenol	120-83-2	39-110	30	2.5	5
2,4-Dimethylphenol	105-67-9	32-119	30	2.5	5
2,4-Dinitrophenol	51-28-5	20-140	30	12.5	25
2,4-Dinitrotoluene	121-14-2	40-139	30	2.5	5
2,6-Dinitrotoluene	606-20-2	50-120	30	2.5	5
2-Chloronaphthalene	91-58-7	60-118	30	2.5	5
2-Chlorophenol	95-57-8	23-134	30	2.5	5
2-Nitrophenol	88-75-5	29-120	30	2.5	5
3,3'-Dichlorobenzidine	91-94-1	30-140	30	2.5	5
4,6-Dinitro-2-Methylphenol	534-52-1	40-145	30	12.5	25
4-Bromophenyl Phenyl Ether	101-55-3	53-127	30	2.5	5
4-Chloro-3-Methylphenol	59-50-7	22-147	30	2.5	5
4-Chlorophenyl Phenyl Ether	7005-72-3	25-158	30	2.5	5
4-Nitrophenol	100-02-7	10-132	30	12.5	25
Acenaphthene	83-32-9	47-145	30	2.5	5
Acenaphthylene	208-96-8	33-145	30	2.5	5
Anthracene	120-12-7	27-133	30	2.5	5
Benzidine	92-87-5	0-155	30	12.5	25
Benzo[a]anthracene	56-55-3	33-143	30	2.5	5
Benzo[a]pyrene	50-32-8	40-135	30	2.5	5
Benzo[b]fluoranthene	205-99-2	40-135	30	2.5	5
Benzo[ghi]perylene	191-2-4-2	40-140	30	2.5	5
Benzo[k]fluoranthene	207-08-9	50-140	30	2.5	5
bis(2-Chloroethoxy)methane	111-91-1	33-105	30	2.5	5
bis(2-Chloroethyl)ether	111-44-4	25-120	30	2.5	5
bis(2-Chloroisopropyl)ether	108-60-1	36-130	30	2.5	5
bis(2-Ethylhexyl)phthalate	117-81-7	45-150	30	2.5	5
Butyl Benzyl Phthalate	85-68-7	50-150	30	2.5	5
Chrysene	218-01-9	50-130	30	2.5	5
Dibenz[ah]anthracene	53-70-3	40-125	30	2.5	5
Diethylphthalate	84-66-2	40-114	30	2.5	5
Dimethylphthalate	131-11-3	25-112	30	2.5	5

TABLE 4B (continued)
MICROBAC'S QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF METHOD 625

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
Di-n-Butyl Phthalate	84-74-2	55-118	30	2.5	5
Di-n-Octyl Phthalate	117-84-0	35-146	30	2.5	5
Fluoranthene	206-44-0	45-137	30	2.5	5
Fluorene	86-73-7	59-121	30	2.5	5
Hexachlorobenzene	118-74-1	50-130	30	2.5	5
Hexachlorobutadiene	87-68-3	25-105	30	2.5	5
Hexachlorocyclopentadiene	77-47-4	20-143	30	2.5	5
Hexachloroethane	67-72-1	40-113	30	2.5	5
Indeno[1,2,3-cd]pyrene	193-39-5	45-135	30	2.5	5
Isophorone	78-59-1	25-110	30	2.5	5
Naphthalene	91-20-3	21-133	30	2.5	5
Nitrobenzene	98-95-3	35-110	30	2.5	5
n-Nitrosodimethylamine	62-75-6	20-110	30	2.5	5
n-Nitrosodiphenylamine	86-30-6	40-110	30	2.5	5
n-Nitrosodipropylamine	621-64-7	25-130	30	2.5	5
Pentachlorophenol	87-86-5	35-140	30	12.5	25
Phenanthrene	85-01-8	54-120	30	2.5	5
Phenol	108-95-2	10-110	30	2.5	5
Pyrene	129-00-0	52-115	30	2.5	5
SURROGATES					
2-Fluorobiphenyl	321-60-8	43-116	NA		
2-Fluorophenol	367-12-4	21-100	NA		
2,4,6-Tribromophenol	118-79-6	10-123	NA		
Nitrobenzene-d5	4165-60-0	35-114	NA		
Phenol-d5	13127-88-3	10-94	NA		
p-Terphenyl-d14	1718-51-0	33-141	NA		

TABLE 5
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF SOIL AND SOLID WASTE BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
0,0,0-Triethyl Phosphorothioate	126-68-1	40-140 (1)	40	82.5	165
1,1'-Biphenyl	92-52-4	40-140 (1)	40	82.5	165
1,2,4,5-Tetrachlorobenzene	95-94-3	40-140 (1)	40	82.5	165
1,2,4-Trichlorobenzene	120-82-1	35-100	40	82.5	165
1,2-Dichlorobenzene	95-50-1	35-95	40	82.5	165
1,2-Diphenylhydrazine	122-66-7	25-150	40	82.5	165
1,3-Dichlorobenzene	541-73-1	35-100	40	82.5	165
1,3-Dinitrobenzene	99-65-0	10-120	40	82.5	165
1,4-Dichlorobenzene	106-46-7	35-105	40	82.5	165
1,4-Dioxane (3)	123-93-1	40-140	40	50	100
1,4-Naphthoquinone	130-15-4	40-140 (1)	40	82.5	165
1-Chloronaphthalene	90-13-1	40-140 (1)	40	82.5	165
1-Methylnaphthalene	90-12-0	35-100	40	82.5	165
1-Naphthylamine	134-32-7	40-140 (1)	40	375	750
2,3,4,6-Tetrachlorophenol	58-90-2	40-140	40	82.5	165
2,4,5-Trichlorophenol	95-95-4	40-110	40	82.5	165
2,4,6-Trichlorophenol	88-06-2	40-110	40	82.5	165
2,4-Dichlorophenol	120-83-2	35-110	40	82.5	165
2,4-Dimethylphenol	105-67-9	30-105	40	82.5	165
2,4-Dinitrophenol	51-28-5	40-130	40	412	825
2,4-Dinitrotoluene	121-14-2	50-130	40	82.5	165
2,6-Dichlorophenol	87-65-0	40-140 (1)	40	82.5	165
2,6-Dinitrotoluene	606-20-2	50-125	40	82.5	165
2-Acetylaminofluorene	53-96-3	40-140 (1)	40	82.5	165
2-Chloronaphthalene	91-58-7	40-105	40	82.5	165
2-Chlorophenol	95-57-8	35-105	40	82.5	165
2-Methylnaphthalene	91-57-6	35-115	40	82.5	165
2-Methylphenol	95-48-7	35-100	40	82.5	165
2-Naphthylamine	91-59-8	40-140 (1)	40	412	825
2-Nitroaniline	88-74-4	45-120	40	415	825
2-Nitrophenol	88-75-5	35-100	40	82.5	165
2-Picoline	109-06-8	40-140 (1)	40	82.5	165
3-, 4-Methylphenol	108-39-4/106-44-5	35-105	40	82.5	165
3,3'-Dichlorobenzidine	91-94-1	40-140	40	165	330
3,3-Dimethylbenzidine	119-93-7	10-120 (1)	40	412	825
3-Methylcholanthrene	56-49-5	40-140 (1)	40	82.5	165

TABLE 5 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF SOIL AND SOLID WASTE BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
3-Nitroaniline	99-09-2	50-130	40	412	825
4,6-Dinitro-2-Methylphenol	534-52-1	45-130	40	412	825
4-Aminobiphenyl	92-67-1	40-140 (1)	40	375	750
4-Bromophenyl Phenyl Ether	101-55-3	40-115	40	82.5	165
4-Chloro-3-Methylphenol	59-50-7	40-100	40	82.5	165
4-Chloroaniline	106-47-8	35-100	40	82.5	165
4-Chlorophenyl Phenyl Ether	7005-72-3	40-110	40	82.5	165
4-Nitroaniline	100-01-6	35-140	40	412	825
4-Nitrophenol	100-02-7	45-140	40	412	825
4-Nitroquinoline 1-Oxide	56-57-5	40-140 (1)	40	412	825
5-Nitro-o-Toluidine	99-55-8	40-140 (1)	40	82.5	165
7,12-Dimethylbenz[a]anthracene	57-97-6	40-140 (1)	40	625	1250
a,a-Dimethylphenethylamine	122-09-8	10-120 (1)	40		825
Acenaphthene	83-32-9	40-110	40	82.5	165
Acenaphthylene	208-96-8	40-110	40	82.5	165
Acetophenone	98-86-2	40-140	40	82.5	165
Aniline	62-53-3	20-100	40	412	825
Anthracene	120-12-7	55-130	40	82.5	165
Aramite	140-57-8	40-140 (1)	40		165
Atrazine	1912-24-9	40-140 (1)	40	82.5	165
Benzaldehyde	100-52-7	40-140 (1)	40	82.5	165
Benzidine	92-87-5	10-120	40	625	1250
Benzo[a]anthracene	56-55-3	50-130	40	82.5	165
Benzo[a]pyrene	50-32-8	50-130	40	82.5	165
Benzo[b]fluoranthene	205-99-2	45-125	40	82.5	165
Benzo[ghi]perylene	191-2-4-2	40-140	40	82.5	165
Benzo[k]fluoranthene	207-08-9	45-135	40	82.5	165
Benzoic Acid	65-85-0	20-110	40	330	5000
Benzyl Alcohol	100-51-6	30-100	40	82.5	165
bis(2-Chloroethoxy)methane	111-91-1	30-100	40	82.5	165
bis(2-Chloroethyl)ether	111-44-4	30-100	40	82.5	165
bis(2-Chloroisopropyl)ether	108-60-1	20-115	40	82.5	165
bis(2-Ethylhexyl)phthalate	117-81-7	50-150	40	82.5	165
Butyl Benzyl Phthalate	85-68-7	50-150	40	82.5	165
Caprolactam	105-60-2	40-140 (1)	40	82.5	165
Carbazole	86-74-8	50-140	40	82.5	165
Chlorobenzilate	510-15-6	40-140 (1)	40	82.5	165
Chrysene	218-01-9	55-140	40	82.5	165

TABLE 5 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF SOIL AND SOLID WASTE BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
Diallate	2303-16-4	40-140 (1)	40	375	750
Dibenz[ah]anthracene	53-70-3	40-140	40	82.5	165
Dibenzofuran	132-64-9	35-110	40	82.5	165
Diethylphthalate	84-66-2	50-130	40	82.5	165
Dimethoate	60-51-5	40-140 (1)	40	82.5	165
Dimethylphthalate	131-11-3	45-115	40	82.5	165
Di-n-Butyl Phthalate	84-74-2	55-118	40	82.5	165
Di-n-Octyl Phthalate	117-84-0	35-146	40	82.5	165
Disulfoton	298-04-4	40-140 (1)	40	412	825
Ethyl Methanesulfonate	62-50-0	40-140 (1)	40	82.5	165
Famphur (Estimated)	52-85-7	(2)	NA	NA	2500
Fluoranthene	206-44-0	55-140	40	82.5	165
Fluorene	86-73-7	45-115	40	82.5	165
Hexachlorobenzene	118-74-1	45-120	40	82.5	165
Hexachlorobutadiene	87-68-3	30-100	40	82.5	165
Hexachlorocyclopentadiene	77-47-4	30-110	40	165	330
Hexachloroethane	67-72-1	30-100	40	82.5	165
Hexachlorophene (Estimated)	70-30-4	(2)	NA	NA	1650
Hexachloropropene	1888-71-7	40-140 (1)	40	82.5	165
Indeno[1,2,3-cd]pyrene	193-39-5	50-135	40	82.5	165
Isodrin	78-59-1	40-140 (1)	40	82.5	825
Isophorone	78-59-1	35-100	40	82.5	165
Isosafrole	120-58-1	40-140 (1)	40	375	750
Kepone (Estimated)	143-50-0	(2)	NA	NA	1650
Methapyrilene	91-80-5	40-140 (1)	40	412	825
Methyl Methanesulfonate	66-27-3	40-140 (1)	40	82.5	165
Naphthalene	91-20-3	35-100	40	82.5	165
Nitrobenzene	98-95-3	35-100	40	82.5	165
n-Nitrosodiethylamine	55-18-5	40-140 (1)	40	82.5	165
n-Nitrosodimethylamine	62-75-6	25-100	40	82.5	165
n-Nitrosodi-n-Butylamine	924-16-3	40-140 (1)	40	82.5	165
n-Nitrosodiphenylamine	86-30-6	50-130	40	82.5	165
n-Nitrosodipropylamine	621-64-7	35-110	40	82.5	165
n-Nitrosomethylethylamine	10595-95-6	40-140 (1)	40	82.5	165
n-Nitrosomorpholine	59-89-2	40-140 (1)	40	82.5	165
n-Nitrosopiperidine	100-75-4	40-140 (1)	40	82.5	165
n-Nitrosopyrrolidine	930-55-2	40-140 (1)	40	82.5	165

TABLE 5 (continued)
QA OBJECTIVES FOR SEMIVOLATILE ORGANIC ANALYSIS OF SOIL AND SOLID WASTE BY GC/MS

Compound	CAS #	ACCURACY (% RECOVERY)	PRECISION (%RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)
o-Toluidine	95-53-4	40-140 (1)	40	82.5	165
p-(Dimethylamino)azobenzene	60-11-7	40-140 (1)	40	82.5	165
Parathion Ethyl	56-38-2	40-140 (1)	40	82.5	165
Parathion Methyl	298-00-0	40-140 (1)	40	82.5	165
Pentachlorobenzene	608-93-5	40-140	40	82.5	165
Pentachloroethane	76-01-7	40-140 (1)	40	82.5	165
Pentachloronitrobenzene	82-68-8	40-140	40	82.5	165
Pentachlorophenol	87-86-5	50-150	40	412	825
Phenacetin	62-44-2	40-140 (1)	40	82.5	165
Phenanthrene	85-01-8	50-130	40	82.5	165
Phenol	108-95-2	35-100	40	82.5	165
Phorate	298-02-2	40-140 (1)	40	82.5	165
p-Phenylenediamine (Estimated)	106-50-3	40-140 (1)	40	NA	825
Pronamide	23950-58-5	40-140 (1)	40	82.5	165
Pyrene	129-00-0	45-135	40	82.5	165
Pyridine	110-86-1	20-100	40	412	825
Safrole	94-59-7	40-140 (1)	40	82.5	165
Sulfotepp	3689-24-5	40-140 (1)	40	82.5	165
Sym-Trinitrobenzene	99-35-4	40-140 (1)	40	82.5	165
Thionazin	297-97-2	40-140 (1)	40	82.5	165
SURROGATES					
2-Fluorobiphenyl	321-60-8	30-115	NA		
2-Fluorophenol	367-12-4	25-121	NA		
2,4,6-Tribromophenol	118-79-6	19-122	NA		
Nitrobenzene-d5	4165-60-0	23-120	NA		
Phenol-d5	13127-88-3	24-113	NA		
p-Terphenyl-d14	1718-51-0	18-137	NA		

(1) Compound not routinely spiked into batch QC, may be added upon request.

(2) Not spiked and/or evaluated.

(3) See Appendix III

TABLE 5A
MICROBAC'S QA OBJECTIVES FOR LOW LEVEL SEMIVOLATILE ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	ACCURACY (% RECOVERY)*	PRECISION (%RPD)*	VERIFIED MDL (ug/L)	REPORTING LIMITS (ug/L)
Acenaphthene	83-32-9	35-110	0-50	12.5	25
Acenaphthylene	208-96-8	35-110	0-50	12.5	25
Anthracene	120-12-7	55-130	0-50	12.5	25
Benzo[a]anthracene	56-55-3	50-120	0-50	12.5	25
Benzo[a]pyrene	50-32-8	50-130	0-50	12.5	25
Benzo[b]fluoranthene	205-99-2	45-125	0-50	12.5	25
Benzo[ghi]perylene	191-24-2	40-140	0-50	12.5	25
Benzo[k]fluoranthene	207-08-9	45-135	0-50	12.5	25
Benzyl Alcohol	100-51-6	29-100	0-50	12.5	25
bis(2-Chloroethoxy)methane	111-91-1	27-100	0-50	12.5	25
bis(2-Chloroethyl)ether	111-44-4	29-100	0-50	12.5	25
bis(2-Chloroisopropyl)ether	108-60-1	20-115	0-50	12.5	25
bis(2-Ethylhexyl)phthalate	117-81-7	50-150	0-50	12.5	25
4-Bromophenyl Phenyl Ether	101-55-3	35-130	0-50	12.5	25
Butyl Benzyl Phthalate	85-68-7	40-150	0-50	12.5	25
4-Chloroaniline	106-47-8	25-100	0-50	12.5	25
4-Chloro-3-Methylphenol	59-50-7	31-100	0-50	12.5	25
2-Chloronaphthalene	91-58-7	35-115	0-50	12.5	25
2-Chlorophenol	95-57-8	30-115	0-50	12.5	25
4-Chlorophenyl Phenyl Ether	7005-72-3	36-110	0-50	12.5	25
Chrysene	218-01-9	55-140	0-50	12.5	25
Dibenz[ah]anthracene	53-70-3	40-140	0-50	12.5	25
Dibenzofuran	132-64-9	35-110	0-50	12.5	25
Di-n-Butyl Phthalate	84-74-2	55-140	0-50	50	100
1,2-Dichlorobenzene	95-50-1	30-105	0-50	12.5	25
1,3-Dichlorobenzene	541-73-1	30-110	0-50	12.5	25
1,4-Dichlorobenzene	106-46-7	30-105	0-50	12.5	25
3,3'-Dichlorobenzidine	91-94-1	40-150	0-50	12.5	25
2,4-Dichlorophenol	120-83-2	30-120	0-50	12.5	25
Diethylphthalate	84-66-2	50-130	0-50	12.5	25
2,4-Dimethylphenol	105-67-9	30-105	0-50	12.5	25

TABLE 5A(continued)
MICROBAC'S QA OBJECTIVES FOR LOW LEVEL SEMIVOLATILE ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	ACCURACY (% RECOVERY)*	PRECISION (%RPD)*	VERIFIED MDL (ug/L)	REPORTING LIMITS (ug/L)
Dimethylphthalate	131-11-3	40-120	0-50	12.5	25
4,6-Dinitro-2-Methylphenol	534-52-1	43-130	0-50	25.0	50
2,4-Dinitrophenol	51-28-5	40-130	0-50	25.0	50
2,4-Dinitrotoluene	121-14-2	50-130	0-50	12.5	25
2,6-Dinitrotoluene	606-20-2	45-125	0-50	12.5	25
Di-n-Octyl Phthalate	117-84-0	40-145	0-50	12.5	25
Fluoranthene	206-44-0	45-125	0-50	12.5	25
Fluorene	86-73-7	40-115	0-50	12.5	25
Hexachlorobenzene	118-74-1	45-120	0-50	12.5	25
Hexachlorobutadiene	87-68-3	30-100	0-50	12.5	25
Hexachlorocyclopentadiene	77-47-4	24-100	0-50	12.5	25
Hexachloroethane	67-72-1	30-90	0-50	12.5	25
Indeno[1,2,3-cd]pyrene	193-39-5	40-135	0-50	12.5	25
Isophorone	78-59-1	30-110	0-50	12.5	25
2-Methylnaphthalene	91-57-6	29-85	0-50	12.5	25
2-Methylphenol	95-48-7	31-100	0-50	12.5	25
4-Methylphenol	106-44-5	30-120	0-50	12.5	25
Naphthalene	91-20-3	30-120	0-50	12.5	25
2-Nitroaniline	88-74-4	32-120	0-50	25	50
3-Nitroaniline	99-09-2	34-130	0-50	25	50
4-Nitroaniline	100-01-6	35-140	0-50	25	50
Nitrobenzene	98-95-3	30-115	0-50	12.5	25
2-Nitrophenol	88-75-5	30-100	0-50	12.5	25
4-Nitrophenol	100-02-7	36-140	0-50	25	50
n-Nitrosodiphenylamine	86-30-6	40-125	0-50	12.50	25
n-Nitrosodipropylamine	621-64-7	31-110	0-50	12.50	25
Pentachlorophenol	87-86-5	47-150	0-50	25.0	50
Phenanthrene	85-01-8	42-130	0-50	12.50	25
Phenol	108-95-2	30-110	0-50	12.50	25
Pyrene	129-00-0	45-135	0-50	12.50	25
1,2,4-Trichlorobenzene	120-82-1	30-105	0-50	12.50	25
2,4,5-Trichlorophenol	95-95-4	35-110	0-50	12.50	25
2,4,6-Trichlorophenol	88-06-2	35-120	0-50	12.50	25

Table 6
Quality Control Criteria
Semivolatile GC/MS Analysis
Method 8270C

Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass spectral ion intensities (DFTPP criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 2	Retune instrument and repeat DFTPP check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered	<30% RSD for CCC compounds, >0.05 average RF for SPCC compounds, <15% RSD for all target compounds, if >15% RSD, then linear regression, if r^2 is <0.990, then quadratic regression, or mean % RSD <15%	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1)
Second Source calibration	After each initial calibration	≤30% drift for each analyte (90% of analytes must pass) or project specific criteria	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Every 12 hours	SPCC RF ≥0.05: CCC %D ≤20%; Other compounds: %RSD ≤40, 90% of compound list must meet this criteria; or project-specific criteria.	CCC/SPCC: Re-analyze CCV; upon second failure repeat calibration. Other compounds: repeat initial calibration if compound fails in more than two consecutive analytical batches (1)
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 sec of IS retention time in ICAL midpoint std and area within –50% to+100% of IS midpoint area.	Check for MS malfunctions or interference; re-analyze sample in accordance with Section 13.7
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	< project reporting limit for each target analyte	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.4
Laboratory Control Sample (LCS)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.5
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated range.	Qualify data and/or address in the report narrative.
Surrogate spike	Every sample, standard and quality control sample	Recoveries within the designated ranges; use project QAAP or standard control criteria (1)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.8

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

Table 6A
Quality Control Criteria
Semivolatile GC/MS Analysis
Method 8270D and 8270 Low Level

Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass spectral ion intensities (DFTPP criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 2	Retune instrument and repeat DFTPP check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered	≤ 20% RSD for all compounds Average RF > minimum values in Table 7, If >20% RSD, then linear regression, If R^2 <0.990 then quadratic regression. No more than 10% may fail.	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1)
Second Source calibration	After each initial calibration	≤ 30% for all compounds. No more than 10% may fail.	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Every 12 hours	≤ 20% for all compounds. RF > minimum values in Table 7. No more than 20% may fail.	CCC/SPCC: Re-analyze CCV; upon second failure repeat calibration. Other compounds: repeat initial calibration if compound fails in more than two consecutive analytical batches (1)
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 sec of IS retention time in ICAL midpoint std and area within –50% to+100% of IS midpoint area.	Check for MS malfunctions or interference; re-analyze sample in accordance with Section 13.7
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	< project reporting limit for each target analyte	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.4
Laboratory Control Sample (LCS)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.5
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated range.	Qualify data and/or address in the report narrative.
Surrogate spike	Every sample, standard and quality control sample	Recoveries within the designated ranges; use project QAAP or standard control criteria (1)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.8

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

Table 6B
Quality Control Criteria
Semivolatile GC/MS Analysis
Method 625

Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass spectral ion intensities (DFTPP criteria)	Every 24 hours prior to ICAL, ICV or CCV	See Table 2	Retune instrument and repeat DFTPP check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered	<35% RSD for all compounds	Evaluate and use linear or higher order calibration equation, recalibrate instrument; or qualify and discuss in narrative (1)
Second Source calibration verification (ICV)	After each initial calibration	<30% drift for each analyte or project specific criteria	Re-analyze ICV; upon second failure, Repeat initial calibration (1)
Continuing calibration verification (CCV)	Every 24 hours	<20% difference	Re-analyze CCV; upon second failure, repeat initial calibration (1)
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 sec of retention time is IS in ICAL midpoint std and area within –50% to +100% of IS midpoint area.	Check for MS malfunctions or interference; re-analyze sample in accordance with Section 13.7
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	< project reporting limit for each target analyte	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.3.
Laboratory Control Sample (LCS)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.4
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated range.	Qualify data and/or address in the report narrative.
Surrogate spike	Every sample, standard and quality control sample	Recoveries within the designated ranges; use project QAAP or standard control criteria (1)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.7

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

Table 7
8270D and 8270 Low Level Minimum Response Factors

Semivolatile Compounds	Minimum Response Factor (RF)
Phenol	0.800
Bis (2-chloroethyl) ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
Bis(2-chloro)isopropyl ether	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
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
2-Chloronaphthalene	0.800
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
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	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
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

Table 7 (continued)
8270D and 8270 Low Level Minimum Response Factors

Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhezyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
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

Figure 1

Run Log ID:23327

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HPMS12 Dataset: 072108
Analyst1: MES Analyst2: NA
Method: 8270C SOP: MSS01 Rev: 14
Method: 625 SOP: MSS02 Rev: 8

Maintenance Log ID: 25077

Column 1 ID: RXI-5MS Column 2 ID: NA
Workgroups: WG277561,WG277490,WG277489
Internal STD: COA13174 Surrogate STD: NA Calibration STD

Comments:

Seq.	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	12M22745	WG277487-01 50PPM DFTPP STD	1	1	STD25193	07/21/09 08:33
2	12M22746	WG277487-02 50PPM MEGAMIX STD	1	1	STD27564	07/21/09 08:52
3	12M22747	WG277256-01 FBLK 7/17	17	1		07/21/09 09:25
4	12M22748	WG277261-01 SPLP BLANK 7/17	16	1		07/21/09 09:59
5	12M22749	WG277469-01 BLANK V10009 PG25	17	1		07/21/09 10:33
6	12M22750	WG277469-02 LCS V10009 PG25	17	1		07/21/09 11:07
7	12M22751	WG277469-03 LCSDUP V10009 PG25	17	1		07/21/09 11:41
8	12M22752	L08070386-02 SOIL	7	1	SOIL	07/21/09 12:15
9	12M22753	L08070386-04 SOIL	7	1	SOIL	07/21/09 12:50
10	12M22754	L08070358-01 TCLP	17	1		07/21/09 13:24
11	12M22755	L08070358-03 TCLP	17	1		07/21/09 13:58
12	12M22756	L08070358-06 TCLP	17	1		07/21/09 14:33
13	12M22757	L08070358-09 TCLP	17	1		07/21/09 15:07
14	12M22758	L08070406-01 TCLP	17	1		07/21/09 15:42
15	12M22759	L08070406-02 TCLP	17	1		07/21/09 16:17
16	12M22760	L08070406-03 TCLP	17	1		07/21/09 16:51
17	12M22761	L08070406-04 TCLP	17	1		07/21/09 17:25
18	12M22762	L08070406-05 TCLP	17	1		07/21/09 17:59
19	12M22763	L08070386-03 SPLP	16	1		07/21/09 18:33
20	12M22764	L08070386-05 SPLP	16	1		07/21/09 19:07
21	12M22765	L08070374-01 10X TCLP	17	10		07/21/09 19:40
22	12M22766	BAKE OUT	17	1		07/21/09 20:14
23	12M22767	BAKE OUT	17	1		07/21/09 20:47

Comments

Seq.	Rerun	Dil	Reason	Analytes
2				
	08 >30% but <40% low and 55 >40% high.			
7				
	PHL is high.			
10				
	PHN and FRY are low due to SMI			
12				
	2FP and PHL are >10%. OK per very basic matrix.			
21	X	2	Analyzed too dilute	

Page: 1 Approved: 22-JUL-09

Figure 2

Checklist ID: 30380

Microbac Laboratories Inc.

Data Checklist

Date: 21-JUL-2008
Analyst: MES
Analyst: NA
Method: 8270
Instrument: HPMS12
Curve Workgroup: NA
Runlog ID: 23327
Analytical Workgroups: L08070374,L08070386,L08070358,L08070406

ANALYTICAL	
System Performance Check	X
DFTPP (MS)	X
Endrin/DDT breakdown (8081/MS)	X
Pentachlorophenol/benzidine tailing (MS)	X
Eluent check (IC)/system pressure (HPLC)	NA
Window standard (FID)	NA
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	X
Continuing calibration blank (CCB) (IC)	NA
Special standards	NA
Blanks	X
TCL hits	X
Surrogate recoveries	X
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	X
MS/MSD/Sample duplicates	NA
Recoveries	NA
%RPD	NA
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	X
Surrogate recoveries	X
Internal standard areas (MS)	X
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	NA
Reruns	X
Manual integrations	X
Project/client specific requirements	X
REPORTING	
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	MES
SUPERVISORY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer: 22-JUL-2008

Secondary Reviewer: 22-JUL-2008

CHECKLIST1 - Modified 03/05/2008
Generated: JUL-22-2008 10:27:49

Appendix I
Low level 8270

For low-level 8270 Analysis (8270UL), the following modifications are made to SOP MSS01:

1.4 The reporting limit (RL) of Method 8270UL for determining an individual compound is approximately 25 ug/kg (wet weight) for soil/sediment samples and 0.5 ug/L for groundwater samples. QC criteria may be found in Tables 4A, 5A and 6A.

7.3 Internal standard solution is diluted from 4000 ug/mL to 100 ug/mL. Each 1 mL of sample extract is spiked with 10 uL of the internal standard solution, resulting in a concentration of 1.0 ng/uL of each internal standard.

7.6 The 50 ug/mL DFTPP solution is diluted to a concentration of 5 ug/mL.

7.8 Calibration standards: A 20 ug/mL intermediate calibration standard is prepared from the 200 ug/mL standard by diluting 1 mL of the standard with 9 mL of $MeCl_2$. The remaining calibration standards are prepared from the intermediate using the following scheme:

15 ug/mL (750 uL stock: 250 uL $MeCl_2$)
10 ug/mL (500 uL stock: 500 uL $MeCl_2$)
5.0 ug/mL (250 uL stock: 750 uL $MeCl_2$)
2.0 ug/mL (100 uL stock: 900 uL $MeCl_2$)
1.0 ug/mL (100 uL 10ppm standard: 900 uL $MeCl_2$)
0.5 ug/mL (100 uL 5ppm standard: 900 uL $MeCl_2$)

1.1 A 5.0 ug/mL alternate source standard is prepared from the intermediate standard in Section 7.8 (above).

8.2 Recommended GC/MS conditions:

Split Time	1.0 minute
Sample volume	2.0 uL

The electron multiplier voltage should be set at a level that does not allow detector saturation when the 20 ug/mL standard is analyzed, typically 50-100 mV below the voltage set by the autotune program.

8.2.3 2.0 uL of the 5 ug/mL DFTPP solution is injected.

8.2.5 The initial calibration curve is established by analyzing 2.0 uL of each calibration standard.

8.2.7 The average minimum response factors must meet the criteria listed in Table 7.

8.2.8, 8.2.9 The RSD should be ≤ 20% for each compound. Due to the large number of compounds that may be analyzed, some may fail to meet this criteria. If the failing compounds are not critical to the project, analysis may proceed and results should be flagged as estimated. If the RSD is >20%, a linear or quadratic regression curve must be used; the R^2 value must be at least 0.990. If more than 10% of the compounds in the calibration fail to meet the %RSD and curve fit criteria, the system is too unstable for analysis.

8.2.10, 8.3.1, 8.3.2 The concentration of the DFTPP solution, continuing calibration standard and alternate source standard is 5.0 ug/mL.

8.3.2 SPCC/CCC criteria are not applicable; see section 8.3.3 and 8.3.4.

8.3.3 The compounds must meet the minimum response factors listed in Table 7.

8.3.4 The percent difference or percent drift for each compound should be less than 20%. If more than 20% of the compounds in the standard fail this criteria, corrective action must be taken.

17.2 SW-846 Method 8270D Revision 4, February 2007.

Appendix II
Method 625

The following changes are made to this SOP for the analysis of 40 CFR 136 Method 625:

1.4 Method 625 is applicable to water samples only. QC criteria may be found in Tables 4B and 6B.

8.2.5, 8.2.6 A minimum of three calibration points must be analyzed.

8.2.7, 8.3.3 Not applicable.

8.2.8 There are no Continuing Calibration Check (CCC) requirements. The %RSD must be <35 for all compounds.

8.2.9 If the %RSD of a compound is <35%, the average response factor may be used for quantitation.

8.4.4 The percent drift for every compound must be <20%.

1.2 Not applicable.

Appendix III
Analysis of 1,4-Dioxane

2.0 This appendix describes a two-part process that allows reporting of 1,4-dioxane with a reporting limit (RL) of 1.0 ug/L for water and 100 ug/kg for soil.

3.0 Initial analysis

3.1 Samples are initially analyzed using the conventional method parameters. Using theses conditions, 1,4-dioxane does not respond reliably below 10 ug/mL. To support this initial reporting limit, an additional calibration standard at 10 ug/mL is added to the working calibration standards from Section 7.8:

3.2 ug/mL (500uL stock:950uL $MeCl_2$)

3.3 An alternative source standard is purchased from Accustandard (ALR-062S) at a concentration of 100 ug/mL. 500 uL of this standard is diluted in an autosampler vial with 500 uL of methylene chloride for a working concentration of 50 ug/mL.

3.4 If 1,4-dioxane is detected in a sample at a concentration >10 ug/L, low-level analysis will be unnecessary. If 1,4-dioxane is not detected, the samples will be re-analyzed using selective ion monitoring (SIM).

4.0 Low-level analysis

4.1 Standards

4.1.1 Primary calibration standard is purchased from Restek (31853) at a concentration of 2000 ug/mL. 500 uL of this standard is added to a 10 mL volumetric flask and brought to volume with methylene chloride to yield a 100 ug/mL intermediate standard.

4.1.2 Internal standard 1,4-dichlorobenzene-d4 is purchased from Accustandard (Z-104J-3) at a concentration of 4000 ug/mL. This standard is diluted to a working concentration of 100 ug/mL as in Appendix I, Section 7.3. Each standard and sample is spiked with this standard in the ration of 10 uL ISTD:1 mL sample.

4.1.3 Calibration standards are prepared from the intermediate standard using the following scheme:

1. 10 ug/mL (100uL stock:900uL $MeCl_2$)
2. 7.5 ug/mL (75uL stock:925uL $MeCl_2$)
3. 5.0 ug/mL (500uL std. #1:500uL $MeCl_2$)
4. 2.5 ug/mL (500uL std. #3:500uL $MeCl_2$)
5. 1.0 ug/mL (100uL std. #1:900uL $MeCl_2$)

3.1.4 100 uL of the alternate standard from Section 2.2 above is dilution with 900 uL $MeCl_2$) to yield a 5 ug/ml working standard.

4.2 The 5 ug/mL DFTPP standard from Appendix I, Section 7.6 is used for tuning.

Other aspects of this analysis are the same as in the main body of MSS01, with these exceptions:

3.2 Calibration:

The 10 ug/mL standard is initially analyzed using full scan to establish retention times for the SIM parameters. The SIM acquisition table is set up to scan for the target ions for 1,4-dioxane and 1,4-dichlorobenzene-d4 (Table 1) during the appropriate retention times and the calibration is analyzed using these parameters.

4.3 Analytical procedures:

The method blank and any non-detect samples are re-prepped for analysis using the 1.0 ng/uL internal standard mix from Appendix I, Section 7.3; an additional low-level LCS is also extracted with the batch and is prepared and analyzed in the same manner.

Appendix IV
South Caroline Requirements

The quadratic regression calibration option may not be used for samples analyzed for the state of South Carolina.

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STANDARD OPERATING PROCEDURE
Low Level PAHs
METHOD 8270

Issue/Implementation Date: 15 February 2010

Last Review Date: 15 February 2010

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1.0 SCOPE AND APPLICATION

1.1 This modification of Method 8270C and 8270D is a capillary column gas chromatography/mass spectroscopy (GC/MS) method utilizing selective ion monitoring (SIM) to determine low level concentrations of polynuclear aromatic hydrocarbons (PAHs) in extracts prepared from solid matrices, soil and water. See Table 1 for the target compound list.

1.2 The reporting limit (RL) for determining an individual compound is approximately 2.5 ug/Kg (wet weight) for soil/sediment samples and 0.05 ug/L for ground water samples. RLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the mass spectrometer detector; such samples may be reanalyzed by conventional Method 8270C.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method on the basis of internal audits by laboratory management and senior analysts.

2.0 SAFETY PRECAUTION

Analysts using this method must be aware of certain hazards associated with performing the analysis. Concentrated organic standards are used to calibrate the instruments. Many of these chemicals have been found to cause cancer and must be handled with extreme care. Gloves should be worn while these compounds are being used. Several zones on the GC/MS are heated to high temperatures, therefore care must be exercised when working around these areas or severe burns to the skin can occur. All dilutions that require the use of glassware must be made with care to reduce the possibility of cuts from broken glass.

3.0 SAMPLE PRESERVATION & STORAGE

3.1 Sample size and collection requirements are beyond the scope of the SOP; refer to extraction lab SOPs and SW-846 for details.

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3.2 After extraction, the samples are stored at 4° C ± 2° C until such time as the instrumental analysis can proceed. The samples must be analyzed within 40 days of the extraction.

4.0 METHOD PERFORMANCE

4.1 Table 4 summarizes the performance data for water analysis; Table 5 summarizes performance data for soil/solid waste analysis; These tables include the analyte list, ranges for accuracy and precision, nominal laboratory reporting limits (RL), and the current laboratory method detection limit(s).

4.2 The laboratory performed an initial assessment of the method detection limits (MDL) using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The limit(s) of detection (LOD), or verified MDL, are presented in Tables 4 and 5 and were established using verification procedures outlined in SOP 45.

4.4 The limit(s) of quantitation (LOQ) are the nominal laboratory reporting limit(s) (RL) and were established as per SOP 45.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. Going forward, the laboratory will use results from laboratory control samples (LCS) to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. As a quality control measure, all extraction blanks, are evaluated on all instruments that analyze samples in that extraction batch. Determination as to the source and extent of interference is essential in order to take corrective action to eliminate the problem.

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5.2 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Highly concentrated samples can cause interferences in several areas of the gas chromatograph system such as injection port systems, injection port liners, and the capillary column. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination. As a final measure to eliminate contamination, a bake-out of the gas chromatograph is effective for most hydrocarbons.

6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph system: Agilent 6890 series

- splitless injection port system
- electronic pressure control
- Agilent 7673 or 6890 autosampler and controller
- capillary column fittings and reducing nuts

6.1.2 Column – Restek Rxi-5MS or equivalent.

- 30 m x 0.32 mm ID 1 um film thickness

6.1.3 Mass spectrometer: Hewlett-Packard/Agilent 5973 Mass Selective Detector

- quadrupole analyzer
- electron impact ionization

6.1.4 Data system: Windows-based Hewlett-Packard/Agilent

- EnvironQuant Chemstation Version A.03.00 or higher
- NIST02 mass spectral library

6.2 Syringe – 5uL autosampler, 10uL, 100uL, 500uL, 1000uL Hamilton syringes or equivalent.

6.3 Volumetric flasks, Class A - 10 ml to 1000 ml

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6.4 Sample vials - glass with Teflon-lined screw caps or crimp tops. (2 ml)

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Reagent grade or better chemicals and solvents shall be used in all tests for this method. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The typical cutoff is 95% purity. All standards are stored at -10° C or less.

7.2 The stock standards are purchased as certified solution mixes at a concentration of 2000 ug/ml. Solution mixes contain all target compounds and surrogates. The stock standard is diluted in a volumetric flask for a final concentration of 10 ug/ml.

7.2.1 The stock standard solution is divided among amber 2 mL autosampler vials and sealed with crimped caps. The stock standard solution is stored at ± 10° C and protected from light to avoid evaporation and photochemical reaction. This stock standard solution should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from the mix.

7.2.2 Stock standard solutions must be replaced every six months or sooner if comparison with quality control check samples indicates a problem.

7.3 Internal standard solution is purchased as a certified mix in methylene chloride at a concentration of 4000 ug/mL and diluted to a concentration of 100 ug/mL. There are six compounds in this mix, but PAH analysis only requires five of them: naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12}. Refer to Table 1 for internal standard analyte assignment for quantitation. Samples are spiked with the internal standard mixture at a ratio of 1 mL sample: 10uL internal standard solution, resulting in a concentration of 1.0 ng/ul of each internal standard.

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7.4 GC/MS tuning standard: a methylene chloride solution containing 5 ug/mL of decafluorotriphenylphosphine (DFTPP). Store at 4° C or less when not in use.

7.5 Calibration standards: Seven calibration standards are prepared from the 10 ug/mL stock standard mix using the following scheme:

10 ug/mL = 1mL stock standard undiluted+10uL internal standard
5 ug/mL = 500uL stock: 500uL $MeCl_2$+10uL internal standard
2.5 ug/mL = 500uL 5 ug/mL standard: 500uL $MeCl_2$+5uL internal standard
1.0 ug/mL = 100uL stock: 900uL $MeCl_2$+10uL internal standard
0.5 ug/mL = 100uL 5 ug/mL standard: 900uL $MeCl_2$+9uL internal standard
0.1 ug/mL = 100uL 1.0 ug/mL standard: 900uL $MeCl_2$+9uL internal standard
0.05 ug/mL=100uL 0.5 ug/mL standard: 900uL$MeCl_2$+9uL internal standard

The high calibration point may be dropped if fewer points satisfy different method criteria (8270, AFCEE, etc.) See 8.2.8. The 0.05 ug/mL calibration standard is at a concentration equal to the reporting limit. The remaining calibration standards correspond to the working range of concentrations found in real samples but do not exceed the working range of the GC/MS system. All standards are stored at ≤-10° C and should be freshly prepared every six months or sooner if check standards indicate a problem.

8.0 CALIBRATION PROCEDURES

8.1 Mass Calibration - Prior to DFTPP analysis, initial calibration, and continuing calibration, the mass spectrometer must be tuned using the system software. This calibration allows for lens polarity and charge intensities to be set for proper mass identification. The tuning need not be repeated on a daily basis unless DFTPP does not pass or maintenance is done on the mass spectrometer.

8.2 Initial calibration recommended GC/MS conditions:

Mass range	Not applicable: see Table 1 for SIM ions
Dwell time	50 – 100ms
Scan time	1 second/scan or less
Electron multiplier	100-200V above DFTPP Autotune value
Temperature program	50° C, hold for 0.5 - 5 minutes 25° C/minute to 300° C, hold until benzo(g,h,i)perylene elutes
Injector temperature	250° C
Transfer line temperature	280 - 300° C

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Source temperature	230° C
MS Quad temperature	150° C
Injector	Splitless
Split time	0.8 - 1.0 minutes
Sample volume	2 ul
Carrier gas	Helium at 45 - 60 cm/sec

8.2.1 DFTPP is analyzed using full scan mode using the same conditions as in 8.2 with the following exceptions:

Mass range	35 – 500 amu
Scan time	1 second/scan or less
Temperature program	Initial temperature: 80° C Ramp 26° C/minute to 310° C, hold 0.8 minutes

8.2.2 Prior to analysis, a 10 ug/mL calibration standard is analyzed using the instrument parameters in 8.2 using full scan mode (mass range 35-500 amu) in order to verify the retention times of all analytes for SIM analysis.

8.2.3 DFTPP hardware tune; prior to any standard or sample analysis, DFTPP must be analyzed. 2 ul of the 5-ug/ml DFTPP solution is injected and must result in a mass spectrum which meets the criteria given in Table 2. These criteria must be demonstrated at the beginning of each 12-hour shift. Three scans (the apex of the peak, the scan immediately before the apex, and the scan immediately after the apex) are averaged and background-subtracted automatically by the instrument software.

8.2.4 The internal standards selected in section 7.3 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. If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for Naphthalene-d_8 use m/z 136 for quantitation.)

8.2.5 The initial calibration curve is established by analyzing 2.0 uL of each of the seven (7) calibration standards and calculating response factors (Rfs) for each target compound using the equation in section 12.1.

8.2.6 The response factors (RFs) and relative standard deviation (RSD) are calculated for each compound using the equation in section 12.2. The RSD should be less than 30% for each compound. For method 8270D, the RSD must be less than

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20% and each compound in each calibration standard must have a minimum response factor greater than the value shown in Table 8.

8.2.7 Calibration options

8.2.7.1 Linear – Using Average RF with RSD ≤ 15%.

The average of RF option is always the preferred method of GC/MS calibration, since linearity may be assumed throughout the full calibration range. However, linear and quadratic models may be used under the conditions discussed in the following sections. (RSD ≤20% for method 8270D).

8.2.7.2 Linear Regression with Coefficient of Determination (COD) (r^2) >0.99

Linear regression is an alternative to average RF, but has the potential for significant bias at the lower concentration levels. It should only be used when re-fitting the lowest calibration standard yields a maximum % drift of 30% (residual test).

8.2.7.3 Quadratic Calibration with COD (r^2) >0.99

Several compounds on the 8270 extended lists do not display consistently linear behavior. Quadratic calibration, employing at least six (6) calibration points, may be used to improve accuracy for these analytes, particularly at the lower calibration levels, and is a better alternative than linear regression when linear fails the re-fitting test. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and should not be used for analytes with a previous history of linear performance.

8.2.7.4 Sample analysis may not proceed without a valid calibration. If a compound fails to meet any of the above criteria, either the calibration must be repeated or the instrument may not be used for analysis of that compound.

8.2.8 Calibration points may be dropped from the upper and lower end for individual compounds so long as there are a minimum of five (5) contiguous standards (6 if quadratic regression is used) and the low standard supports the reporting limit for that compound.

8.2.9 After the initial calibration, the 1.0 ug/mL alternate source standard is analyzed and compared to the curve using the %D calculation (see Section 12.4). This is not a pass/fail but is used to evaluate the condition of the calibration standards. The %D should be ≤30. For AFCEE the %D must be ≤25. If the ICV does not

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meet acceptance criteria, the standard (from the same source) may be reanalyzed once. Upon a second failure, corrective action must be taken and the initial calibration must be repeated.

8.2.10 If there is time remaining in the 12 hour tune time, samples may be analyzed immediately following the curve.

8.3 Daily Calibration

8.3.1 Each GC/MS system must be hardware tuned with DFTPP to meet the criteria listed in Table 2.

8.3.2 A 1.0 ug/ml continuing calibration standard containing each compound of interest, surrogates, and internal standards, must be analyzed every 12 hours during analysis. The response factors from this standard are compared with the average response factors from the latest calibration curve. If any compound has a %D ≤20, the standard (from the same source) may be reanalyzed once. Upon a second failure, corrective action must be taken. Retention times of all internal standards must not shift more than 30 seconds from that in the midpoint standard of the most recent calibration curve. If no source or trouble can be found, a new initial calibration must be analyzed.

8.3.2.1 For method 8270D, the compounds must meet the minimum response factor in Table 8.

9.0 SAMPLE PREPARATION

Sample preparation - Samples for analysis by 8270C are prepared by one of the following extraction methods prior to GC/MS analysis. The most current version of the methods will be used.

Matrix	Methods	SOP #
Water	3510C, 3520C	EXB01, EXB08
Soil/Sediment	3550B, 3545	EXB02, ASE01

Direct injection - In limited applications direct injection (sample analysis without extraction) of the sample into the GC/MS system with a 10 ul syringe may be appropriate. The detection limit is approximately 10,000 ug/L for direct injection; therefore, it is only acceptable where concentrations in excess of 10,000 ug/L are expected.

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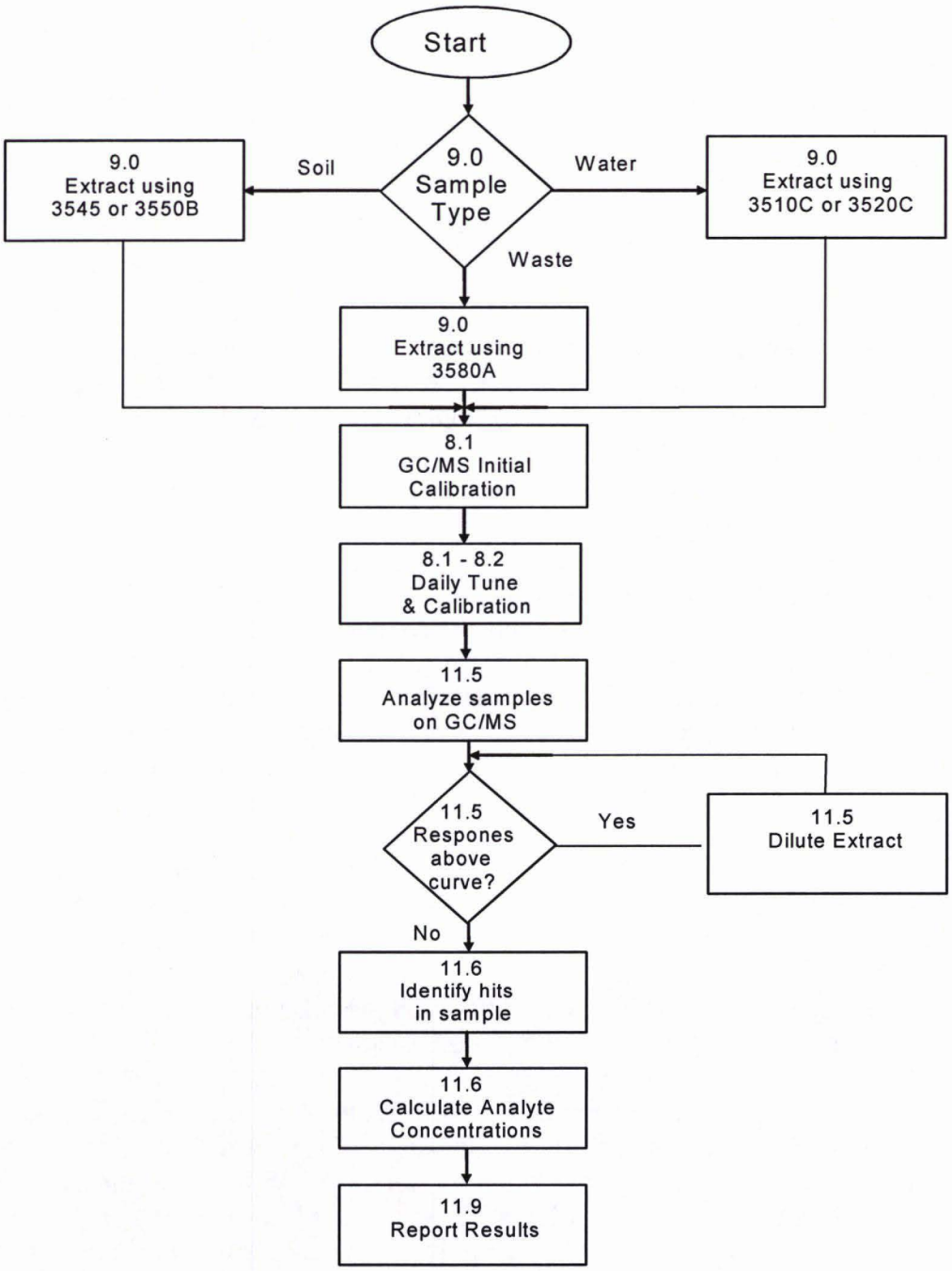
10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES



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11.0 ANALYTICAL PROCEDURES

11.1 The samples are first extracted using the appropriate extraction technique as described in section 9 of this SOP.

11.2 DFTPP and calibration analysis is performed according to section 8.0 in this SOP.

11.3 Samples are run within the 12 hours tune time, which begins with the injection of DFTPP. At the end of this time, a new DFTPP and continuing calibration standard must be analyzed.

11.4 Prior to injection, a 400uL aliquot of each extract is spiked with 4 uL of internal standard.

11.5 After sample analysis, the computer produces a quantitation report listing all target analytes and concentrations found in the sample. Mass spectra are generated for each analyte found at or above the minimum detection limit and are evaluated for validity.

11.6 Qualitative analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of the sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

11.6.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.6.2 All ions present in the standard mass spectrum at a relative intensity greater than 10%, (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of the characteristic ions must agree within 40% of the relative intensities of these ions in the reference spectrum. Analyst judgment may be used even if these criteria are not met.

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The positive identification of a hit should not be made based solely on the criteria mentioned above.

11.7 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Dilutions are prepared by using syringes to transfer aliquots of extract into appropriate amounts of solvent in autosampler vials and adding appropriate amounts of internal standard in order to maintain the 40 ug/mL concentration in the extract. Examples are presented below.

Dilution	Amount sample extract	Amount CH2Cl2	Final dilution volume	Amount ISTD
2x	200 uL	200 uL	400 uL	4 uL
5x	100 uL	400 uL	500 uL	5 uL
10x	100 uL	900 uL	1000 uL	10 uL

Higher dilutions are prepared by performing serial dilutions, e.g. for a 100x dilution, a 10x dilution is diluted again by a factor of 10.

11.8 The data is transferred by the analyst to the LIMS report sheet, or directly downloaded into the LIMS using automation procedures specifically designed for this task.

12.0 DETAILS OF CALCULATIONS

12.1 Response factors are calculated as follows:

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

where:

A_s = peak area of the analyte or surrogate
A_{is} = Peak area of the internal standard

C_s = Concentration of the analyte or surrogate (ug/L)
C_{is} = Concentration of the internal standard (ug/L)

12.2 The % RSD is calculated using the formula:

$$RSD = \left(\frac{s}{\bar{x}}\right)100$$

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where:
s = Standard deviation
x = Average

12.3 Accuracy as % difference:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:
C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.4 Coefficient of correlation

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

where:
X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.5 The concentration of a target analyte using average response factors is calculated as follows:

$$C_x = \frac{(A_x)(C_{is})(V_f)(D)}{(A_{is})(\overline{RF})(V_i)}$$

where:
A_x = Peak area for the analyte or surrogate
C_{is} = Concentration of the internal standard (ug/mL)
V_f = Final volume of the sample extract (mL)
D = Dilution factor for the samples (1/10 = 10)
A_{is} = Peak area of the internal standard
RF = Mean RF for the analyte or surrogate
V_i = Initial volume of the sample extracted (L)

C_x = Calculated concentration of the analyte or surrogate (ug/L)

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12.6 Linear calibration calculations:

12.6.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x / R_{istd}

x = Concentration ratio = Conc(x)/Conc(istd) = C_x / C_{istd}

And m and b are the slope and intercept from the regression equation

12.6.2 For a given response ratio we can solve for C_x / C_{istd} :

$$C_x / C_{istd} = [R_x / R_{istd} - b]/m$$

Use equations 12.4 or 12.5 to calculate the unknown concentration, C_x.

12.6 Quadratic calibration calculations:

12.7.1 The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

12.7.2 $y = ax^2 + bx + c$

or

12.7.3 $ax^2 + bx + (c-y) = 0$

12.7.4 Solving for x using the quadratic equation:

$$x = \frac{b +/- \sqrt{(b^2 - 4a(c-y))}}{2a}$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x / R_{istd}

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x = Concentration ratio = Conc(x)/Conc(istd) = C_x / C_{istd}

a,b and c are constants from the regression equation

Use equations 12.4 or 12.5 to calculate the unknown concentration, C_x.

12.8 Solving for the concentration in water sample:

12.8.1 For a given concentration ratio, compute the unknown, C_x

$$C_x = (C_{is})\left(C_x / C_{istd}\right)\left(V_f / V_i\right)(D)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = Initial sample volume (mL)
D = dilution factor
C_x = Concentration of the sample in ug/L

12.9 Solving for the concentration in soil sample:

$$C_x = (C_{is})\left(C_x / C_{istd}\right)\left(V_f / W_i\right)(D)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)

W_i = Initial sample weight (g)
D = dilution factor
C_x = concentration of the sample (ug/kg) (as received)

12.10 The dry weight concentration of a target analyte is further calculated as follows:

$$C_d = \frac{(C_x)(100)}{(\%solids)}$$

where:

C_x = The uncorrected wet weight concentration

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% solids = the percent solids content of the sample

12.11 Precision

Precision is expressed as relative standard deviation

$$RSD = \left(\frac{s}{\bar{x}}\right)100$$

12.12 Accuracy as % difference:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.13 Precision of duplicate measurements

Precision of duplicate measurements is expressed as relative percent difference

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.14 The LCS recovery is calculated as follows:

$$\%R = \left(\frac{C_x}{C_T}\right)100$$

where:

C_x = the concentration of the analyte in the LCS.
C_t = the theoretical spike concentration.

%R = percent recovery

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12.15 Accuracy as Percent Recovery

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
%R = percent recovery

13.0 QUALITY CONTROL (QC) REQUIREMENTS

The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), laboratory control samples (LCS), matrix spikes (MS), matrix spike duplicates (MSD), laboratory control sample duplicates (LCSD), and surrogate compounds. Additional procedures were defined in section 8 for initial calibration, initial calibration verification (ICV) using a second source, and continuing calibration verification (CCV), and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Tables 6 and 7.

13.1 A batch is defined as a group of samples, which are extracted together. A batch contains a maximum of 20 samples. With each batch of samples extracted, a laboratory control sample (LCS) and a method blank must also be extracted. It is recommended that at least one sample for the batch be extracted three times.

The last two extractions are fortified with a spiking solution to provide a matrix spike, and matrix spike duplicate (MS & MSD). All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch.

13.2 The method blank cannot contain amounts of any target analytes, which are over the reporting limits (RL). If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL for the presents of target analytes. Any amount of target analytes found in

the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch. Some projects

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require that blanks are evaluated to a concentration of ½ the RL, consult the specific QAPP for guidance.

13.3 The Laboratory Control Sample (LCS) is spiked by the extraction laboratory with every target analyte at 1.0 ug/L for water samples and 50 ug/Kg for soil. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 4 and 5 are used in the absence of a project QAPP, or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare a non-conformance report (NCR), and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare a NCR to be reviewed by the department supervisor. Corrective action will consist of re-extraction and re-analysis of the affected samples for, at a minimum, the COC for which a result was derived outside control limits, unless the client's representative and the quality assurance officer (QAO) approve of another course of action. An LCSD is extracted when the batch does not include MS/MSDs. The acceptance criteria for the LCSD is identical to the LCS.

13.4 The matrix spike (MS) and matrix spike duplicate (MSD) are spiked by the extraction laboratory for every target analyte at 1.0 ug/L for water and 50 ug/Kg for soil. The results are included in the QC Summary Report and the data is used to monitor matrix accuracy and precision.

13.5 Internal standard areas must be between 50 and 200% of the areas from the continuing calibration standard and their retention times must be within 30 seconds of the retention times from the daily CCV standard (some QAPPs require the comparison of the internal standard areas and retention times to the ICAL midpoint standard). All samples failing to meet this criteria must be re-analyzed to confirm matrix interference unless interference can be shown by the sample chromatogram. If a matrix effect is suspected, the sample may be diluted for reanalysis.

13.6 Surrogate recoveries must fall within the limits found in Table 3. Samples with surrogates outside these limits must be examined for matrix interference. If none can be proven, the sample must be returned for re-extraction and re-analysis. Samples with surrogate recoveries above the control limits may be approved if

no target compounds are found. All surrogate recoveries must be > 10% or a re-extract and re-analysis is warranted unless matrix interference is demonstrated

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by examination of the chromatogram. Samples failing one surrogate will not be re-extracted, provided all recoveries are above 10% and no target analytes were detected. **Note:** For Ohio VAP work all surrogates in samples, method blanks and LCS/LCSD must be within limits except in the case of obvious or known matrix interference. Otherwise, the entire batch must be re-extracted.

13.7 Back Up: All raw data files are automatically saved to the Microbac server on a daily basis.

13.8 Gases: Helium Tanks must be changed when pressure drops to 500 PSI.

13.9 When new standards are made, they must be checked against the existing curve before they can be used.

13.10 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13 of SOP LQAP (Laboratory Quality Assurance Program), SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and SOP GP-RCA (Root Cause Analysis).

13.10.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 6 does not meet acceptance criteria. When nonconforming data occurs the employee initiates a Nonconformance Report (NCR) and proceeds with indicated corrections as per Tables 6 and 7.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)

- Re-sampling and reanalysis (client decision)

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13.10.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per SOP GP-CAPA. The corrective action process includes a root cause analysis as per SOP GP-RCA, corrections, corrective action (s) and evidence of effectiveness.

13.10.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Prior to data entry into the LIMS (either manual or automatic), all data must undergo two (2) levels of review in the department. The primary review is performed by the analyst and the secondary review is performed by either the department supervisor (or a designee) or another qualified analyst. Any manual integrations must also be signed by the analyst and reviewed and signed by the supervisor or a designee, as per Microbac SOP41.

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14.1.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.1.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.1.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.1.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

15.0 PREVENTIVE MAINTENANCE

In order to minimize the downtime of the instrumentation preventative maintenance is performed on a routine basis. The injection port liners and septa are changed regularly. The source is cleaned on an "as needed" basis, which is determined, when the mass calibration of the instrument becomes increasingly difficult to perform. The front portion of the column may be protected by a guard column to prevent any high boiling contaminants from interfering with the performance of the analytical column. Additionally, from time to time when the peak shape of the standards are deformed, the front portion of the analytical column is clipped to improve performance (approximate 10 cm).

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our

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explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Method 8270C, Revision 3, December 1996.

2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Method 8270D, Revision 4, February 2007.

3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000B, Revision 2, December 1996.

4. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 8000C, Revision 3, March 2003.

5. Microbac SOP LQAP, "Laboratory Quality Assurance Plan"

6. Microbac SOP45, "Standard Operating Procedures for Method Validation Procedures"

7. Microbac SOP GP-CAPA, "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

8. Microbac SOP GP-RCA, "Root Cause Analysis"

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9. Microbac SOP33, "Laboratory Waste Management"

10. Microbac SOP EXB01, "Continuous Liquid-Liquid Extraction for Semivolatiles (BNA)," Method 3510C

11. Microbac SOP EXB08, "Separatory Funnel Liquid-Liquid Extraction for Semivolatiles (BNA)," Method 3510C

12. Microbac SOP EXB02, "Sonication Extractions for Semivolatiles", Method 3550B

13. Microbac SOP ASE01, "Accelerated Solvent Extractor", SW846, Method 3545A

14. Microbac SOP41, "Manual Integration of Chromatographic Peaks"

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TABLE 1
TARGET COMPOUND LISTS
(WITH CORRESPONDING INTERNAL STANDARDS)

Compound	SIM Group	Primary Ion	Secondary Ion(s)
Naphthalene-d8 (ISTD)	1	136	
Nitrobenzene-d5 (SURR)	s1	82	128
Naphthalene	1	128	127
2-Methylnaphthalene	2	142	141
1-Methylnaphthalene	2	142	141
Acenaphthene-d10 (ISTD)	4	164	
2-Fluorobiphenyl (SURR)	s2	172	171
Acenaphthylene	3	152	151
Acenaphthene	4	154	153
Fluorene	5	166	165
Phenanthrene-d10 (ISTD)	6	188	
Phenanthrene	6	178	176
Anthracene	6	178	176
Fluoranthene	7	202	203
Chrysene-d12 (ISTD)	8	240	
Pyrene	7	202	203
p-Terphenyl-d14 (SURR)	s3	244	243
Benzo[a]anthracene	8	228	229
Chrysene	8	228	229
Perylene-d12 (ISTD)	10	264	
Benzo[b]fluoranthene	9	252	253
Benzo[k]fluoranthene	9	252	253
Benzo[a]pyrene	10	252	253
Indeno[1,2,3-cd]pyrene	11	276	277
Dibenz[ah]anthracene	11	278	279
Benzo[ghi]perylene	12	276	277

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TABLE 2
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17-23% of mass 442

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TABLE 3
SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES

Surrogate Compound	Water	Soil/Waste
Nitrobenzene-d_5	35–114	23 - 120
2-Fluorobiphenyl	43-116	30 - 115
p-Terphenyl-d_{14}	33-141	18 - 137

TABLE 4
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR METHOD 8270C-MODIFIED FOR LOW LEVEL PAHS ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)	PRECISION (% RPD)	MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Acenaphthene	83-32-9	8270C	40-120	0-20	0.025	0.05
Acenaphthylene	208-96-8	8270C	40-120	0-20	0.025	0.05
Anthracene	120-12-7	8270C	45-130	0-20	0.025	0.05
Benzo(a)anthracene	56-55-3	8270C	80-130	0-20	0.025	0.05
Benzo(b)fluoranthene	205-99-2	8270C	70-130	0-20	0.025	0.05
Benzo(k)fluoranthene	207-08-9	8270C	50-130	0-20	0.025	0.05
Benzo(g,h,i)perylene	191-24-2	8270C	40-140	0-20	0.025	0.05
Benzo(a)pyrene	50-32-8	8270C	80-130	0-20	0.025	0.05
Chrysene	218-01-9	8270C	80-130	0-20	0.025	0.05
Dibenzo(a,h)anthracene	53-70-3	8270C	40-140	0-20	0.025	0.05
Fluoranthene	206-44-0	8270C	60-140	0-20	0.025	0.05
Fluorene	86-73-7	8270C	40-130	0-20	0.025	0.05
Indeno(1,2,3-cd)pyrene	193-39-5	8270C	50-130	0-20	0.025	0.05
2-Methylnaphthalene	91-57-6	8270C	40-120	0-20	0.025	0.05
Naphthalene	91-20-3	8270C	45-110	0-20	0.025	0.05
Phenanthrene	85-01-8	8270C	50-110	0-20	0.025	0.05
Pyrene	129-00-0	8270C	60-120	0-20	0.025	0.05
1-Methylnaphthalene	90-12-0	8270C	40-120	0-20	0.025	0.05

Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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TABLE 5
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR METHOD 8270C-MODIFIED FOR LOW LEVEL PAHS ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD*	ACCURACY (% RECOVERY)	PRECISION (% RPD)	MDL SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
Acenaphthene	83-32-9	8270C	35-100	0-30	1.25	2.5
Acenaphthylene	208-96-8	8270C	35-100	0-30	1.25	2.5
Anthracene	120-12-7	8270C	35-100	0-30	1.25	2.5
Benzo(a)anthracene	56-55-3	8270C	60-130	0-30	1.25	2.5
Benzo(b)fluoranthene	205-99-2	8270C	50-130	0-30	1.25	2.5
Benzo(k)fluoranthene	207-08-9	8270C	60-130	0-30	1.25	2.5
Benzo(g,h,i)perylene	191-24-2	8270C	60-130	0-30	1.25	2.5
Benzo(a)pyrene	50-32-8	8270C	60-130	0-30	1.25	2.5
Chrysene	218-01-9	8270C	55-130	0-30	1.25	2.5
Dibenzo(a,h)anthracene	53-70-3	8270C	55-130	0-30	1.25	2.5
Fluoranthene	206-44-0	8270C	60-120	0-30	1.25	2.5
Fluorene	86-73-7	8270C	35-100	0-30	1.25	2.5
Indeno(1,2,3-cd)pyrene	193-39-5	8270C	60-130	0-30	1.25	2.5
2-Methylnaphthalene	91-57-6	8270C	40-100	0-30	1.25	2.5
Naphthalene	91-20-3	8270C	40-100	0-30	1.25	2.5
Phenanthrene	85-01-8	8270C	40-100	0-30	1.25	2.5
Pyrene	129-00-0	8270C	60-120	0-30	1.25	2.5
1-Methylnaphthalene	90-12-0	8270C	35-100	0-30	1.25	2.5

Values are statistically derived from laboratory control samples and are evaluated annually. Actual control limits may vary.

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TABLE 6
Quality Control Criteria
Semivolatile GC/MS Analysis
Method 8270C-PAHL

Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass spectral ion Intensities (DFTPP criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 2	Retune instrument and repeat DFTPP check
Initial Calibration (ICAL)	Initially and upon failure of two consecutive CCVs	Linear regression with $r^2 \geq 0.990$ for each analyte; Non-linear COD ≥0.99 with 6 points for second order, 7 points for higher	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1)
Second source calibration verification (ICV)	After each initial calibration	≤ 30% drift for each analyte or project specific criteria	Re-analyze ICV; upon second failure, Repeat initial calibration (1)
Continuing calibration verification (CCV)	Each 12 hour tune	SPCC average RF ≥ 0.3: CCC percent drift ≤ 20%; other target analytes ≤ 20 %	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time in CCV within 30 sec of retention time in ICAL midpoint. Retention time in samples within 30 sec of retention time in CCV. Areas within -50% to +100% of areas in CCV.	Check for MS malfunction or interference; re- analyze sample in accordance with Section 13.6
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	< project reporting limit for each target analyte	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.3.
Laboratory Control Sample (LCS)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated ranges; use project QAAP or standard control criteria. (1,2)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.4.
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated range.	Qualify data and/or address in the report narrative.
Surrogate spike	Every sample, standard, and quality control sample	Recoveries within the designated ranges: use project QAAP or standard control criteria. (1)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.7

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

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TABLE 7
Quality Control Criteria
Semivolatile GC/MS Analysis
Method 8270D-PAHL

Control Item	Frequency	Acceptance Criteria	Corrective Action
Mass spectral ion intensities (DFTPP criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 2	Retune instrument and repeat DFTPP check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered	≤ 20% RSD for all compounds RF > minimum values in Table 8, If >20% RSD, then linear regression, If R^2 <0.990 then quadratic regression.	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1)
Second Source calibration	After each initial calibration	≤ 30% for all compounds.	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Every 12 hours	≤ 20% for all compounds. RF > minimum values in Table 8. Internal standard: Retention time within 30 sec of IS retention time in ICAL midpoint std and area within –50% to+100% of IS midpoint area.	Re-analyze CCV; upon second failure repeat calibration. Other compounds: repeat initial calibration if compound fails in more than two consecutive analytical batches (1)
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 sec of IS retention time in CCV midpoint std and area within –50% to+100% of CCV area.	Check for MS malfunctions or interference; re-analyze sample in accordance with Section 13.5
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch.	< project reporting limit for each target analyte	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.2
Laboratory Control Sample (LCS)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.3
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch.	Target compounds within the designated range.	Qualify data and/or address in the report narrative.
Surrogate spike	Every sample, standard and quality control sample	Recoveries within the designated ranges; use project QAAP or standard control criteria (1)	Notify supervisor and initiate NCR; investigate; Re-extract and re-analyze samples in accordance with Section 13.6

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

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TABLE 8
Minimum Response Factors for Low-Level PAH Analysis by Method 8270D

Compound	Minimum Response Factor (RF)
Naphthalene	0.700
2-Methylnaphthalene	0.400
1-Methylnaphthalene	0.400
Acenaphthylene	0.900
Acenaphthene	0.900
Fluorene	0.900
Phenanthrene	0.700
Anthracene	0.700
Fluoranthene	0.600
Pyrene	0.600
Benzo[a]anthracene	0.800
Chrysene	0.700
Benzo[b]fluoranthene	0.700
Benzo[k]fluoranthene	0.700
Benzo[a]pyrene	0.700
Indeno[1,2,3-cd]pyrene	0.500
Dibenz[ah]anthracene	0.400
Benzo[ghi]perylene	0.500

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Figure 1
Semi-Volatile GC/MS Checklist

Checklist ID: 31387

Microbac Laboratories Inc.

Data Checklist

Date: 26-AUG-2008
Analyst: CAA
Analyst: NA
Method: 8270L
Instrument: HPMS7
Curve Workgroup: NA
Runlog ID: 23952
Analytical Workgroups: L08080447, L08080568, L08080512(PE)

ANALYTICAL	
System Performance Check	X
DFTPP (MS)	X
Endrin/DDT breakdown (8081/MS)	NA
Pentachlorophenol/benzidine tailing (MS)	NA
Eluent check (IC)/system pressure (HPLC)	NA
Window standard (FID)	NA
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	X
Continuing calibration blank (CCB) (IC)	NA
Special standards	NA
Blanks	X
TCL hits	X
Surrogate recoveries	X
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	X
MS/MSD/Sample duplicates	NA
Recoveries	NA
%RPD	NA
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	X
Surrogate recoveries	X
Internal standard areas (MS)	X
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	NA
Reruns	NA
Manual integrations	NA
Project/client specific requirements	X
REPORTING	X
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	CAA
SUPERVISOPY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer: 27-AUG-2008

Secondary Reviewer: 27-AUG-2008

CHECKLIST1 - Modified 03/05/2008
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Figure 2
Semi-Volatile GC/MS Runlog

Run Log ID: 23952

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HPM97 Dataset: 082608
Analyst1: CAA Analyst2: NA
Method: 627CL SOP: MSS03 Rev: 5

Maintenance Log ID: 25571

Column 1 ID: RXI-5MS Column 2 ID: NA
Workgroups: WG280904, WG280903
Internal STD: COA13174 Surrogate STD: NA Calibration STD:
Comments:

Seq	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	7M39561	WG280887-01 5ppm DFTPF Std	1	1	STD25193	08/26/08 15:17
2	7M39562	WG280887-02 2.5ppm PAHL Std	1	1	STD27641	08/26/08 15:33
3	7M39563	WG280813-01 BLK 8/25 10010 P19	7	1	SOIL	08/26/08 15:59
4	7M39564	WG280813-02 LCS 8/26 10010 P19	7	1	SOIL	08/26/08 16:25
5	7M39565	WG280813-03 LCS DUP 8/26 10010 P19	7	1	SOIL	08/26/08 16:51
6	7M39566	WG280812-01 BLK 8/25 10011 P11	1	1		08/26/08 17:17
7	7M39567	WG280812-02 LCS 8/26 10011 P11	1	1		08/26/08 17:43
8	7M39568	WG280812-03 LCS DUP 8/26 10011 P11	1	1		08/26/08 18:09
9	7M39569	L08080447-01	1	1		08/26/08 18:35
10	7M39570	L08080568-02	1	1		08/26/08 19:02
11	7M39571	L08080512-02 RE 2X SOIL	7	2	SOIL	08/26/08 19:28
12	7M39572	BAKEOUT	1	1		08/26/08 19:54
13	7M39573	BAKEOUT	1	1		08/26/08 20:20
14	7M39574	BAKEOUT	1	1		08/26/08 20:46

Comments

Seq	Rerun	Dil	Reason	Analytes

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STANDARD OPERATING PROCEDURES FOR ANALYSIS OF VOLATILE ORGANIC ANALYTES BY METHODS 8260A AND 8260B SOP MSV01

Revision 14

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The following persons have read and understand this SOP and are using the latest version of the test method referenced on the Title Page:

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1.0 SCOPE AND APPLICATION

1.1 Microbac Standard Operating Procedure (SOP) MSV01 pertains to the determination of volatile organic compounds (VOC) in solid and liquid matrices using purge and trap gas chromatography / mass spectroscopy (GC/MS).

1.2 This method references USEPA SW846 Methods 8000B (December 1996), 8000C (March 2003), 8260A (September 1994), 8260B (December 1996), 5030A (July 1992), 5030B (December 1996), 5030C (May 2003), and 5035A (July 2002); AFCEE QAPP's 1998, 2001, and 2005.

1.3 SOP MSVO1 applies to all volatile mass spectral analyses except where client specific Quality Assurance Project Plan's (QAPP) override this method's quality assurance plan. Soils collected in widemouth bottles for compliance with the Ohio EPA Contract and the Ohio Voluntary Action Program (Ohio VAP) must be reported with references to SW-846 method 5030A and 8260A. This SOP fulfills the requirements of Methods 8260A and 8260B. SOP revisions for Ohio VAP must undergo agency review and approval prior to implementation. Method 8260A will not be used for South Carolina samples.

1.4 Table 1 lists the target compound list for this method.

1.5 Appendix I contains procedures for selected ion monitoring (SIM) analysis for 1,4-dioxane. Appendix II contains suggested primary, secondary, and tertiary chlaracteristic ions. Appendix III contains the procedures for "Ultra low-level water analysis". Refer to Section 11.13 for the analysis of wipe samples.

1.6 Refer to Section 16.0 of Microbac's Laboratory Quality Assurance Plan (LQAP) for specific OVAP requirements.

2.0 SAFTY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Gloves must be worn while handling any chemicals, standards, or samples. Other required personal protective equipment (PPE) includes lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following VOC's have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene	chloroform
carbon tetrachloride	vinyl chloride

The toxicity or carcinogenicity of the other reagents and analytes used in this method have not been precisely defined, therefore, each chemical and sample shall be treated as a potential health hazard and exposure reduced to the lowest possible level. Procedures involving primary standards and sample preparation shall be performed in a fume hood.

2.3 Material Safety Data Sheets (MSDS) for each analyte and reagent used within the laboratory are available to all employees. Consult MSDS's prior to handling chemicals.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Pre-cleaned 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices utilizing methods 5030C and 5035A. Soil samples not utilizing Method 5035A must be collected in 125 mL pre-cleaned glass screw cap jars with teflon-lined lids. Soil samples collected via 5035A may also be collected in Encore (or equivalent) containers then transferred to 40 mL VOA vials for analysis. Refer to Microbac SOP PAT01 for additional requirements.

3.2 Water samples preserved with HCl (pH $\leq$ 2) must be analyzed within 14 days of sample collection. Unpreserved water samples (pH > 2) must be analyzed within 7 days of sample collection. Water samples for acrolein and acrylonitrile may be preserved with sodium thiosulfate (pH between 4 and 6). Waste, oil, soil, and sludge samples do not require the addition of preservative but should be stored at 4° C (± 2° C). Solid samples utilizing Method 5035A require preservation if analysis cannot be performed within 48 hours of collection. Waste, soil, oil, and sludge samples have a holding time of fourteen days from the date of collection. Soil samples collected in widemouth bottles (8260A), are stored at 4° C ± 2° C. Samples collected via 5035A may be stored at -6° C to -20° C.

3.3 Sample hold time is defined as time elapsed from sample collection date and time to sample analysis date and time.

3.4 Samples are stored in assigned locations until expiration of hold times. After hold-time expiration, samples are removed from storage refrigerators and returned to sample archive. Samples requiring internal chain-of-custody are returned to the sample receiving custodian.

3.5 Temperature logs are maintained for all refrigerator and freezer storage units. Temperatures are recorded daily.

4.0 METHOD PERFORMANCE

4.1 Table 2 summarizes the performance data for water analysis; Table 3 summarizes performance data for soil/solid waste analysis. These tables include the analyte list, ranges for accuracy and precision, current laboratory method detection limits (MDL), nominal laboratory reporting limits (RL), and True values.

4.2 MDL's are derived in accordance with 40 CFR Part 136 and represent a composite of multiple instruments available for Methods 8260A and 8260B. MDL's presented in Tables 2 and 3 are verified quarterly and evaluated annually. Verification consists of analyzing a fortified blank (MDL check standard) spiked at 2 times the observed MDL and performed after each initial MDL study and quarterly thereafter. This MDL check standard is used to verify that the laboratory MDL, which is derived from multiple instruments and analysts, is routinely achievable on a specific instrument and method over the course of time. The actual instrument detection limit may be lower. Precision and accuracy data was derived from laboratory control sample results from the previous year and verified annually. Reporting limits (RL) are nominal laboratory values but project RL's may vary. Additional details on MDL studies may be found in SOP 45.

4.3 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE MEASURES

5.1 Samples for volatile organics analyses are susceptible to laboratory contaminants (e.g.: methylene chloride, acetone, n-hexane). To eliminate the potential for interferences from other areas of the laboratory, the Volatiles Laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Samples preserved with HCl or sodium bisulfate may result in the loss of 2-chloroethylvinylether as a target or spiked analyte due to its reactivity with the preservatives.

5.3 Soil analyses may result in low internal and/or surrogate standard recovery due to the poor purging efficiencies of some matrices. Reanalysis may be performed to confirm matrix interference.

5.4 Carry-over contamination may occur when a sample containing low levels of VOC's is analyzed immediately following a sample containing high levels of VOC's. If this situation occurs during a non-monitored analysis, the sample containing the low concentration VOC's may require reanalysis.

5.5 Samples may become contaminated by diffusion of volatile organics 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, handling, and analysis steps serves as a check on such contamination.

5.6 Storage blanks are placed in refrigerator and freezer units used for the storage of samples for volatiles analysis. Refer to Section 13.0 for storage blank procedures.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/MS systems: Hewlett-Packard (HP) 6890 gas chromatographs equipped with HP 5973 mass spectrometers. Systems utilize HP Enviroquant software. Refer to Table 4 for suggested operating parameters.

6.2 Purge-and-trap units: Tekmar liquid sample concentrator (LSC) 2000, 3000, Velocity, and Stratum; Varian (or equivalent), Archon auto-sampler. Refer to Table 4 for suggested operating parameters.

6.3 Top loading balances: Ohaus Navigator, Mettler PE600,

6.4 Capillary columns: 60 M Restek 502.2, 0.32 mm ID, 1.8 μm film thickness

6.5 Traps: Supelco Vocarb 3000; Tekmar trap #9.

6.6 Volumetric flasks: Class A; 1 mL to 200 mL

6.7 Mininert vials with septum valves: 1 mL to 10 mL

6.8 40 mL VOA vials: Eagle Pitcher, ESS

6.9 Syringes: Hamilton Gas tight with Luer lock tip: 25 mL, 5 mL; Gas tight with fixed needles: 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, 1000 uL (Hamilton syringes accuracy: ±1% at or above 10% of syringe volume)

6.10 Steel and wooden spatulas

6.11 Disposable Pasteur pipets

6.12 Oven: Blue M baking oven

6.13 Equivalent equipment and supplies may be used.

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Primary calibration standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
502.2 CAL200 Mega Mix	Restek	30432	200 ug/mL
VOC Mix 6 (Gases)	Supelco	48799-u	2000 ug/mL
Custom VOA Mix 3 (Additionals)	CPI	Z-122281-04	250 ug/mL
2-Chloroethyl vinyl ether (2-CEVE)	Restek	30265	2000 ug/mL
Acrolein	Chem Service	F2S	100 ug/mL
1,1,2-Trichloro-1,2,2-trifluoroethane (freon 113)	Supelco	48411	1000 ug/mL
Custom VOA Mix 2	Restek	551289	250 ug/mL
Vinyl Acetate	Restek	30216	2000 ug/mL
Mass. Oxygenates Standard	Restek	558894	50 - 100 ug/Ml
1,3-Butadiene	Accustandard	S-406A-10X	2000 ug/mL

7.2 Primary internal and surrogate standard mixtures:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
8260A / 8260B Internal Standards	Restek	30241	2500 ug/mL (fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4)
8260A / 8260B Surrogate Standard Mixture	Restek	30240	2500 ug/mL (dibromofluorobenzene, 1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene)

7.3 Primary laboratory control sample (LCS) / matrix spike (MS), alternate source (ICV) standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
Volatile Organic Compound (VOC) Mixture	Accustandard	M-502A-R-PAK	200 ug/mL
Volatile Organic Compounds (VOC) Additional Mixture 8260 Calibration Mix 2	Supelco	46831-U	200 ug/mL
MA Oxygenates Alternate Source	Supelco	21103590	50 ug/mL – 100 ug/mL
Custom Gases	CPI	Z-120313-03	250 ug/mL
Vinyl Acetate	Accustandard	APP-9-211-20X	2000 ug/mL
Acrolein	Accustandard	APP-9-007	100 ug/mL
1,3-Butadiene	Supelco	20958097	200 ug/mL

NOTE: Source of LCS/MS/ICV standards different then primary calibration standards.

7.4 Primary 4-bromofluorobenzene (BFB) standard: Chem Service # F8335; 2000 ug/mL

7.5 Intermediate calibration standards: Primary calibration standards diluted to prepare intermediate standards as follows:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
502.2 CAL200 Mega Mix	200	1250	5	50
Custom Mix 3	250	1000	5	50
2-CEVE	2000	250	10	50
Acrolein	100	*	*	100
Custom Mix 2	250	1000	5	50
VOC Mix 6 (gases)**	2000	125	5	50
Freon 113**	1000	250	5	50
Vinyl Acetate	2000	250	10	50
1,3-Butadiene	200	1250	5	50
Mass Oxygenates	50-100	*	*	50-100

* Denotes transfer contents to vial without diluting
** Combined to create one intermediate standard

7.6 Intermediate internal and surrogate standards preparation:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
Intermediate internal and surrogate standards	250	1000	10	25

7.7 Intermediate LCS / MS / ICV standards prepared as follows:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
VOC Mix **	200	1000	10	20
VOC Adds Mix	200	1000	10	20
Custom Gases **	250	800	10	20
Vinyl acetate	2000	100	10	20
Acrolein	100	*	*	100
1,3-Butadiene	200	1000	10	20
MA Oxygenates	50-100	*	*	50-100

* Denotes transfer contents to vial without diluting
** Denotes combined into one solution

7.8 50 ug/mL BFB intermediate solution preparation:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
BFB intermediate solution	2000	125	5	50

7.9 Working standards preparation

7.9.1 Working standards used for initial calibration and calibration verification are prepared by diluting intermediate standards in deionized water as follows:

Soil Initial Calibration Standards, ug/Kg
(suggested preparation)

STOCK STANDARD, CONCENTRATION	WORKING STANDARD, ug/Kg									
	0.5	1 [5]	2 [10]	5 [25]	10 [35]	20 [50]	50* [100]	75 [150]	100 [200]	200 [400]
VOA Mega mix (8260 Standard) 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Custom Gases 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Custom Mix 2 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Custom Mix 3 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
2-Chloroethylvinyl ether 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Vinyl acetate 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Acrolein 100 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Surrogate Standard 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
1,3Butadiene 50 ug/mL	1 uL	1 uL	2 uL	5 uL	1 uL	2 uL	5 uL	7.5 uL	10 uL	20 uL
Oxy STD 50-100 ug/L	-	5 uL	10 uL	25 uL	3.5 uL	5 uL	10 uL	15 uL	20 uL	40 uL
Final volume, DI water (mL)	100	50	50	50	5	5	5	5	5	5

* Denotes CCV
[ ] Denotes Oxygenates

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD, CONCENTRATION	WORKING STANDARDS CONCENTRATIONS (ug/L)									
	0.3	0.4	1 [5]	2 [25]	5 [50]	20 [75]	50* [100]	100 [200]	200	300 [300]
VOC Mega Mix (50 ug/mL)	1.2 uL	1.6 uL	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	N/A
VOC Mix 6 (Gases) (50 ug/mL)	N/A	1.6 uL	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	N/A
Custom VOA Mix 2 (50 ug/mL)	N/A	N/A	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	300 uL
Custom VOA Mix 3 (50 ug/mL)	N/A	1.6 uL	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	N/A
2-CEVE (50 ug/mL)	N/A	N/A	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	300 uL
Acrolein (100 ug/mL)	N/A	N/A	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	300 uL
Vinyl Acetate (50 ug/mL)	N/A	N/A	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	300 uL
Surrogate Standard (50 ug/mL)	N/A	N/A	0.5 uL	1 uL	2.5 uL	10 uL	25 uL	50 uL	100 uL	N/A
1,3-Butadiene (50 ug/mL)	N/A	N/A	1 uL	2 uL	5 uL	20 uL	50 uL	100 uL	200 uL	300 uL
OXY Std (50-100 ug/mL)	N/A	N/A	5 uL	25 uL	50 uL	75 uL	100 uL	200 uL	N/A	300 uL
Final Volume, DI Water (mL)	200	200	50	50	50	50	50	50	50	50

* Denotes CCV
[ ] Denotes Oxygenates

7.9.2 Procedure for preparing working standard in volumetric flask: The appropriate volume of intermediate standard is injected into the expanded area of a volumetric flask containing deionized water. The flask is adjusted to volume then inverted three times. An aliquot is transferred to a 5 mL Luer lock syringe or 40 mL VOA vial and placed on the autosampler.

7.9.3 Procedure for preparing standard in 5 mL Luer lock syringe: The volume of stock standard is injected into a 5 mL Luer lock syringe containing deionized water.

7.10 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards in deionized water as follows:

Water Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/L)	ICV FINAL CONCENTRATION (ug/L)
VOC mixture (20 ug/mL)	50	125	50	20	50
VOC additional Mix (20 ug/mL)	50	125	50	20	50
Vinyl acetate (20 ug/mL)	50	125	50	20	50
Acrolein (100 ug/mL)	50	125	50	20	50
1,3-Butadiene (20 ug/mL)	50	125	50	20	50
MA Oxy Alt. Source (50-100 ug/mL)	100	250	50	100-200	200-400

Soil Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/Kg)	ICV FINAL CONCENTRATION (ug/Kg)
VOC mixture (20 ug/mL)	5	12.5	5	20	50
VOC additionals Mix (20 ug/mL)	5	12.5	5	20	50
Vinyl acetate (20 ug/mL)	5	12.5	5	20	50
Acrolein (100 ug/mL)	5	12.5	5	20	50
1,3-Butadiene (20 ug/mL)	5	12.5	5	20	50
MA Oxy Alt. Source (50-100 ug/mL)	10	25	5	100-200	200-400

7.11 50 ng BFB: Prepared by diluting 10 uL of BFB intermediate solution in 50 mL of DI water then purging 5 mL (else 1 uL of BFB intermediate solution is injected into the GC injection port).

7.12 Purge and trap grade methanol: (EM Science)

7.13 Reagent water (ASTM Type II deionized water, UV treated)

7.14 Purified Sand: J.T. Baker (Baked 150°C)

7.15 Concentration of calibration standards may vary depending on, but not limited to, availability, purity, and project requirements, therefore, recipes for standards preparation will be adjusted accordingly. Archon autosamplers add 1 uL of 250 ug/mL internal standards mixture.

7.16 Equivalent standards and reagents may be used.

7.17 Standards are stored at < 0°C or per manufacturer's instructions. Standards are stored in glass vials with Teflon-lined lids and/or mininert vials. Expiration dates for primary standards are per manufacturer's instructions; intermediate standards have a 30 day expiration date from the preparation date.

7.18 If required, sodium bisulfate is added to low-level soil standards and QC samples.

8.0 CALIBRATION PROCEDURES

8.1 The GC/MS system is hardware-tuned via auto-tune or manual tune.

8.2 50 ng of BFB is analyzed via direct inject or purging and the mass spectrum is compared to acceptance criteria in Table 5. Evaluation is performed using the "Autofind" option of the Enviroquant software [the average of the apex, 1(-)apex, 1(+)apex is calculated and a background scan is then subtracted]. Once acceptance criteria is met, an initial calibration or calibration verification may be performed. All standards, samples, and QC samples associated with a BFB analysis must use identical mass spectrometer instrument conditions. A

8.3 For Initial calibration a minimum of five calibration levels containing target analytes and surrogate standards is required. The lowest calibration level must be equal to or below the required reporting limit for each analyte.

8.4 Initial calibration standards are analyzed using the introduction method of choice (5030C or 5035A). Standards used for soil calibration are loaded into 40 mL VOA vials containing 5.00 g (±0.1g) of oven baked reagent sand and utilize a heated purge (40° C).

8.5 Following analysis of the initial calibration, relative response factors (RRF) and average RRF for each surrogate and target analyte are calculated.

8.6 Five analytes designated as system performance check compounds (SPCC) must meet minimum average response factor criteria ($\overline{RRF}$) as follows:

COMPOUND	MINIMUM $\overline{RRF}$
chloromethane	0.10
1,1-dichloroethane	0.10
bromoform	0.10
chlorobenzene	0.30
1,1,2,2-tetrachloroethane	0.30

8.7 The percent relative standard deviation (%RSD) is calculated for all surrogates and target analytes. The % RSD for all target analytes must be less than 15%,

however 6 analytes designated calibration check compounds (CCC) must have % RSD less than or equal to 30%. The CCC's are:

COMPOUND	ICAL MAX % RSD	CCV MAX % D
1,1-dichloroethene	± 30	± 20
chloroform	± 30	± 20
1,2-dichloropropane	± 30	± 20
toluene	± 30	± 20
ethyl benzene	± 30	± 20
vinyl chloride	± 30	± 20

8.8 Method 8260 Calibration Options

8.8.1 Linear – Using Average RF with RSD ≤ 15%

If the % RSD for all target analytes is less than or equal to 15%, then the response factor is assumed constant over the calibration range. Average response factor, therefore, may be used for quantitation. If the CCC's are not target analytes for a specific project, all required analytes must be ≤ 30% RSD.

If more than 5 calibration levels were analyzed, high and/or low points for poor responding and/or saturated compounds may be removed. The low calibration levels must be at or below the required reporting limit. The curve still must contain a minimum of 5 levels.

The average RF option is the preferred method of GC/MS calibration, since linearity may be assumed throughout the full calibration range. However, linear and quadratic models may be used under the conditions discussed in the following sections. If the % RSD for any target analyte is greater than 15%, one of the following procedures may be employed.

8.8.2 Linear Regression with Coefficient of Determination (COD) r^2 > 0.99

Linear regression is an alternative to average RF, but has the potential for significant bias at the lower concentration levels. It should only be used when re-fitting the lowest calibration standard yields a maximum % drift of 40% (residual test). If a particular analyte exceeds 15% RSD, then linear regression may be utilized for that analyte. The fit for the equation (r^2) must be > 0.99.

8.8.3 Quadratic Calibration with COD (r^2) > 0.99

Several compounds on the 8260/8270 extended lists and the EPA Appendix IX list do not display consistently linear behavior. Quadratic calibration, employing at least six calibration points, may be used to improve accuracy for these analytes, particularly at the lower calibration levels, and is a better alternative than linear regression when linear fails the re-fitting test. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and should not be used for analytes with a previous history of linear performance. Quadratic regression may be employed provided the COD (r^2) is $\geq$ 0.990, however a minimum of 6 calibration standards must be used. Those analytes utilizing first and/or second order calibration are noted on the initial calibration report. **NOTE:** Origin not forced when using linear and quadratic regressions. Quadratic regression cannot be used to extend the calibration range.

8.8.4 For samples received from California, quadratic models for analytes that normally display linear responses in the calibration ranges will not be employed. Listed below are 8260 compounds that do not consistently exhibit linear behavior:

8260 Compounds	**Additional 8260 Compounds**
vinyl acetate	t-butyl alcohol
vinyl chloride	1,4-dioxane
2-chloroethylvinyl ether	propionitrile
naphthalene	tetrahydrofuran
acetone	acrolein
	iodomethane
	1-chlorohexane
	1-butanol
	isobutyl alcohol

8.9 Following the initial calibration an initial calibration verification (ICV) is performed. Acceptance criteria is ± 25% drift.

8.10 The mid-point standard of the calibration curve will be used to establish the relative retention time window position for each analyte and surrogate.

8.11 A calibration verification (CCV) is performed every 12 hours of analysis time following an acceptable BFB. Acceptance criteria:

8.11.1 SPCC's meet minimum $\overline{RRF}$ criteria in Section 8.6.

8.11.2 CCC's in Section 8.7 $\leq$ 20 % difference when using average response factor or $\leq$ 20 % drift when using regression fit. Non-CCC's should be $\leq$ 20% difference/drift

with sporadic marginal failures permitted to ≤ 40 % difference/drift. The following poor performing analytes may exceed ± 40% difference/drift: dichlorodifluoromethane, chloromethane, bromomethane, trichlorofluoromethane, chloroethane, 2-chloroethyl vinyl ether, acetone, vinyl acetate, 2-butanone, 2-hexanone, 4-methyl-2-pentanone, 1,2-dibromo-3-chloropropane, bromoform, acrolein, iodomethane, dimethyl disulfide, t-1,4-dichloro-2-butene, 1,3-butadiene, acetonitrile, 2-chloro-1,3-butadiene, ethyl acetate, methacrylonitrile, isobutal alcohol, 1-butanol, methyl methacrylate, 2-nitropropoane, cyclohexanone.

8.11.3 CCV internal standard response and retention times within –50% to +100% and ±30 seconds, respectively, compared to the same calibration standard in the initial calibration.

8.12 Single-point calibration may be performed for Appendix IX and F list analytes. Analytes detected above reporting limits require reanalysis using a multi-point calibration curve.

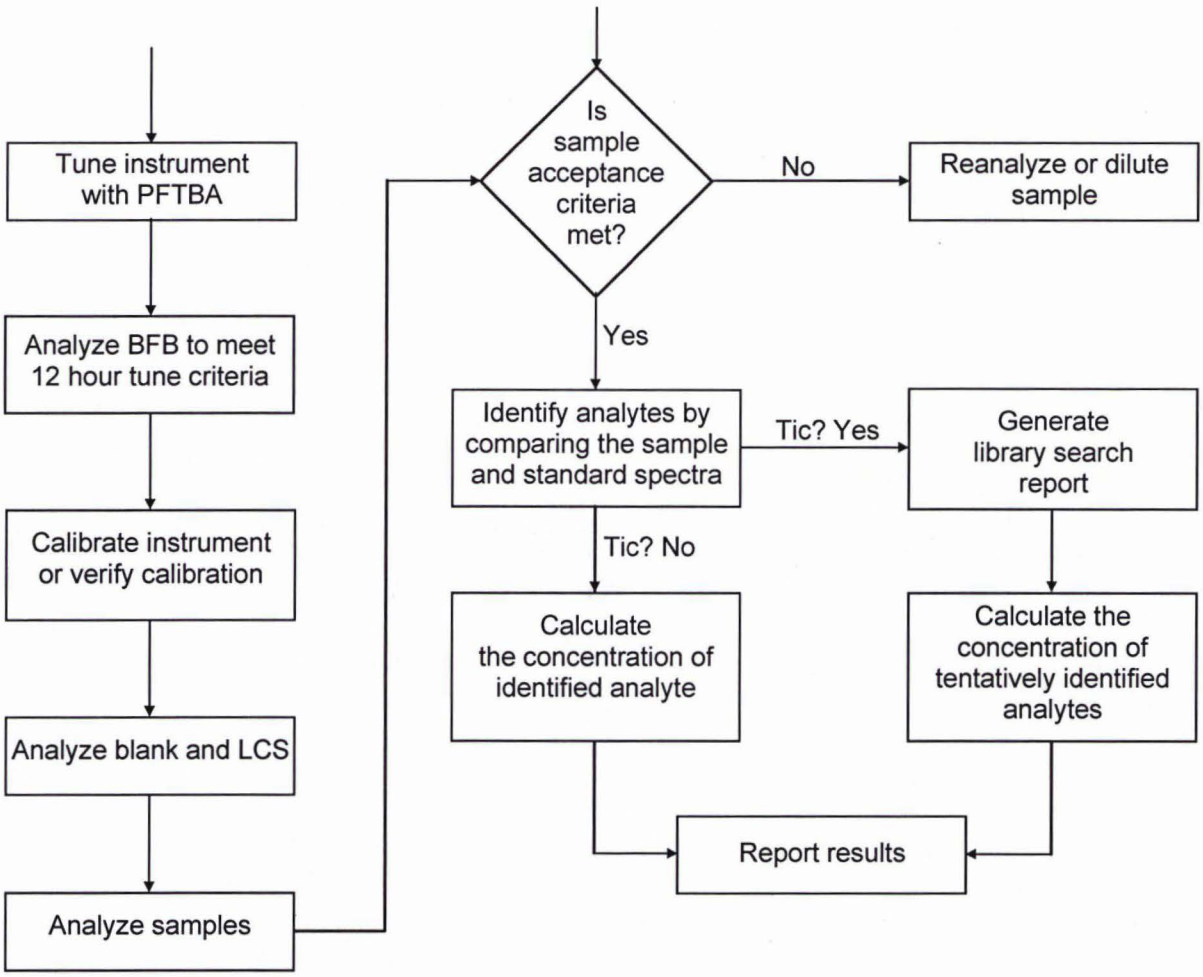
8.13 For analysis of OVAP samples by 8260A and 8260B, n-hexane will be included in the list of calibration check compounds (CCC). Therefore n-hexane will be required to pass CCC acceptance criteria.

8.14 Refer to Section 13.0 for corrective action.

9.0 SAMPLE PREPARATION

9.1 Purge-and-trap procedures are found in Microbac SOP PAT01 for 5030C and 5035A.

10.0 DIAGRAM OR TABLES TO OUTLINE PROCEDURES


Tune instrument with PFTBA
Analyze BFB to meet 12 hour tune criteria
Calibrate instrument or verify calibration
Analyze blank and LCS
Analyze samples
Is sample acceptance criteria met?
No
Reanalyze or dilute sample
Yes
Identify analytes by comparing the sample and standard spectra
Tic? Yes
Generate library search report
Tic? No
Calculate the concentration of identified analyte
Calculate the concentration of tentatively identified analytes
Report results

11.0 ANALYTICAL PROCEDURES

11.1 Prior to sample analysis, instruments must pass tuning and calibration criteria per Section 8.0.

11.2 Method blank: Analyzed prior to environmental samples. Method blanks are matrix specific.

11.2.1 Preparation of method blank

11.2.2 Water blank preparation: Fill a 40 mL VOA vial with UV-treated deionized water (headspace should not be present). Place vial in Autosampler. Archon autosampler adds 1 uL of 250 ug/mL internal and surrogate standard mixtures.

11.2.3 Soil blank preparation: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial containing a stir bar. 5 mL of UV-treated deionized water is added to the vial. The vial is placed on the Autosampler. The Archon autosampler adds 5 mL of UV-treated deionized water containing 1 uL of 250 ug/mL internal and surrogate standards mixtures. A 2 minute preheat (40° C) and heated purge (40° C) is utilized.

11.2.4 Middle-level extraction blank: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 10 mL of methanol is added to the vial. The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The Archon autosampler adds 1 uL of 250 ug/mL internal standard mixture and surrogate standards mixtures.

11.3 Following the method blank a matrix specific LCS containing selected 8260B target analytes is analyzed. An LCS/LCS duplicate analyses is performed when the client does not provide sufficient volume for MS/MSD analyses.

11.3.1 Water and low-level soil LCS preparation: Refer to Section 7.0. **NOTE:** For low-level soil LCS, 2 minute preheat and heated purge (40° C) is utilized.

11.3.2 Middle-level extraction LCS: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 8.5 mL of methanol and 0.5 mL of the LCS mixtures are added to the vial (**NOTE:** 8.5 mL methanol volume is dependent upon the number of LCS mixtures added; extract final volume is 10 mL). The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The dilution is loaded on the Autosampler and analyzed.

11.4 Matrix spike/matrix spike duplicates (MS/MSD) are analyzed when the client provides appropriate sample volume.

11.4.1 Water MS/MSD preparation: Refer to Section 7.0 with the exception of sample used in place of DI water.

11.4.2 Low-level and mid-level soil MS/MSD preparation: Refer to Section 7.0 with the exception of 5.00 g (± 0.1 g) sample used in place of reagent sand.

11.5 Sample/sample duplicate analyses may be analyzed providing there is appropriate volume. Sample/sample duplicate analyses are generally associated with concentrated soil/waste samples and oils and are used to determine precision.

11.6 Samples are prepared per Microbac SOP PAT01.

11.7 Samples are analyzed within the 12 hour tune, which begins with the injection of BFB. At the end of tune time, a new BFB and CCV must be injected.

11.8 Once sample analysis is complete, a computer generated quantitation report containing all target analytes and their concentrations is generated. Also, detailed spectrum are generated for all target analytes detected above a nominal amount.

11.9 Qualitative analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra may be obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of the sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

11.9.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.9.2 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst judgment may be used even if these criteria are not met. The positive identification of a hit should not be made based solely on the criteria mentioned above.

11.10 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Water and mid-level soil dilutions are prepared by using syringes or pipets to transfer aliquots of sample into a volumetric flask containing DI water. Examples of water dilutions are presented below.

DILUTION	SAMPLE VOLUME (mL)	DI WATER VOLUME (mL)	FINAL DILUTION VOLUME (mL)
10x	5	45	50
50x	1	49	50
100x	0.5	49.5	50
1000x	0.05	49.95	50

Low-level soil dilutions are prepared by weighing an aliquot less than 5.00 g in a 40 mL VOA vial. Examples of low-level soil dilutions:

DILUTION	SAMPLE AMOUNT (g)	DI WATER VOLUME (mL)
2x	2.5	5
2.5x	2.0	5
5x	1.0	5

11.10.1 Low level soils collected via 5035A must utilize the mid-level aliquot for dilutions.

Mid-level soil dilutions are prepared by diluting an aliquot of the methanol extract in a volumetric flask. Examples of mid-level soil dilutions:

DILUTION	METHANOL EXTRACT VOLUME (mL)	DI WATER VOLUME (mL)	FINAL VOLUME (mL)
50x	1	49	50
100x	0.5	49.5	50
500x	0.1	49.9	50
1000x	0.05	49.95	50

11.11 The raw data is processed using the chem station software and the data is uploaded into the LIMS. The laboratory then performs a primary and secondary review of the raw data and quality control forms.

11.12 Tentatively identified compounds (TIC): For samples containing components not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. Guidelines for making tentative identification are:

11.12.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

11.12.2 The relative intensities of the major ions must agree within ±40% for TIC's. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% -70%.)

11.12.3 Molecular ions present in the reference spectrum must be present in the sample spectrum.

11.12.4 Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

11.12.5 Ions present in the reference spectrum but not in the sample spectrum must 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.

11.13 Wipe Sample analysis: Wipes must be stored in 40 mL VOA vials containing 10 mL of methanol after collection. Prior to analysis, a glass rod (or similar) is inserted through the septum to completely immerse the wipe in methanol. Vortex the vial for 20 seconds. Remove ample volume of methanol by inserting the needle of a syringe (or similar) through the septum for a 50x dilution. Prepare as per the mid-level extraction procedures.

12.0 DETAILS OF CALCULATIONS

12.1 Relative response factor (RRF):

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_x = Area of the characteristic ion for the surrogate or 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 surrogate or compound being measured.

12.2 Average RRF $(\overline{RRF})$:

$$\overline{RRF} = \frac{\sum_1^n RRF}{n}$$

12.3 Standard deviation (s):

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n-1}}$$

12.4 Percent relative standard deviation (%RSD):

$$\%RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:

$\bar{x} = \overline{RRF}$: $\overline{RRF} = \frac{\sum_1^n RRF}{n}$

s = standard deviation(s): $s = \sqrt{\frac{\sum (x - \bar{x})^2}{n-1}}$

12.5 Percent recovery (%R)

12.5.1 LCS, surrogate:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.5.2 MS/MSD:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.6 Relative percent difference (RPD):

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = concentration of the first sample
C_2 = concentration of the second sample

12.7 Percent difference (%D), percent drift (% drift):

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.8 Coefficient of correlation (r):

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.8 Coefficient of determination (COD):

$$\left[\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}\right]^2$$

12.9 Sample concentration using $\overline{RRF}$:

12.9.1 Water (ug/L):

$$ug/L = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

where :

A_x = area of characteristic ion for compound being measured
I_s = amount of internal standard injected (250ng)
A_{is} = area of characteristic ion for the internal standard
$\overline{RRF}$ = mean relative response factor for compound being measured

V_o = volume of water purged (10mL)
DF = dilution factor

12.9.2 Low-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)}{(A_{is})(\overline{RRF})(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

12.9.3 Medium-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(\overline{RRF})(V_o)(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, V_o = same as for water
W_s = weight of sample extracted in grams
V_t = volume of total extract (mL) = $V_m + \left[(W_s)\left(\frac{100-D}{100}\right)\right]$
V_i = volume of extract added (mL) for purging
D = % dry weight of sample (not applicable for a wet-weight basis)
DF = dilution factor
V_m = volume of methanol added (mL)

12.10 Linear calibration calculations:

12.10.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
And m and b are the slope and intercept from the regression equation

For a given response ratio we can solve for C_x/C_{istd} :

12.10.2 $$C_x/C_{istd} = \left[R_x/R_{istd} - b\right]/m$$

Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x.

12.11 Quadratic calibration calculations:

The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

12.11.1 $$y = ax^2 + bx + c$$

OR

12.11.2 $$ax^2 + bx + (c - y) = 0$$

Solving for x using the quadratic equation:

12.11.3 $$x = \frac{b \pm \sqrt{(b^2 - 4a(c - y))}}{2a}$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
a ,b, c are constants from the regression equation
Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x

12.12 Solving for the concentration in water sample:

For a given concentration ratio, compute the unknown, C_x

$$C_x = (C_{is})\left(C_x/C_{istd}\right)\left(V_f/V_i\right)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample in ug/L

12.13 Solving for the concentration in soil sample:

$$C_x = (C_{is})\left(C_x / C_{istd}\right)\left(V_f / W_i\right)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample (ug/Kg) (as received)

12.14 Tentatively identified compounds (TIC) estimated concentration determination:

12.14.1 TIC water (ug/L):

$$ug/L = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

where :

A_x = total area of the peak from the total ion chromatogram
I_s = amount of internal standard injected (250ng)
A_{is} = total area of the internal standard from the total ion chromatogram
$\overline{RRF}$ = 1
V_o = volume of water purged (10mL)
DF = dilution factor

12.14.2 TIC low-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)}{(A_{is})(\overline{RRF})(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

12.14.3 TIC medium-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(\overline{RRF})(V_o)(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, V_o = same as for water
W_s = weight of sample extracted in grams
V_t = volume of total extract (mL) = $V_m + \left[(W_s)\left(\frac{100-D}{100}\right)\right]$
V_i = volume of extract added (mL) for purging
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis
DF = dilution factor
V_m = volume of methanol added (mL)

12.15 Wipe:

$$ug/wipe = (C)(D_F)(E_V)$$

where:

C = extract concentration, ug/L
D_F = dilution factor
E_V = extract volume, L/wipe **NOTE:** E_V assumed to be 0.01 L

13.0 QUALITY CONTROL (QC) REQUIREMENTS

13.1 The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for method blanks (MB), laboratory control samples (LCS), matrix spikes (MS), matrix spike duplicates (MSD), laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in Section 8.0 for initial calibration, initial calibration verification (ICV) using a second source, and continuing calibration verification (CCV), and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 8.

13.2 Workgroups are analytical batches that contain instrument performance checks (BFB), calibration standards (ICAL, ICV, CCV), QC samples, and client samples.

13.3 Workgroups are comprised of:

- Instrument performance check: BFB tune evaluation to verify detector is working properly
- ICAL/CCV standards: used to calibrate instrument or verify accuracy of the calibration curve
- ICV: standard from an alternate source used to verify accuracy of the calibration curve
- Method blank: verify system is free of contaminants and interferences
- LCS/LCSD: verify precision and accuracy of the system
- MS/MSD: measure matrix effect of environmental sample on target analytes; measure precision
- Sample/sample duplicate: dual analysis of environmental system to measure precision
- Environmental sample: samples submitted for analysis

13.4 Method blank analyzed per method requirements. Target analytes must be less than ½ the RL. All blanks will be evaluated down to the current MDL for the presence of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL will be reported in the LIMS and these values will appear on the QC summary sheet for the batch.

13.5 The LCS must be evaluated using acceptance criteria listed in Tables 3 and 4, as well as any project specific criteria. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 3 and 4 are used in the absence of a project QAPP or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare a corrective action report (CAR), and contact the department supervisor

for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare a (CAR) to be reviewed by the department supervisor.

13.6 The MS/MSD is analyzed per method requirements. MS/MSD results are included in the QC summary report and are used to monitor matrix accuracy and precision. For MS/MSD, Sample/sample duplicate, failure to meet surrogate and internal standard areas acceptance criteria does not necessarily warrant corrective action. Sample MS/MSD or sample/duplicate results may be used to confirm sample matrix interference. In obvious cases of error, reanalysis would be performed.

13.7 When ICAL acceptance criteria is not met, corrective action may include (but is not limited) to the following:

- Evaluate individual data points and reanalyze
- Evaluate calibration standards and reanalyze
- Prepare fresh calibration standards and reanalyze
- Perform instrument maintenance to include but not limited to:
    - Reanalyze calibration curve
    - Tune instrument
    - Replace trap
    - Bake analytical system
    - Replace column
    - Replace transfer line(s)
    - Service auto-sampler, sample concentrator, gas chromatograph, and/or mass spectrometer
- Qualify results and address in case narrative

13.8 When CCV acceptance criteria is not met, corrective action may include (but not, limited to) the following:

- Reanalyze CCV
- Prepare fresh standards
- Bake analytical system
- Perform instrument maintenance

13.9 Surrogate is added to all standards, QC samples, and environmental samples. Table 7 lists surrogate acceptance limits.

(Note: Ohio VAP requires that all surrogates be within the acceptable limits, except in cases of obvious matrix interference, and the run may not be reported if the method blank or LCS have any surrogates out.)

13.10 Storage blanks are placed in sample refrigerators and freezers to monitor potential cross contamination. Storage blanks consists of 40 mL of analyte free DI water or 5 mL DI water and 5.00 g of sand stored for 14 days in each VOA refrigerator/freezer. Analyses are performed via Method 8260B with results quantitated to the MDL. Storage blanks are prepared weekly and logged into the LIMS laboratory account. Weekly, (after storage blanks have been stored for two full weeks) storage blanks are analyzed via 8260B (storage blanks may be analyzed outside the 12 hour tune time).

Target analytes must be less than ½ the reporting limit with the exception of common lab contaminants. Common lab contaminants must be less than the reporting limit. During primary review of the data, the analyst will review storage blank results to ensure acceptance criteria are met. If acceptance criteria are not met the analyst must initiate corrective action. Corrective action begins with determining the non-compliant analyte(s) and recording any known reason for the failure then reanalysis of the duplicate vial of the storage blank. If reanalysis of the storage blank yields results within acceptance criteria then no further corrective action is required. If the reanalysis results confirm the initial analysis results or the reason for the initial failure is not evident then a Form NC02 is initiated. After primary review, results are uploaded to the LIMS. The laboratory will conduct an internal investigation and assess impact on associated samples if these criteria are not met with the next group of storage blanks analyzed. The laboratory will attempt to identify the source of contamination, and evaluate the impact on data reported for the contaminant during the period of storage. Clients may be contacted based on the investigation, if the QAO judges that to be necessary.

13.11 Procedures for handling out-of-control data:

When out-of-control situations are encountered, the laboratory will proceed as outlined in Table 8, unless the project QAPP specifies other corrective actions. Corrective actions may include a variety of actions such as recalibration and re-analyzing all affected samples. If these measures fail, the client may be informed of the problem in order to obtain directions for re-preparation and re-analysis, data qualification or rejection (R-flagging), or re-sampling. The laboratory (analyst) will document all out-of-control situations with the preparation of the "Analytical Corrective Action" (CAR) forms, which are reviewed and signed by the department manager and the QAO. The laboratory will submit copies of all CAR's to the client service manager, so that the service representative may inform the client(s) affected by the non-compliant data. These forms are kept on file and are available for review.

13.12 Common contingencies not requiring re-analysis:

In many situations it may not be necessary to perform sample re-analysis for the following quality control failures, provided they are not a chronic problem or indicative of a trend. The laboratory provides documentation in the report narrative and the project files. There are some standard contingencies to normal corrective action measures that may be invoked provided they comply with the project QAPP requirements.

13.12.1 An LCS or surrogate recovery exceeds the upper control limit, but the corresponding samples results are non-detect.

13.12.2 A method blank exceeds the upper limit, but the corresponding samples results are non-detect.

13.12.3 A method blank exceeds the upper limit, but the corresponding samples results are greater than twenty (20) times the level in the blank.

13.13 Table 7 contains method 8260B quality control criteria.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review:

14.1.1 All data undergoes a 100% primary review to ensure method and project specific compliance, reduce the data into reportable results, and generate appropriate QC forms. All items in Figure 1 (data review checklist) are reviewed and results are uploaded to the LIMS.

14.1.2 Following the primary review the data undergoes a 100% peer review. All items in 14.1.1 are repeated by the peer. The peer review may be performed by the supervisor or designee.

14.2 Data reporting:

14.2.1 Following peer review all uploaded results are reviewed, verified, and qualified.

14.2.2 Default reporting units are "ug/L" for water and "ug/Kg" for soil/sediments/oils.

14.2.3 All uploaded results are uploaded to a maximum number of significant figures dictated by the LIMS. The number of significant figures in the final report vary per project requirements.

14.2.4 Dilution and sample matrix confirmation analyses are uploaded into the LIMS and per the client's request may be reported as separate analyses or combined (concatenated) into one set of results.

14.3 Quantitative results between the MDL and RL will be qualified as "estimated" if requested by the client.

14.4 Refer to Microbac SOP 41 for acceptable procedure on manual integration if necessary.

15.0 PREVENTATIVE MAINTENANCE

15.1 Gas pressures are monitored daily. Other maintenance performed as needed. Laboratory maintenance log books maintained per instrument.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Laboratory policies and procedures for management of hazardous waste are found in SOP 33-Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal hazardous waste that this is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA, and Ohio VAP rules allow the suspension of our certification for failure to comply with these laws.

Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams are as follows:

- Volatile Laboratory – non-halogenated solvents, solid waste (methanol)

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste,* SW-846, US-EPA, Office of Solid Waste, including updates I, II, III, IV:

8000B	December 1996
8000C	March 2003
8260A	September 1994
8260B	December 1996
5030A	July 1992
5030B	December 1996
5030C	May 2003
5035A	July 2002

17.2 AFCEE 1998 QAPP, Version 3.0, March 1998

17.3 AFCEE 2001 QAPP, Version 3.1, August 2001

17.4 AFCEE 2005 QAPP, Version 4.0, February 2005

Table 1
Method Analytes for MSV01

ANALYTE	CAS NUMBER
1,1,1,2-tetrachloroethane	630-20-6
1,1,1-trichloroethane	71-55-6
1,1,2,2-tetrachloroethane	79-34-5
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1
1,1,2-trichloroethane	79-00-5
1,1-dichloroethane	75-34-3
1,1-dichloroethene	75-35-4
1,1-dichloropropene	563-58-6
1,2,3-trichlorobenzene	87-61-6
1,2,3-trichloropropane	98-18-4
1,2,4-trimethylbenzene	95-63-6
1,2,4-trimethylbenzene	95-63-6
1,2-dibromo-3-chloropropane	96-12-8
1,2-dibromoethane	106-93-4
1,2-dichlorobenzene	95-50-1
1,2-dichloroethane	107-06-2
1,2-dichloropropane	78-87-5
1,3,5-trimethylbenzene	108-67-8
1,3-butadiene	106-99-0
1,3-dichlorobenzene	541-73-1
1,3-dichloropropane	142-28-9
1,4-dichlorobenzene	106-46-7
1,4-dioxane	123-91-1
1-butanol	71-36-3
1-chlorohexane	544-10-5
2,2-dichloropropane	594-20-7
2-butanone	78-93-3
2-chloroethylvinylether	110-75-8
2-chlorotoluene	95-49-8
2-hexanone	591-78-6
2-nitropropane	79-46-9
4-chlorotoluene	106-43-4
4-methyl-2-pentanone	108-10-1
acetone	67-64-1
acetonitrile	75-05-8
acrolein	107-02-8
acrylonitrile	107-13-1
allylchloride (3-chloroprene)	107-05-1
a-methyl styrene	98-83-9
benzene	71-43-2
bromobenzene	108-86-1
bromochloromethane	74-97-5
bromodichloromethane	75-27-4
bromoform	75-25-2
bromomethane	74-83-9
carbon disulfide	75-15-0
carbon tetrachloride	56-23-5
chlorobenzene	108-90-7
chloroethane	75-00-3
chloroform	67-66-3
chloromethane	74-87-3
-	-

ANALYTE	CAS NUMBER
chloroprene (2-chloro-1,3-butadiene)	126-99-8
cis-1,2-dichloroethene	156-59-2
cis-1,3-dichloropropene	10061-01-5
cyclohexane	110-82-7
cyclohexanone	108-94-1
dibromochloromethane	124-48-1
dibromomethane	74-95-3
dichlorodifluoromethane	75-71-8
diethyl ether	60-29-7
diisopropyl ether	108-20-3
dimethyl disulfide	624-92-0
dimethylsulfide	75-18-3
ethyl acetate	141-78-6
ethyl ether	60-29-7
ethyl methacrylate	97-63-2
ethyl t-butyl ether	637-92-3
ethylbenzene	100-41-4
hexachlorobutadiene	87-68-3
iodomethane	74-88-4
isobutanol	78-83-1
isoprene	78-79-5
isopropyl benzene	98-82-8
m+p-xylene	108-38-3 & 106-42-3
methacrylonitrile	126-98-7
methyl acetate	79-20-9
methyl cyclohexane	108-87-2
methylene chloride	75-09-2
methylmethacrylate	80-62-6
methyl-tert-butyl-ether	1634-04-4
naphthalene	91-20-3
n-butyl-benzene	104-51-8
n-heptane	142-82-5
n-hexane	110-54-3
n-propyl benzene	103-65-1
o-xylene	95-47-6
p-isopropyl-toluene	99-87-6
propionitrile (ethyl cyanide)	107-12-0
sec-butyl-benzene	135-98-8
styrene	100-42-5
t-amylmethyl ether	994-05-8
t-butanol	75-65-0
tert-butyl-benzene	98-06-6
tetrachloroethene	127-18-4
tetrahydrofuran	109-99-9
toluene	108-88-3
trans-1,2-dichloroethene	156-60-5
trans-1,3-dichloropropene	10061-02-6
trans-1,4-dichloro-2-butene	110-57-6
trichloroethene	79-01-6
trichlorofluoromethane	75-69-4
vinyl acetate	108-05-4
vinyl chloride	75-01-4

Table 2
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
1,1,1,2-tetrachloroethane	630-20-6	80-130	20	0.25	5	20	5-200
1,1,1-trichloroethane	71-55-6	80-134	20	0.25	5	20	5-200
1,1,2,2-tetrachloroethane	79-34-5	79-125	20	0.125	5	20	5-200
1,1,2-trichloro-1,2,2-trif	76-13-1	40-160	20	1	10	20	5-200
1,1,2-trichloroethane	79-00-5	80-125	20	0.25	5	20	5-200
1,1-dichloroethane	75-34-3	80-125	20	0.125	5	20	5-200
1,1-dichloroethene	75-35-4	80-132	20	0.5	5	20	5-200
1,1-dichloropropene	563-58-6	75-130	20	0.25	5	20	5-200
1,2,3-trichlorobenzene	87-61-6	55-140	20	0.125	5	20	5-200
1,2,3-trichloropropane	96-18-4	75-125	20	0.75	5	20	5-200
1,2,4-trichlorobenzene	120-82-1	65-135	20	0.2	5	20	5-200
1,2,4-trimethylbenzene	95-63-6	80-125	20	0.25	5	20	5-200
1,2-dibromo-3-chloropropane	96-12-8	50-130	20	1.0	5	20	5-200
1,2-dibromoethane	106-93-4	80-129	20	0.25	5	20	5-200
1,2-dichlorobenzene	95-50-1	80-125	20	0.125	5	20	5-200
1,2-dichloroethane	107-06-2	80-129	20	0.25	5	20	5-200
1,2-dichloroethene (total)	156-59-2 + 156-60-5	80-124	20	0.25	5	40	5-200
1,2-dichloropropane	78-87-5	80-120	20	0.2	5	20	5-200
1,3,5-trimethylbenzene	108-67-8	80-127	20	0.25	5	20	5-200
1,3-butadiene	106-99-0	10-200	20	1	10	20	5-200
1,3-dichlorobenzene	541-73-1	80-120	20	0.25	5	20	5-200
1,3-dichloropropane	142-28-9	80-120	20	0.2	5	20	5-200
1,4-dichlorobenzene	106-46-7	80-120	20	0.125	5	20	5-200
1,4-dioxane	123-91-1	20-160	20	50	100	200	5-200
1-butanol	71-36-3	50-150	20	50	100	200	50-400
1-chlorohexane	544-10-5	80-127	20	0.125	1	20	5-200
2,2-dichloropropane	594-20-7	80-133	20	0.25	5	20	5-200
2-butanone	78-93-3	10-170	20	2.5	10	20	5-200
2-chloroethyl vinyl ether	110-75-8	45-160	20	2.0	10	20	5-200
2-chlorotoluene	95-49-8	80-127	20	0.125	5	20	5-200
2-hexanone	591-78-6	55-130	20	2.5	10	20	5-200
2-nitropropane	79-46-9	10-150	20	5	50	100	5-200
3-chloro-1-propene	107-05-1	70-130	20	NA	10	20	5-200
4-chlorotoluene	106-43-4	80-126	20	0.25	5	20	5-200
4-methyl-2-pentanone	108-10-1	64-140	20	2.5	10	20	5-200
acetone	67-64-1	40-180	20	2.5	10	20	5-200
acetonitrile	75-05-8	70-130	20	5	100	100	10-200
Acrolein	107-02-8	10-200	20	20	100	20	5-200
acrylonitrile	107-13-1	50-150	20	2.5	100	20	5-200
alpha-methyl-styrene	98-83-9	50-150	20	0.5	10	20	5-200
benzene	71-43-2	80-121	20	0.125	5	20	5-200
bromobenzene	108-86-1	80-120	20	0.125	5	20	5-200
bromochloromethane	74-97-5	65-130	20	0.2	5	20	5-200
bromodichloromethane	75-27-4	80-131	20	0.25	5	20	5-200
bromoform	75-25-2	70-130	20	0.5	5	20	5-200

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)

bromomethane	74-83-9	30-145	20	0.50	10	20	5-200
carbon disulfide	75-15-0	58-128	20	0.50	5	20	5-200
carbon tetrachloride	56-23-5	65-140	20	0.25	5	20	5-200
chlorobenzene	108-90-7	80-120	20	0.125	5	20	5-200
chloroethane	75-00-3	60-135	20	0.5	10	20	5-200
chloroform	67-66-3	80-125	20	0.125	5	20	5-200
chloromethane	74-87-3	40-125	20	0.25	10	20	5-200
chloroprene	126-99-8	70-130	20	2.5	100	100	5-200
cis-1,2-dichloroethene	156-59-2	70-125	20	0.25	5	20	5-200
cis-1,3-dichloropropene	10061-01-5	70-130	20	0.25	5	20	5-200
cyclohexane	110-82-7	70-130	20	0.58	10	20	5-200
cyclohexanone	108-94-1	10-140	20	5	100	100	5-200
dibromochloromethane	124-48-1	60-135	20	0.25	5	20	5-200
dibromomethane	74-95-3	75-125	20	0.25	5	20	5-200
dichlorodifluoromethane	75-71-8	40-160	20	0.25	5	20	5-200
diethyl ether	60-29-7	70-130	20	5	10	100	5-200
diisopropyl ether	108-20-3	70-130	20	5	10	100	5-200
dimethyl disulfide	624-92-0	70-130	20	1.0	10	20	5-200
dimethyl sulfide	75-18-3	70-130	20	0.5	10	20	5-200
ethyl acetate	141-78-6	70-130	20	5	50	100	5-200
ethyl benzene	100-41-4	80-122	20	0.25	5	20	5-200
ethyl methacrylate	97-63-2	70-130	20	1.0	10	20	5-200
ethyl-tert-butyl ether	637-92-3	70-130	20	5	10	100	5-200
hexachlorobutadiene	87-68-3	72-132	20	0.25	5	20	5-200
iodomethane	74-88-4	10-160	20	0.5	10	20	5-200
Isobutanol	78-83-1	10-180	20	50	100	200	50-400
isoprene	78-79-5	70-130	20	0.53	10	20	5-200
isopropylbenzene	98-82-8	80-122	20	0.25	5	20	5-200
m+p-xylene **	108-38-3 + 106-42-3	80-122	20	0.5	5	40	5-400
methacrylonitrile	126-98-7	70-130	20	2.5	5	100	5-200
Methyl acetate	79-20-9	50-190	20	1	10	20	5-200
Methyl cyclohexane	108-87-2	80-130	20	1	10	20	5-200
methyl methacrylate	80-62-6	70-130	20	2.5	5	100	5-200
methylene chloride	75-09-2	80-123	20	0.25	5	20	5-200
methyl-tert-butyl ether	1634-04-4	65-125	20	0.5	5	20	5-200
naphthalene	91-20-3	59-149	20	0.2	5	20	5-200
n-butylbenzene	104-51-8	80-131	20	0.25	5	20	5-200
n-heptane	142-82-5	70-130	20	2.5	5	20	5-200
n-hexane	110-54-3	74-137	20	0.56	5	20	5-200
o-xylene	95-47-6	80-122	20	0.25	5	20	5-200
p-isopropyl-toluene	99-87-6	80-122	20	0.25	5	20	5-200
propionitrile	107-12-0	50-150	20	2.5	5	100	5-200
propylbenzene	103-65-1	80-129	20	0.125	5	20	5-200
sec-butylbenzene	135-98-8	80-127	20	0.25	5	20	5-200
styrene	100-42-5	80-123	20	0.125	5	20	5-200
tert-amyl-methyl ether	994-05-8	70-130	20	5	10	100	5-200
tert-butyl alcohol	75-65-0	10-180	20	50	100	200	50-400

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
tert-butylbenzene	98-06-6	80.126	20	0.25	5	20	5-200
tetrachloroethene	127-18-4	80-124	20	0.25	5	20	5-200
tetrahydrofuran	109-99-9	60-140	20	5	50	100	5-200

Toluene	108-88-3	80-124	20	0.25	5	20	5-200
trans-1,2-dichloroethene	156-60-5	80-127	20	0.25	5	20	5-200
trans-1,3-dichloropropene	10061-02-6	80-130	20	0.5	5	20	5-200
trans-1,4-dichloro-2-butene	110-57-6	50-150	20	2.0	10	20	5-200
trichloroethene	79-01-6	80-122	20	0.25	5	20	5-200
trichlorofluoromethane	75-69-4	62-151	20	0.25	5	20	5-200
vinyl acetate	108-05-4	10-190	20	2.5	10	20	5-200
vinyl chloride	75-01-4	50-170	20	0.25	10	20	5-200
xylenes (total)	108-38-3 + 106-42-3 + 95-47-6	80-121	20	0.5	15	60	5-200

** Unresolvable compound

Table 3
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
1,1,1,2-tetrachloroethane	630-20-6	71-137	30	0.5	5	20	5-200
1,1,1-trichloroethane	71-55-6	70-135	30	0.5	5	20	5-200

1,1,2,2-tetrachloroethane	79-34-5	55-130	30	0.5	5	20	5-200
1,1,2-trichloro-1,2,2-trif	76-13-1	70-130	30	1.0	10	20	5-200
1,1,2-trichloroethane	79-00-5	60-125	30	0.5	5	20	5-200
1,1-dichloroethane	75-34-3	75-125	30	1.0	5	20	5-200
1,1-dichloroethene	75-35-4	65-135	30	0.5	5	20	5-200
1,1-dichloropropene	563-58-6	57-138	30	0.5	5	20	5-200
1,2,3-trichlorobenzene	87-61-6	60-135	30	0.5	5	20	5-200
1,2,3-trichloropropane	96-18-4	65-130	30	1.0	5	20	5-200
1,2,4-trichlorobenzene	120-82-1	65-130	30	0.5	5	20	5-200
1,2,4-trimethylbenzene	95-63-6	75-132	30	0.5	5	20	5-200
1,2-dibromo-3-chloropropane	96-12-8	40-135	30	2.0	5	20	5-200
1,2-dibromoethane	106-93-4	69-128	30	0.5	5	20	5-200
1,2-dichlorobenzene	95-50-1	70-130	30	0.5	5	20	5-200
1,2-dichloroethane	107-06-2	63-133	30	0.5	5	20	5-200
1,2-dichloroethene (total)	156-59-2 + 156-60-5	74-127	30	0.5	5	40	5-200
1,2-dichloropropane	78-87-5	72-130	30	0.5	5	20	5-200
1,3,5-trimethylbenzene	108-67-8	74-133	30	0.5	5	20	5-200
1,3-butadiene	106-99-0	40-160	30	1	10	20	10-200
1,3-dichlorobenzene	541-73-1	70-130	30	0.5	5	20	5-200
1,3-dichloropropane	142-28-9	65-128	30	0.5	5	20	5-200
1,4-dichlorobenzene	106-46-7	70-130	30	0.5	5	20	5-200
1,4-dioxane	123-91-1	50-150	30	50	100	200	5-200
1-butanol	71-36-3	50-150	30	50	100	200	50-400
1-chlorohexane	544-10-5	40-160	30	0.5	3	20	5-200
2,2-dichloropropane	594-20-7	66-135	30	0.5	5	20	5-200
2-butanone	78-93-3	37-180	30	2.5	100	20	5-200
2-chloroethyl vinyl ether	110-75-8	35-154	30	2.0	10	20	5-200
2-chlorotoluene	95-49-8	63-147	30	0.5	5	20	5-200
2-hexanone	591-78-6	45-145	30	2.5	10	20	5-200
2-nitropropane	79-46-9	60-140	30	5	50	100	5-200
3-choro-1-propene	107-05-1	50-150	30	2.5	10	20	5-200
4-chlorotoluene	106-43-4	70-138	30	0.5	5	20	5-200
4-methyl-2-pentanone	108-10-1	47-146	30	2.5	10	20	5-200
acetone	67-64-1	20-160	30	5.0	100	20	5-200
acetonitrile	75-05-8	50-150	30	50	100	100	5-200
acrolein	107-02-8	50-150	30	20	100	20	5-200
acrylonitrile	107-13-1	60-140	30	2.5	100	20	5-200
alpha-methyl-styrene	98-83-9	70-130	30	0.5	10	20	5-200
benzene	71-43-2	70-130	30	0.5	5	20	5-200
bromobenzene	108-86-1	72-131	30	0.5	5	20	5-200
bromochloromethane	74-97-5	70-130	30	0.5	5	20	5-200
bromodichloromethane	75-27-4	72-137	30	0.5	5	20	5-200
bromoform	75-25-2	49-136	30	0.5	5	20	5-200
bromomethane	74-83-9	37-143	30	1.0	10	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
carbon disulfide	75-15-0	39-139	30	0.5	5	20	5-200
carbon tetrachloride	56-23-5	59-136	30	0.5	5	20	5-200
chlorobenzene	108-90-7	70-130	30	0.5	5	20	5-200
chloroethane	75-00-3	52-135	30	1.0	10	20	5-200
chloroform	67-66-3	74-129	30	0.5	5	20	5-200
chloromethane	74-87-3	30-131	30	2.0	10	20	5-200
chloroprene	126-99-8	50-150	30	2.5	5	100	5-200

cis-1,2-dichloroethene	156-59-2	70-130	30	0.5	5	20	5-200
cis-1,3-dichloropropene	10061-01-5	70-142	30	0.5	5	20	5-200
cyclohexane	110-82-7	70-130	30	0.5	10	20	5-200
cyclohexanone	108-94-1	60-140	30	5	50	100	5-200
dibromochloromethane	124-48-1	59-136	30	0.5	5	20	5-200
dibromomethane	74-95-3	69-130	30	0.5	5	20	5-200
dichlorodifluoromethane	75-71-8	25-130	30	1.0	5	20	5-200
diethyl ether	60-29-7	60-140	30	5	10	100	5-200
diisopropyl ether	108-20-3	60-140	30	5	10	100	5-200
dimethyl disulfide	624-92-0	60-140	30	0.5	10	20	5-200
dimethyl sulfide	75-18-3	60-140	30	0.5	10	20	5-200
ethyl acetate	141-78-6	60-140	30	5	50	100	5-200
ethyl benzene	100-41-4	70-130	30	0.5	5	20	5-200
ethyl methacrylate	97-63-2	60-140	30	1.0	10	20	5-200
ethyl-tert-butyl ether	637-92-3	60-140	30	5	10	100	5-200
hexachlorobutadiene	87-68-3	65-135	30	0.5	5	20	5-200
iodomethane	74-88-4	20-288	30	1.0	10	20	5-200
isobutanol	78-83-1	50-150	30	50	100	200	10-400
Isoprene	78-79-5	40-140	30	2.0	10	20	5-200
isopropylbenzene	98-82-8	68-129	30	0.5	5	20	5-200
m+p-xylene **	108-38-3 + 106-42-3	70-130	30	0.5	5	40	5-200
methacrylonitrile	126-98-7	60-140	30	2.5	5	100	5-200
methyl acetate	79-20-9	70-130	30	1	10	20	5-200
methyl cyclohexane	108-87-2	70-130	30	1	10	20	5-200
methyl methacrylate	80-62-6	70-130	30	2.5	5	100	5-200
methylene chloride	75-09-2	74-128	30	1.0	5	20	5-200
methyl-tert-butyl ether	1634-04-4	54-151	30	0.5	5	20	5-200
naphthalene	91-20-3	50-146	30	0.5	5	20	5-200
n-butylbenzene	104-51-8	70-136	30	0.5	5	20	5-200
n-heptane	142-82-5	60-140	30	2.5	5	20	5-200
n-hexane	110-54-3	58-142	30	0.5	10	20	5-200
o-xylene	95-47-6	70-130	30	0.5	5	20	5-200
p-isopropyl-toluene	99-87-6	72-128	30	0.5	5	20	5-200
propionitrile	107-12-0	60-140	30	2.5	5	100	5-200
propylbenzene	103-65-1	72-136	30	0.5	5	20	5-200
sec-butylbenzene	135-98-8	71-132	30	0.5	5	20	5-200
styrene	100-42-5	74-130	30	0.5	5	20	5-200
tert-amyl-methyl ether	994-05-8	60-140	30	5	10	100	5-200
tert-butyl alcohol	75-65-0	50-150	30	50	100	200	50-400
tert-butylbenzene	98-06-6	72-130	30	0.5	5	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
tetrachloroethene	127-18-4	72-130	30	0.5	5	20	5-200
tetrahydrofuran	109-99-9	50-150	30	25	50	100	5-200
toluene	108-88-3	77-126	30	0.5	5	20	5-200
trans-1,2-dichloroethene	156-60-5	72-127	30	0.5	5	20	5-200
trans-1,3-dichloropropene	10061-02-6	65-139	30	0.5	5	20	5-200
trans-1,4-dichloro-2-butene	110-57-6	50-150	30	1.0	10	20	5-200
trichloroethene	79-01-6	72-126	30	0.5	5	20	5-200
trichlorofluoromethane	75-69-4	48-154	30	1.0	5	20	5-200
vinyl acetate	108-05-4	10-150	30	2.5	10	20	5-200

vinyl chloride	75-01-4	45-140	30	1.0	10	20	5-200
xylenes (total)	108-38-3 + 106-42-3 + 95-47-6	70-130	30	0.5	5	60	5-200

** Unresolvable compound

Table 4*

GC/MS PURGE AND TRAP PARAMETERS	
purge time	9 – 11 minutes
dry purge time	0 – 2 minutes
desorb preheat	245° C
desorb	0.5 – 1 minute at 250° C
bake	10 -12 minutes at 260° C
LSC temp	valve 150° C, lines: 150° C
Archon temp	valve 120° C, lines 100° C
GAS CHROMATOGRAPH PARAMETERS	
carrier gas	helium, 99.999 %
injector temperature	220° C
oven temperature program	35° C for 4 minutes 10° C/minute to 240° C (hold 2 minutes)
MASS SPECTROMETER PARAMETERS	
beginning mass	35
ending mass	265
scan rate / sampling	8 scans/sec

* Denotes suggested parameters; systems may be adjusted to improve efficiency.

Table 5
BFB Key Ion Abundance Criteria

MASS	ION ABUNDANCE CRITERIA
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174

176	greater than 95%, but less than 101% of mass 174
177	5 to 9% of mass 176

Table 6
Surrogate Standards Recovery Limits

SURROGATE COMPOUND	8260 WATER *	AFCEE 1998 QAPP WATER	8260 SOIL *	AFCEE 1998 QAPP (3.0) SOIL	2001/2005 AFCEE WATER	2001/2005 AFCEE SOIL	8260 OIL
dibromofluoromethane	86-118	75-125	80-120	65-135	85-115	65-135	52-122
Toluene-d_8	88-110	75-125	81-117	65-135	81-120	84-116	35-127
4-bromofluorobenzene	86-115	75-125	74-121	65-135	76-119	84-118	26-158
1,2-dichloroethane-d_4	80-120	62-139	80-120	52-149	72-119	52-149	43-128

* Denotes one outlier permitted given % R > 10%. Reanalysis required if two or more recovery results are outside acceptance limits.

Table 7
Quality Control Criteria
Volatile GC/MS Analysis
Method 8260B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Mass spectral ion intensities (BFB criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 6	Retune instrument and repeat BFB check

Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered.	≤30% RSD for CCC compounds, < 15% RSD for all target compounds, if >15% RSD, then linear regression, provided r ≥ 0.995,then quadratic regression, provided $r^2 \geq 0.990$ SPCC minimum $\overline{RRF}$	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1) See section 13.7 for additional corrective action.
Second source calibration verification (ICV)	After each initial calibration	≤ 30% drift for each analyte (1)	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Each 12 hours	SPCC minimum RRF, CCC percent drift ≤ 20%; other target analytes ≤ 40% (1)	Re-analyze CCV; upon second failure, repeat initial calibration (1) See Section 13.8 for additional corrective action.
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 seconds of IS retention time in ICAL midpoint STD and area within –50% t +100% of IS midpoint area	Check for MS malfunctions or interference; re-analyze sample
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch	< ½ RL	Notify supervisor and initiate CAR; investigate; re-analyze samples
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate CAR; investigate; re-analyze samples
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD) Sample/Sample Duplicate	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Qualify data and/or address in the report narrative
Surrogate spike	Every sample, standard, and quality control sample	Recoveries within designated ranges: use project QAPP or standard control criteria; (one surrogate outlier permitted provided % R > 10%) (1)	Notify supervisor and initiate CAR; investigate; re-analyze samples

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

NOTE: See Scope and Application section of SOP for OVAP specific requirements.

Figure 1

Checklist ID: 6687

Microbac Laboratories Inc.

Data Checklist

Date:
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Runlog ID number:	
System Performance Check	
BFB	
Initial Calibration	
Average RF	
Linear Reg or Higher Order Curve	
Second Source standard % Difference	
Continuing Calibration Check Standards	
Project/Client Specific Requirements	
Special Standards	
Blanks	
TCL's	
Surrogates	
LCS (Laboratory Control Sample)	
Recoveries	
Surrogates	
MS/MSD/Duplicates	
Samples	
TCL Hits	
Spectra of TCL Hits	
Surrogates	
Internal Standards Criteria	
Library Searches	
Calculations & Correct Factors	
Dilutions Run	
Reruns	
Manual Integrations	
Case Narrative	
Results Reporting/Data Qualifiers	
KOBRA Workgroup Data	
Check for Completeness	
Primary Reviewer	
Secondary Reviewer	
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Check the reasonableness of the results	X

Primary Reviewer: Secondary Reviewer:

CHECKLIST1 - Modified 03/05/2008
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Microbac

Figure 2
VOA Preparation/Preservation and Extraction Log

Microbac Laboratories Inc.
VOA Preparation/Preservation/Extraction Log

Workgroup (AAB#):WG270339
Method:8260
Reagent ID:RGT10001

Analyst:DGB
Run Date:06/05/2008 09:50

SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total Wt	Sample Wt	Water	MeOH	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.86			30.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	B	04/20/08 11:25	06/05/08 09:51	77.08	5.12	30.1	24.98	5		5	5
L08020355-09	C	04/20/08 11:25	06/05/08 09:52	77.08			25.08		10	15.748816	5

Comments:
1 = improperly sealed cap
2 = preserved out of hold
3 = effervesced
4 = preserved with NaHSO4
5 = preserved by freezing
6 = preserved in field

Analyst: Douglas Butcher

Appendix I
The Analysis of 1,4-Dioxane by Selected Ion Monitoring (SIM), Revision 1

1. Selected ion monitoring (SIM) is used in the acquisition of 1,4-dioxane and associated internal and surrogate standards. The following lists primary and secondary ions:

Parameter	Primary ion	Secondary ion
1,4-dioxane	88	58
1,4-dioxane-d8 (IS)	96	70
dibromofluoromethane (SS)	111	113
1,2-dichloroethane-d4 (SS)	65	67

IS: denotes internal standard
SS: denotes surrogate standard

2. Sample preparation: Water: Allow sample to warm to ambient temperature. Place sample vial in auto-sampler. Auto-sampler transfers 10mL of sample for purging. Heated purge is performed utilizing a 4 minute pre-heat at 60C then a 9 minute purge at 60C. After analysis record sample pH from opened vial. If possible, dilutions performed using un-opened vial.

3. Sample preparation: Soil: Refer to Microbac SOP PAT01.

NOTE: Standards and QC samples also utilize a heated purge; auto-sampler adds internal and surrogate standards.

4. The suggested calibration range is 2 ug/L to 200 ug/L for water; 2 ug/Kg to 200 ug/Kg for soil. The following lists suggested QC acceptance criteria:

MATRIX	RL	MDL*	ACCURACY** (%R)	PRECISION** (RPD)	TRUE VALUE, LCS, MS/MSD	SURROGATE LIMITS** (%R)	SURROGATE TRUE VALUE
Water (ug/L)	2.0	1.0	60-140	30	10	50-150	2.5
Soil (ug/Kg)	2.0	1.0	50-150	30	10	50-150	2.5

* denotes verified MDL (verified annually)
** denotes advisory limits (control limits may vary)

5. This method is not applicable to OVAP samples.

6. Following is corrective action procedures and QC acceptance criteria for SIM 1,4-dioxane analysis.

Quality Control Criteria
Volatile GC/MS SIM, 1,4-Dioxane

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (6)
Mass Spectral ION Intensities (BFB criteria)	Every 12 hours prior to ICAL, ICV, or CCV	Per Table 6	Evaluate, re-inject, re-tune, instrument maintenance
Initial Calibration (ICAL)	When Continuing Calibration is out of control of when system conditions have been altered	%RSD ≤ 30%	Evaluate and use linear or higher order calibration equation, perform corrective action (1)
Second Source Calibration Verification (ICV)	After each initial calibration	% D ≤ 30 %	Reanalyze ICV; upon second failure repeat initial calibration (1)
Continuing Calibration Verification (CCV)	Every 12 hours	%D ≤ 30%	Rerun CCV once more, upon 2nd failure perform corrective action (1)
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch	< ½ RL	Reanalyze blank, perform corrective action.
Laboratory Control Sample/Sample Duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch	Within designated ranges (1,2)	Investigate, evaluate, perform corrective action (5)
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch	Within designated ranges (1,2)	Evaluate, none, if errors not detected
Surrogate Spike	Every sample, standard, and quality control sample	Within designated ranges (1)	Reanalyze if %R < 10% 1 outlier permitted providing %R > 10%.

(1) Evaluation criteria is often project specific, Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

Appendix II
8260A and 8260B Suggested Quantitation Ions

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
fluorobenzene (internal std)	**96**	**77**	**96**
dichlorodifluoromethane	85	87	85
chloromethane	50	52	50
vinyl chloride	62	64	62
1,3-butadiene	N/A	N/A	54
bromomethane	94	96	94
chloroethane	64	66	64
trichlorofluoromethane	151	101, 153	101
diethyl ether	74	45, 59	59
isoprene	67	53	67
acrolein	56	55, 58	56
trichlorotrifluoromethane	101	151	101
acetone	58	43	43
1,1-dichloroethene	96	61, 63	96
t-butyl alcohol	N/A	N/A	59
dimethyl sulfide	62	47	62
iodomethane	142	127, 141	142
methyl acetate	N/A	N/A	43
acetonitrile	41	40, 39	41
methylene chloride	84	86, 49	84
carbon disulfide	76	78	76
acrylonitrile	53	52, 51	53
methyl-tert-butyl ether	73	57	73
3-chloro-1-propene	N/A	N/A	41
trans-1,2-dichloroethene	96	61, 98	96
n-hexane	N/A	N/A	57
diisopropyl ether	N/A	N/A	45
vinyl acetate	43	86	43
1,1-dichloroethane	63	65, 83	63
ethyl-t-butyl ether	N/A	N/A	59
2-butanone	72	43	43
2-chloro-1,3-butadiene	53	88, 90, 51	53
propionitrile	54	52, 55, 40	54
2,2-dichloropropane	77	97	77
cis-1,2-dichloroethene	96	61, 98	96
chloroform	83	85	83
ethyl acetate	88	43, 45, 61	43
bromochloromethane	128	49, 130	128
methacrylonitrile	41	67, 39, 52, 66	67
isobutyl alcohol	43	41, 42, 74	73
tetrahydofuran	N/A	N/A	42
dibromofluoromethane **(surrogate)**	111	113	111
1,1,1-trichloroethane	97	99, 61	97
cyclohexane	56	84	56
1,1-dichloropropene	75	110, 77	75
t-amyl-methyl ether	N/A	N/A	73
carbon tetrachloride	117	119	117
1,2-dichloroethane-d4 **(surrogate)**	65	67	65
Heptane	N/A	N/A	57
1,2-dichloroethane	62	98	62

Appendix II (continued)

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
1-butanol	N/A	N/A	56
benzene	78	77, 52	78
trichloroethene	95	97, 130, 132	130
methylcyclohexane	N/A	N/A	83
1,2-dichloropropane	62	112	62
Methyl methacrylate	69	41, 100, 39	41
1,4-dioxane	88	58, 43, 47	88
bromodichloromethane	83	85, 127	83
2-nitropropane	46	N/A	43
dibromomethane	93	95, 174	93
2-chloroethylvinyl-ether	63	65, 106	63
4-methyl-2-pentanone	100	43, 58, 85	58
cis-1,3-dichloropropene	75	77, 39	75
dimethyl disulfide	79	94	79
chlorobenzene-d5 (internal std)	**117**	**82**	**117**
toluene-d8 **(surrogate)**	98	100	98
toluene	92	91	91
ethyl methacrylate	69	41, 99, 86, 114	69
trans-1,3-dichloropropene	75	77, 39	75
1,1,2-trichloroethane	83	97, 85	97
2-hexanone	43	58, 58, 57, 100	43
1,3-dichloropropane	76	78	76
tetrachloroethene	164	129, 131, 166	164
dibromochloromethane	129	127	129
1,2-dibromoethane	107	109, 188	107
1-chlorohexane	91	55	91
chlorobenzene	112	77, 114	112
1,1,1,2-tetrachloroethane	131	133, 119	131
ethylbenzene	91	106	106
m+p-xylene	106	91	106
cyclohexanone	N/A	N/A	55
o-xylene	106	91	106
styrene	104	78	104
bromoform	173	175, 254	173
isopropylbenzene	105	120	105
1,4-dichlorobenzene-d4 (internal std)	**152**	**115, 150**	**152**
1,1,2,2-tetrachloroethane	83	131, 85	83
p-bromofluorobenzene **(surrogate)**	95	174, 176	95
1,2,3-trichloropropane	75	77, 110	110
trans-1,4-dichloro-2-butene	53	88, 75	53
n-propyl-benzene	91	120	91
bromobenzene	156	77, 158	156
1,3,5-trimethylbenzene	105	120	105
2-chlorotoluene	91	126	91
4-chlorotoluene	91	126	91
alpha-methyl-styrene	118	103	118
tert-butyl-benzene	119	91, 134	119
1,2,4-trimethylbenzene	105	120	105
sec-butyl-benzene	105	134	105
p-isopropyl-toluene	119	134, 91	119
1,3-dichlorobenzene	146	111, 148	146
1,4-dichlorobenzene	146	111, 148	146

Appendix II (continued)

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
n-butyl-benzene	91	92, 134	91
1,2-dichlorobenzene	146	111, 148	146
1,2-dibromo-3-chloropropane	75	115, 157	157
1,2,4-trichlorobenzene	180	182, 145	180
hexachlorobutadiene	225	223, 227	225
naphthalene	128	127	128
1,2,3-trichlorobenzene	180	182, 145	180

Appendix III
8260B Ultra Low Level Water Purge-and Trap Analysis ("8260UL")

1. Water samples are analyzed using a 20mL purge volume to achieve reporting limits approximately one hundred times lower than standard 8260B reporting limits.

2. The following lists 8260UL target analytes and performance data:

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
1,1,1,2-tetrachloroethane	630-20-6	70-130	20	0.05	0.25	0.5	0.05-10
1,1,1-trichloroethane	71-55-6	70-130	20	0.025	0.05	0.5	0.05-10
1,1,2,2-tetrachloroethane	79-34-5	70-130	20	0.05	0.25	0.5	0.05-10
1,1,2-trichloroethane	79-00-5	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloroethane	75-34-3	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloroethene	75-35-4	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloropropene	563-58-6	70-130	20	0.025	0.05	0.5	0.05-10
1,2,3-trichlorobenzene	87-61-6	70-130	20	0.05	0.1	0.5	0.05-10
1,2,3-trichloropropane	96-18-4	70-130	20	0.025	0.05	0.5	0.05-10
1,2,4-trichlorobenzene	120-82-1	70-130	20	0.025	0.05	0.5	0.05-10
1,2,4-trimethylbenzene	95-63-6	70-130	20	0.25	0.5	0.5	0.05-10
1,2-dibromo-3-chloropropane	96-12-8	70-130	20	0.25	0.5	0.5	0.05-10
1,2-dibromoethane	106-93-4	70-130	20	0.1	0.2	0.5	0.05-10
1,2-dichlorobenzene	95-50-1	70-130	20	0.025	0.05	0.5	0.05-10
1,2-dichloroethane	107-06-2	70-130	20	0.025	0.05	0.5	0.05-10
1,2-dichloropropane	78-87-5	70-130	20	0.025	0.05	0.5	0.05-10
1,3,5-trimethylbenzene	108-67-8	70-130	20	0.025	0.05	0.5	0.05-10
1,3-dichlorobenzene	541-73-1	70-130	20	0.025	0.05	0.5	0.05-10
1,3-dichloropropane	142-28-9	70-130	20	0.05	0.1	0.5	0.05-10
1,4-dichlorobenzene	106-46-7	70-130	20	0.025	0.05	0.5	0.05-10
2,2-dichloropropane	594-20-7	70-130	20	0.05	0.1	0.5	0.05-10
2-chlorotoluene	95-49-8	70-130	20	0.025	0.05	0.5	0.05-10
4-chlorotoluene	106-43-4	70-130	20	0.025	0.05	0.5	0.05-10
benzene	71-43-2	70-130	20	0.025	0.05	0.5	0.05-10
bromobenzene	108-86-1	70-130	20	0.025	0.05	0.5	0.05-10
bromochloromethane	74-97-5	70-130	20	0.05	0.1	0.5	0.05-10
bromodichloromethane	75-27-4	70-130	20	0.025	0.05	0.5	0.05-10
bromoform	75-25-2	70-130	20	0.1	0.25	0.5	0.05-10

8260UL target analytes (continued)

bromomethane	74-83-9	70-130	20	0.025	0.05	0.5	0.05-10
carbon disulfide	75-15-0	70-130	20	0.05	0.10	0.5	0.05-10
carbon tetrachloride	56-23-5	70-130	20	0.025	0.05	0.5	0.05-10
chlorobenzene	108-90-7	70-130	20	0.025	0.05	0.5	0.05-10
chloroethane	75-00-3	70-130	20	0.025	0.05	0.5	0.05-10
chloroform	67-66-3	70-130	20	0.05	0.1	0.5	0.05-10
chloromethane	74-87-3	70-130	20	0.05	0.1	0.5	0.05-10
cis-1,2-dichloroethene	156-59-2	70-130	20	0.025	0.05	0.5	0.05-10
cis-1,3-dichloropropene	10061-01-5	70-130	20	0.025	0.05	0.5	0.05-10
dibromochloromethane	124-48-1	70-130	20	0.025	0.05	0.5	0.05-10
dibromomethane	74-95-3	70-130	20	0.025	0.05	0.5	0.05-10
dichlorodifluoromethane	75-71-8	70-130	20	0.025	0.05	0.5	0.05-10
ethyl benzene	100-41-4	70-130	20	0.025	0.05	0.5	0.05-10
hexachlorobutadiene	87-68-3	70-130	20	0.025	0.05	0.5	0.05-10
isopropylbenzene	98-82-8	70-130	20	0.025	0.05	0.5	0.05-10
m+p-xylene **	108-38-3 + 106-42-3	70-130	20	0.025	0.05	1.0	0.05-10
methylene chloride	75-09-2	70-130	20	0.5	1	0.5	0.05-10
naphthalene	91-20-3	70-130	20	0.05	0.1	0.5	0.05-10
n-butylbenzene	104-51-8	70-130	20	0.025	0.05	0.5	0.05-10
o-xylene	95-47-6	70-130	20	0.025	0.05	0.5	0.05-10
p-isopropyl-toluene	99-87-6	70-130	20	0.025	0.05	0.5	0.05-10
propylbenzene	103-65-1	70-130	20	0.025	0.05	0.5	0.05-10
sec-butylbenzene	135-98-8	70-130	20	0.025	0.05	0.5	0.05-10
styrene	100-42-5	70-130	20	0.025	0.05	0.5	0.05-10
tert-butylbenzene	98-06-6	70-130	20	0.05	0.1	0.5	0.05-10
tetrachloroethene	127-18-4	70-130	20	0.025	0.05	0.5	0.05-10
Toluene	108-88-3	70-130	20	0.025	0.05	0.5	0.05-10
trans-1,2-dichloroethene	156-60-5	70-130	20	0.025	0.05	0.5	0.05-10
trans-1,3-dichloropropene	10061-02-6	70-130	20	0.025	0.05	0.5	0.05-10
trichloroethene	79-01-6	70-130	20	0.025	0.05	0.5	0.05-10
trichlorofluoromethane	75-69-4	70-130	20	0.025	0.05	0.5	0.05-10
vinyl chloride	75-01-4	70-130	20	0.1	0.25	0.5	0.05-10

3. 8260UL standards preparation:

3.1 Intermediate calibration standards: Primary calibration standards in Section 7.0 diluted to prepare intermediate standards as follows:

PRIMARY CALIBRATION STANDARD	PRIMARY CALIBRATION STANDARD CONCENTRATION	VOLUME	FINAL VOLUME (METHANOL)	INTERMEDIATE STANDARD FINAL CONCENTRATION
502.2 CAL200 Mega Mix	200 ug/mL	50 uL	10 mL	1 ug/mL
Custom Mix 3	250 ug/mL	40 uL	10 mL	1 ug/mL
Custom Mix 2	250 ug/mL	40 uL	10 mL	1 ug/mL
VOC Mix 6*	2000 ug/mL	5 uL	10 mL	1 ug/mL*
Freon 113*	1000 ug/mL	10 uL	10 mL	1 ug/mL*

* Combined to create one intermediate standard ("8260 Gases")

3.2 Intermediate internal and surrogate standards: 25 ug/mL; prepared by diluting 100 uL of the primary standards into 10 mL of methanol.

3.3 Working standards used for instrument calibration and calibration verification are prepared by diluting intermediate standards in deionized water as follows:

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD CONCENTRATION	WORKING STANDARD, ug/L						
	0.05	0.10	0.25	0.50	1.0**	5.0	10
VOA Mega mix (8260 Standard) 50 ug/mL	5uL	10uL	12.5uL	25uL	50uL	5uL	10uL
Custom Gases 50 ug/mL	5uL	10uL	12.5uL	25uL	50uL	5uL	10uL
Custom Mix 2 50 ug/mL	5uL	10uL	12.5uL	25uL	50uL	5uL	10uL
Custom Mix 3 50 ug/mL	5uL	10uL	12.5uL	25uL	50uL	5uL	10uL
Surrogate Standard 50 ug/mL	5uL	10uL	12.5uL	25uL	50uL	5uL	10uL
Final volume, DI water (mL)	100	100	50	50	50	50	50

** Denotes CCV

3.4 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards Section 7.0 in deionized water as follows:

Water Analyses

STOCK STANDARD CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL)	FINAL CONCENTRATION (ug/L)
VOC mixture 8260 QC (20 ug/mL)	2.5	100	0.5
VOC additionals Adds QC (20 ug/mL)	2.5	100	0.5
8260 extra additionals (20 ug/mL)	2.5	100	0.5

4. Configure instrument per the following:

GC/MS PURGE AND TRAP PARAMETERS	
purge time	12 minutes
dry purge time	1 minute
desorb preheat	245° C
desorb	1 minute at 250° C
bake	10 minutes at 260° C
LSC 2000, 3000 temps	valve 150° C, lines: 150° C
Archon 5100 temps	valve 95° C, lines 120° C
GAS CHROMATOGRAPH PARAMETERS	
carrier gas	helium, 99.999 %
injector temperature	220° C
oven temperature program	35° C (hold 4 minutes)
oven ramp	10° C/minute to 240° C (hold 2 minutes)
split ratio	25 : 1
MASS SPECTROMETER PARAMETERS	
beginning mass	45
ending mass	230
scan rate / sampling	8 scans/sec

5. Calibration performed per 8.0 using 8260UL standards.

6. Sample preparation and analysis:

6.1 Allow samples to warm to ambient.

6.2 Insert samples into auto-sampler tray.

6.3 Program auto-sampler to utilize a 20mL sample purge volume.

6.4 Record pH of samples after analysis.

6.5 Target analyte results above the upper calibration limit of the instrument require a dilution. The appropriate sample volume is diluted in a volumetric flask to obtain results within the calibrated range of the instrument.

7.0 Analytical procedures, QC requirements, and data review and reporting per MSV01 SOP, using 20mL purge volume.

NOTE: Analysis of acetone, 2-butanone, 4-metyl-2-pentanone, 2-hexanone, 2-chloroethyl vinyl ether, and vinyl acetate performed using the standard 8260B method procedures.

Appendix IV
Definitions

The following definitions may be used in the production of this SOP.

Batch:

A group of samples, which are processed together as a unit that undergo the same extraction, concentration, and cleanup.

Batch QC:

The quality control samples within a batch such as a blank, LCS, MS, MSD, and/or dup.

Blank:

An analyte free matrix that is processed with a batch to monitor contamination.

Cleanup:

A prescribed treatment of an extract to prepare the extract for analysis. This treatment may be physical or chemical in nature.

CAR:

Corrective Action Request is a program report that documents the actions taken for an out-of-control event, routine or non-routine.

CCV:

Continuing Calibration Verification is an analytical sample included in the analyses of a batch to verify that the process or measurement is in calibration.

COC:

Chain of Custody

DI Water:

Water that has had all the ions removed by an ion exchange process.

Digestate:

A sample medium treated by a digestion process to release or remove specific target analytes for subsequent analyses.

Digestion:

The process of releasing or removing target analytes from a sample so that they may be quantified in subsequent tests.

Dilution:

The process of reducing the concentration of target analytes by the addition of solvent.

Duplicate:

A split sample that is used to assess precision.

ECD:

Electron Capture Detector

EPA:

Environmental Protection Agency

EQ Blank:

Equipment blank is a sample of DI water poured into, over, or pumped into the sampling device that assesses the effectiveness of equipment decontamination.

Extraction:

The process, which releases or removes specific target analytes for subsequent analyses.

FID:

Flame Ionization Detector

Holding Time:

The maximum elapse of time that one can expect to store a sample without unacceptable changes in analyte concentrations. These times apply under prescribed storage conditions and deviations in storage conditions may affect the holding time. Extraction or digestion holding time refers to the time elapsed from sample collection to sample preparation.

Homogenized:

Samples that are in a uniform mixture and are particle sized so that the particles are uniformly small and evenly distributed.

HPLC:

High Performance Liquid Chromatography

ICV:

Internal Calibration Verification

IDL:

Instrument Detection Limit, the measure of instrument sensitivity.

LCS:

Laboratory Control Sample, a known matrix (DI water, sand, etc.) that is spiked with compound(s) representative of target analytes. This is used to document and assess the extraction or digestion.

Leachate:

A liquid that has percolated through waste, soil, rock, or other material and has mobilized chemical species in the process.

Leaching:

The separation or dissolving out of soluble constituents from a solid material or matrix by the natural action of percolating water or chemicals.

Limit of detection (LOD):

Laboratory verified MDL per SOP 45

Limit of quantitation (LOQ):

Laboratory supported quantitation limit as per SOP 45

LIMS:

Laboratory Information Management System

Matrix:

The component or substrate (water, soil, sludge, etc.) that contains the analyte of interest.

MS:

Matrix Spike is an aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the bias of a method in a given sample matrix.

MSD:

Matrix Spike Duplicate is the same as the matrix spike and it documents the precision of a method in a given sample matrix.

MDL:

Method Detection Limit is a measure of instrument sensitivity using solutions that have been subjected to sample preparation steps.

Precision:

A concept used to describe dispersion of measurements with respect to a measure of location or central tendency.

Preparation:

The action or process of making sample ready for analysis.

Reagent:

Chemical of known purity that is used in analytical methods.

Solvent:

The dissolving agent that usually makes up the greater proportion of a solution.

Soluble:

The dissolving of one substance into another substance.

SOP:

Standard Operation Procedure

Surrogate:

An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

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STANDARD OPERATING PROCEDURE FOR THE PERKIN ELMER ELAN 6100 INDUCTIVELY COUPLED PLASMA/MASS SPECTROMETER (SW-846 6020 / EPA Method 200.8) SOP ME700

Revision 6

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The following persons have read and understand this SOP and are using the latest version of the test method referenced on the Title Page:

Signature	Date

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1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples according to U.S. EPA Methods 6020,6020A and 200.8 for the elements listed as analytes in Table I using the ELAN 6100 ICP-MS.

1.2 This method is applicable to the following sample matrices: ground waters, surface waters, industrial wastes, sludges, and soil samples.

1.3 Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and for other solid wastes for which total (acid leachable) elements are required. Sample preparation methods may be found in EPA Publication SW-846 as Methods 3015 and 3051 and are detailed in SOP ME407 and ME406.

1.4 Routine operation and maintenance procedures for the ELAN 6100 ICP-MS may be found in the ELAN Hardware Manual provided by the instrument manufacturer.

1.5 Detailed instructions on operating of the ELAN 6100 ICP-MS operating software may be found in the ELAN 6100 Software Manual.

1.6 Aqueous sample digestates, leachates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a R.F. plasma. There the sample is ionized. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector.

2.0 SAFETY PRECAUTIONS

2.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices must be followed at all times.

2.2 Safety glasses & gloves must be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

2.3 Liquid argon represents a potential cryogenic hazard and safe handling procedures must be used when handling liquid argon tanks at all times.

2.4 The ELAN 6100 is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the ELAN if any safety interlock is known to be disabled or malfunctioning.

2.5 Spilled samples, reagents, and water must be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills the acid must be neutralized with sodium bicarbonate solution before cleanup.

2.6 All additional company safety practices must be followed at all times.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Vol./Wt. Req.*	Collection Vol./Wt.	Preservative	Holding Time **
Total recoverable	40 mL	250 - 1000 mL	HNO3 to pH <2	6 months
Dissolved	40 mL	250 - 1000 mL	Filter on-site HNO3 to pH <2	6 months
Suspended	40 mL	250 - 1000 mL	Filter on-site	6 months
Total	40 mL	250 - 1000 mL	HNO3 to pH <2	6 months
Soil	0.5 g	200 g		6 months

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

4.0 METHOD PERFORMANCE

Table 1 ELAN 6100 IDLs, MDLs, and Linear Ranges

Analyte	Mass	IDL* (µg/L)	Linear Range ** (µg/L)	Water MDL (µg/L)	Water RL (µg/L)	Soil MDL (mg/Kg)	Soil RL mg/Kg
V	51	0.093	100	0.25	1.0	0.125	0.5
Cr	52	0.206	100	0.5	2.0	0.1	0.4
Mn	55	0.167	100	0.5	2.0	0.05	0.2
Co	59	.0.037	100	0.25	1.0	0.125	0.5
Ni	60	0.177	100	1.0	4.0	0.2	0.8
Cu	65	0.107	100	0.5	2.0	0.15	0.6
Zn	66	1.544	100	5.0	25.0	0.625	2.5
As	75	0.163	100	0.25	1.0	0.075	0.3
Se	82	0.453	100	0.5	1.0	0.1	0.2
Ag	107	0.007	100	0.25	1.0	0.05	0.2
Cd	114	0.018	100	0.125	0.5	0.025	0.1
Sb	123	0.111	100	0.25	1.0	0.05	0.1
Ba	135	0.04	100	0.5	3.0	0.075	0.3
Tl	203	0.005	100	0.05	0.2	0.01	0.02
Pb	208	0.044	100	0.25	1.0	0.1	0.2
U	238	0.001	100	0.25	1.0	0.1	0.4

* updated quarterly
** Values reported are 90% of the verified upper linear range

Instrument Detection Limits (IDLs)

4.1 IDLs are determined according to the procedure outlined in Section 8.2 of Method 6020. This is accomplished by calculating the average of the standard deviations of the three runs on three nonconsecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate sample (ie, with rinsing in between).

IDLs must be determined quarterly and kept with the instrument log book.

Method Detection Limits (MDLs)

4.2 The reference procedure for determination of Method Detection Limits is in Microbac SOP 45. Additional details on MDL studies may be found in SOP 45.

4.3 In order to determine the MDL in a matrix, the analytes must be spiked into the matrix of interest (e.g., a Method 3050 or Method 3051 sample preparation blank, ground water matrix, or other sample preparation procedure matrix) at a level that is three to five times the estimated MDL. The spiked matrix is then carried through the entire sample preparation procedure.

The MDL is calculated by multiplying the standard deviation obtained from a minimum of three analyses of the matrix spike by the one-sided 99% confidence level t-statistic. The table of t-statistics can be found in Chapter 1 of SW-846.

4.4 The most commonly used variation of this procedure and the one used in this SOP is to analyze the spiked matrix seven times and then multiply the standard deviation of the seven measurements by 3.14 (the t-statistic for 7 samples).

4.5 MDL check samples are analyzed quarterly to verify the MDLs listed in table 1.

4.6 MDLs must be redetermined whenever there is any change to the sample preparation procedure, or any significant change to the instrument (new detector or different sample introduction system used).

Linear calibration ranges

4.7 Calibrate the instrument, as described in Section 8.

4.8 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

4.9 The definition of the Linear Range is not specifically addressed in either SW-846 Method 6020 or in Chapter 1 (Quality Control) of the SW-846 Methods manual. In order to use an established definition of linear range, the definition from EPA Method 200.8 will be used. In this reference, the linear range is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard. The values reported in Table 1 are 90% of the verified upper linear range.

4.10 The upper Linear Dynamic Range must be redetermined when ever one of the following occurs. A new detector is installed. The detector (analog and/or pulse count)voltages are changed. A new PA tube is installed in the RF generator. A different sample introduction system (change in nebulizer or spray chamber type) is installed.

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 Isobaric Interferences occur when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98). Corrections for isobaric interferences must be made by measuring the intensity 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. Most commonly used corrections for isobaric interferences are already present as default interference equations in the ELAN NT software. 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.

5.2 Molecular interferences are caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.) Predictions about the type of molecular interferences must 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 Ar40Cl35 on As at mass 75 must be made by measuring the intensity of ArCl present at mass 77 (Ar40Cl37) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl37 to Cl35. A list of the corrections used is given in the listing of the isotopes monitored in the ELAN NT 6020 method in Table 2.

Table 2
Table of Isotopes Monitored and Equations Used

Analyte	Symb	Isotopes Monitored	Correction Equations
Aluminum	Al	27	
Antimony	Sb	121,123	Sb 123 = Sb 123 – 0.127189 * Te 125
Arsenic	As	75	As 75 = As 75 – 3.127 * [ArCl 77 – (0.815 * Se 82)]
Barium	Ba	135,137	
Beryllium	Be	9	
Cadmium	Cd	106,108,111,114	Cd 111 = Cd 111 – 1.073 * [MoO 108 – (0.712*Pd 106)] Cd 114 = Cd 114 – 0.026826 * Sn 118
Chromium	Cr	52,53	
Cobalt	Co	59	
Copper	Cu	63,65	
Lead	Pb	206,207,208	Pb 208 = Pb 208 + 1 * Pb 206 + 1 * Pb 207
Manganese	Mn	55	
Nickel	Ni	60,62	
Selenium	Se	77,82	Se 82 = Se 82 – 1.008696 * Kr 83
Silver	Ag	107,109	
Thallium	Tl	203,205	
Vanadium	V	51	V 51 = V 51 – 3.127 * [ClO 53 – (0.113 * Cr 52)]
Zinc	Zn	66,67,68	
Internal Standards			
Lithium	Li	6	
Scandium	Sc	45	
Yttrium	Y	89	
Rhodium	Rh	103	
Indium	In	115	
Terbium	Tb	159	
Bismuth	Bi	209	
Germanium	Ge	72	
(Information Only)			
Molybdenum	Mo	95,97,98	Mo 98 = Mo 98 – 0.110588 * Ru 101
Thorium	Th	232	
Uranium	U	238	
Calcium	Ca	44	
Magnesium	Mg	24	
Sodium	Na	23	
Potassium	K	39	
Iron	Fe	54	Fe 54 = Fe 54 – 0.028226 * Cr 52

6.0 EQUIPMENT AND SUPPLIES

6.1 Perkin-Elmer ELAN 6100 ICP-MS system.

6.1.1 Includes ELAN 6100 instrument, computer system, ELAN NT software, printer, and autosampler.

6.2 Peristaltic pump tubing:

6.2.1 Black/White - 0.125 mm I.D. (for drain)

6.2.2 Yellow/Orange - 0.51 mm I.D. (for internal standard introduction)

6.2.3 Black/Black – 0.32 mL/min (for rinse station)

6.3 Mixing Block

6.4 Calibrated mechanical pipettes:

6.4.1 10-100 µL

6.4.2 100 - 1000 µL

6.4.3 1000 - 5000 µL

6.5 Metal-free plastic pipette tips (for the pipettes specified in 6.4).

6.6 Metal-free15-mL plastic test tubes

6.7 Metal-free 50-mL plastic test tubes

6.8 Class A glass pipettes – for preparation of standard solutions.

7.0 STANDARDS AND REAGENTS

7.1 All reagents may contain impurities that may affect the integrity of the analytical results. Due to the high sensitivity of ICP-MS, high-purity reagents, water, and acids must be used whenever possible. All acids used for this method must be of ultra high-purity grade. Nitric acid is preferred for ICP-MS in order to minimize polyatomic interferences. It should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for poly-atomic ion interferences must be used. All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Nitric acid, Instra-analyzed reagent, J.T. Baker

7.3 Reagent water equivalent to ASTM Type I water (ASTM D 1193).

7.4 2% HNO_3 (in reagent water) – 20 mL HNO_3 in 1000 mL reagent grade water.

Internal Standard Stocks

7.5 ICP-MS Internal Standard - 100 mg/L Li, Sc, Y, In, Tb and Bi in 1% HNO_3, CPI International.

7.6 Germanium – 1000 mg/L in 2% HNO_3, CPI International.

7.7 Internal Standard Working Solutions - Prepare the working solution by pipetting 40 mL of nitric acid into approximately 1000 mL of reagent water in a 2000 mL volumetric flask. Pipette 1 mL of ICP-MS internal standard and 1 mL of germanium single-element standard into the flask. Dilute to 2000 mL with reagent water. The concentrations of the working internal standard solution can be found in
Table 7-1.

Tuning Solution

7.8 6020TS – 10 mg/L Co, In, Li and Tl in 2% HNO_3 – Inorganic Ventures

7.9 Rhodium, - 1000 mg/L in 5% Hcl – Inorganic Ventures, Inc.

7.10 Barium, - 1000 mg/L in 0.1% HNO_3 – Inorganic Ventures, Inc.

7.11 Cerium – 1000 mg/L in 5% HNO_3 – Inorganic Ventures, Inc.

7.12 Lead – 1000 mg/L in 0.35% HNO_3 – Inorganic Ventures, Inc.

7.13 Magnesium, 1000 mg/L in 0.1 HNO_3 – Inorganic Ventures, Inc.

7.14 Beryllium, 1000 mg/L in 2% HNO_3 – Inorganic Ventures, Inc.

7.15 Working Tuning Solution - Prepare the working tuning solution by pipetting 20 mL of nitric acid into approximately 500 mL of reagent water in a 1000 mL volumetric flask. Pipette 1 mL each of 6020TS and 0.01 mL each of single element rhodium, barium, cerium, lead, beryllium and magnesium into the flask. Dilute to 1000 mL with deionized water. The concentrations of the working tuning solution can be found in Table 7-2.

Calibration Solutions

7.16 Purchased Standard Mixes: CAL-MS1 – 10 mg/L Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Ag, Tl, V and Zn in 3.5% HNO_3 – Inorganic Ventures, Inc.
CAL- MS2 - 1000 mg/L Ca, Fe, Mg, K and Na in 2% HNO_3 – Inorganic Ventures, Inc.

7.17 1000 ug/mL Mo – Inorganic Ventures, Inc.

7.18 1000 ug/mL Ti – Inorganic Ventures, Inc.

7.19 1000 ug/mL Sn – Inorganic Ventures, Inc.

7.20 1000 ug/mL U – Inorganic Ventures, Inc.

7.21 Working Calibration Solution - Prepare the working solution (high standard) by pipetting 10 mL of nitric acid into approximately 250 mL deionized water in a 500 mL volumetric flask. Pipette 5 mL each of CAL-MS1 and CAL-MS2 into the flask. Add 0.1 mL each of the single element solutions Mo and Ti; and 0.05 mL of Sn and U. Dilute to 500 mL with deionized water. The concentration of the working solution can be found in Table 7-3.

7.22 Working Calibration Solutions - Prepare calibration standards using a 2% HNO_3 solution (7.4) and the working solution (high standard) (7.21).

7.23 Blank (for establishing internal standard intensities) and S1 (STD 1) – 2 % HNO_3 Solution. (7.4).

7.24 S2 (Std 2) – 1/200 high standard:2% HNO_3

7.25 S3 (Std 3) – 1/2 high standard: 2% HNO_3

7.26 S4 (Std 4) – high standard solution (7.20)

7.27 Working Continuing Calibration Verification Solution (CCV) - Prepare the working solution (CCV-QC STD 6) by pipetting 20 mL of nitric acid into approximately 500 mL deionized water in a 1000 mL volumetric flask. Pipette 5 mL each of CAL-MS1 and CAL-MS2 into the flask. Add 0.1 mL each of the single element solutions Mo and Ti; and 0.05 mL of Sn and U. Dilute to 1000 mL with deionized water. The concentration of the working solution can be found in Table 7-4

Quality Control Standard Solutions

7.28 Initial Calibration Verification (ICV – QCStd 1) This must originate from a different source than that of the calibration standard (i.e. second vendor).

7.29 Purchased Standard Mixes: Microbac QC-MS1 – 10 mg/L As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb, Se, Ag, Tl, V and Zn in 2% HNO_3 and tr HF – CPI International.

7.30 Microbac QC-MS2-1000 mg/L Ca, Fe, Mg, K and Na in 5% HNO_3 – CPI.

7.31 1000 ug/mL Mo – CPI

7.32 1000 ug/mL Ti – CPI

7.33 1000 ug/mL Sn – CPI

7.34 1000 ug/mL U – SCP Science

7.35 Working Initial Calibration Verification Solution (ICV) - Prepare working solution by pipetting 10 mL of HNO_3 into approximately 250 mL of deionized water into a 500 mL volumetric flask. Pipette 2.5 mL each of QC-MS1 and QC-MS2 into the flask. Add 0.05 mL of the single element solutions Mo and Ti and 0.025 mL of Sn and U. Dilute to 500 mL with reagent water. The concentrations can be found in Table 7-4.

7.36 Initial Calibration Blank (ICB – QCStd 2) and Continuing Calibration Blank (CCB – QCStd 7). – 2% HNO_3 solution (7.4).

Interference Check Solutions (ICSA and ICSAB)

7.37 6020ICS-9A – 21215 mg/L Cl^-, 2000 mg/L C, 3000 ppm Ca, 25000 mg/L Fe and Na, 1000 mg/L Al, Mg, P, K and S and 20 ppm Mo and Ti in 1% HNO_3 – Inorganic Ventures, Inc.

7.38 1000 ug/ML U – SCP Science

7.39 Microbac QC-MSI -10 mg/L As, Al, Ba, Be, Cd, Co, Cr, Cu, Pb, Mn, Ni, Sb, Se, Ag, Tl, V and Zn in 2% HNO_3 and trHF–CPI International.

7.40 Working ICSA Solution - Prepare working solution by pipetting 2 mL of HNO3 into approximately 50 mL of deionized water in a 100 mL volumetric flask. Pipette 1 mL 6020ICS-9A and dilute to 100 mL with deionized water. The concentrations can be found in Table 7-5.

7.41 Working ICSAB Solution - Prepare the working solution by pipetting 2 mL of HNO3 into approximately 50 mL of deionized water in a 100 mL volumetric flask. Pipette 1 mL of 6020ICS-9A, 1 mL of Microbac QC-MSI and 0.01 mL of 1000ppm U – SCP Science into the flask. Dilute to 100 mL with deionized water. The concentration can be found in Table 7-5.

7.42 Dual Det. Calibration Solution

Table 7-1
Internal Standard Solution

Analyte	Stock Concentration (mg/L)	Secondary Concentration (mg/L)
Li	100	0.05
Sc	100	0.05
Ge	1000	0.5
Y	100	0.05
In	100	0.05
Tb	100	0.05
Bi	100	0.05

Table 7-2
Tuning Solution

Analyte	Stock Concentration (mg/L)	Secondary Concentration (mg/L)
Ba	1000	0.01
Ce	1000	0.01
Co	10	0.01
In	10	0.01
Pb	1000	0.01
Li	10	0.01
Mg	1000	0.01
Rh	1000	0.01
Tl	10	0.01
Be	1000	0.01

Table 7-3
Calibration Solutions

Analyte	Stock Conc. (mg/L)	S1 (mg/L)	S2 (mg/L)	S3 (mg/L)	S4 (mg/L)
Al	10	0	0.0004	0.05	0.1
Sb	10	0	0.0004	0.05	0.1
As	10	0	0.0004	0.05	0.1
Ba	10	0	0.0004	0.05	0.1
Be	10	0	0.0004	0.05	0.1
Ca	1000	0	0.04	5	10
Cd	10	0	0.0004	0.05	0.1
Cr	10	0	0.0004	0.05	0.1
Co	10	0	0.0004	0.05	0.1
Cu	10	0	0.0004	0.05	0.1
Fe	1000	0	0.04	5	10
Pb	10	0	0.0004	0.05	0.1
Mg	1000	0	0.04	5	10
Mn	10	0	0.0004	0.05	0.1
Mo	1000	0	0.0008	0.1	0.2
Ni	10	0	0.0004	0.05	0.1
K	1000	0	0.04	5	10
Se	10	0	0.0004	0.05	0.1
Ag	10	0	0.0004	0.05	0.1
Na	1000	0	0.04	5	10
Ti	1000	0	0.0008	0.1	0.2
Sn	1000	0	0.0004	0.05	0.1
Tl	10	0	0.0004	0.05	0.1
U	1000	0	0.0004	0.05	0.1
V	10	0	0.0004	0.05	0.1
Zn	10	0	0.0004	0.05	0.1

Table 7-4
ICV/CCV Solutions

Analyte	Stock Concentration (mg/L)	ICV/CCV Concentration (mg/L)
Al	10	0.05
Sb	10	0.05
As	10	0.05
Ba	10	0.05
Be	10	0.05
Ca	1000	5
Cd	10	0.05
Cr	10	0.05
Co	10	0.05
Cu	10	0.05
Fe	1000	5
Pb	10	0.05
Mg	1000	5
Mn	10	0.05
Mo	1000	0.1
Ni	10	0.05
K	1000	5
Se	10	0.05
Ag	10	0.05
Na	1000	5
Ti	1000	0.1
Sn	1000	0.05
Tl	10	0.05
U	1000	0.05
V	10	0.05
Zn	10	0.05

Table 7-5
Interference Check Solutions

Analyte	Stock Concentration (mg/L)	ICSA Concentration (mg/L)	ICSAB Concentration (mg/L)
Al	1000	10	10
Sb	0	0	0.1
As	10	0	0.1
Ba	0	0	0.1
Be	0	0	0.1
Ca	3000	30	30
Cd	10	0	0.1
Cr	20	0	0.1
Co	20	0	0.1
Cu	20	0	0.1
Fe	2500	25	25
Pb	0	0	0.1
Mg	1000	10	10
Mn	20	0	0.1
Ni	20	0	0.1
K	1000	10	10
Se	10	0	0.1
Ag	5	0	0.1
Na	2500	25	25
Tl	0	0	0.1
U	1000	0	0.1
V	20	0	0.1
Zn	10	0	0.1
Cl^-*	21215	212.15	212.5
C*	2000	20	20
P*	1000	10	10
S*	1000	10	10

* Although these are non-metals, they are essential for verifying the absence of mass interferences.

8.0 CALIBRATION PROCEDURES

8.1 Initial Calibration - The instrument is calibrated before analysis of any samples with a blank and three calibration standards. The concentrations of the standards used must be entered into the calibration page of the analytical method in the ELAN software according to the values of the standards prepared in Section 7.22, 7.23, 7.24 and 7.25. A Weighted Linear (with calculated intercept) curve type must be selected for all analytes. A blank (7.22) will be run as a blank, before the analysis of any actual calibration standards. This blank is used to monitor the internal standard intensities throughout the analytical sequence. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

The low calibration standard must contain the elements of interest at concentrations at or below the reporting limit or a low level calibration check standard at or below the reporting limit must be analyzed after calibration and before sample analysis. See tables 13-1 and 13-4 for acceptance criteria and corrective action for the curve and low level calibration check standard.

8.2 Initial Calibration Verification – (ICV) Analysis – ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13-1 and 13-4 for acceptance criteria and corrective actions.

8.3 Continuing Calibration Verification (CCV) Analysis -. The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13-1 and 13-4 for acceptance criteria and corrective action.

8.4 Initial and Continuing Calibration Blank (CCB) Analysis -The solution used is the calibration blank (7.23). See Tables 13-1 and 13-4 for acceptance criteria and corrective action.

8.5 ICSA and ICSAB Analysis - Required at the beginning of analytical run or every 12 hours, whichever is more frequent. See Tables 13-1 and 13-4 for acceptance criteria and corrective action.

8.6 Internal Standards - Intensities must be monitored in all solutions. Intensities of internal standards in all subsequent analyses of CCV and CCB solutions must

be greater than 80% and less than 120% of the levels in the original calibration blank. Failure action is to terminate the analysis, correct the problem, re-calibrate, and reanalyze all affected samples. See Tables 13-1 and 13-4 for acceptance criteria and corrective actions for analytical and quality control samples.

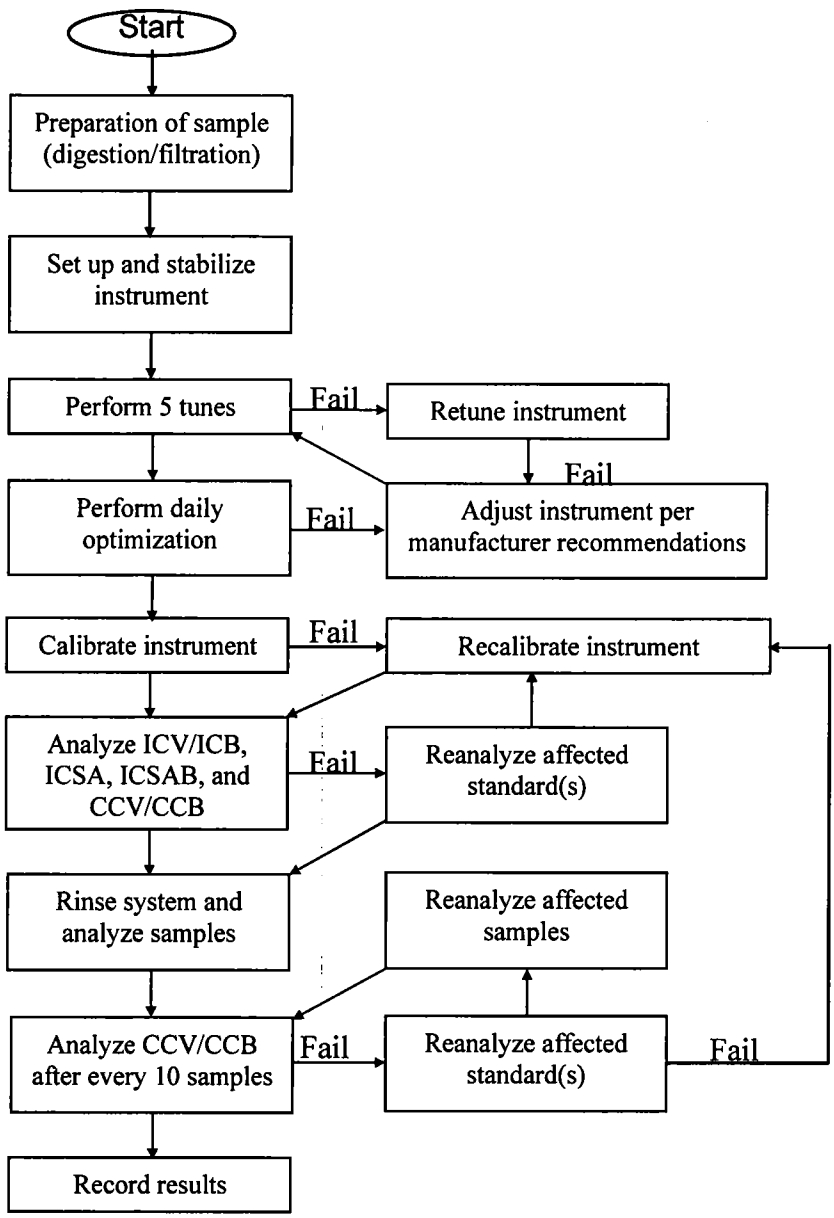
9.0 SAMPLE PREPARATION

Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

ME406 - Microwave Digestion of Sediments/Sludges/Soils/Oils

ME407 - Microwave Digestion - Aqueous

10.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 Initiate the plasma and allow a warm-up of at least 30 minutes. The tuning procedures must be carried out after warm-up

11.2 Perform five acceptable tunes at least once a day prior to analysis:

11.1.1 Place a tube containing tuning solution in cup position 8.

11.2.2 Open the Method window by clicking on Method on the ELAN tool bar. Select File/Open from the tool bar. When the Open window appears, choose Service/6020 Methods/EPA 6020 TUNING.mth.

11.2.3 In the Method window, select the Sampling tab and click on Probe. Designate cup 8 by clicking the right arrow on the horizontal bar until it reads "Tube Number: 8." Depress the Go to Tube # button. Click on OK.

11.2.4 Open Tuning window by clicking on Tuning on the ELAN Tool Bar. Depress the Tune Mass Spec button. The instrument will now perform the tuning procedure and the results will automatically print when the procedure is completed.

11.2.5 The measured mass of each isotope must be ± 0.1 amu of the exact mass and the RSD must not exceed 5%.

11.2.6 This procedure must be repeated until five successful tunes have been performed.

11.2.7 Save the last tune by selecting File/Save and saving under the file for the current month.

11.2.8 Close the Tuning window.

11.3 Perform a daily optimization before calibration to ensure the instrument is functioning at optimum levels.

11.3.1 Open the Method window. Select File/Open. When the Open window appears, choose Service/6020 Methods/6020 daily.mth.

11.3.2 Open the Sampling window on the ELAN tool bar. Select the Manual tab and the Details button. Edit the following settings:

A/S Location: 8
Sample Flush (s): 35
Read Delay (s):35
(rpm): -15

Click on OK

11.3.3 Depress the Analyze Sample button. The instrument will now perform the daily performance check. The results will print automatically upon completion of analysis.

11.3.4 The following criteria must be met for best results:

Mg intensity > 20,000 cps
Rh intensity > 150,000 cps
Pb intensity > 100,000 cps
$Ba^{+2}/Ba < 0.03$
CeO/Ce < 0.03
Bkgd < 30 cps @ mass 220

11.3.5 If these criteria are not met, further optimizations may be needed. See the instrument manuals for assistance.

11.4 Calibrate the instrument at least once a day followed by the analysis of the ICV, ICB, ICSA, ICSAB, CCV and CCB prior to the analysis of samples. See Tables 13-1 and 13-4 for acceptance criteria and corrective actions.

11.4.1 Prepare the four calibration solutions using the high standard and/or 2% HNO_3 (in polished water). The calibration standards are prepared as follows:

Blank: 2% HNO_3 water
S1: 2% HNO_3 water
S2: 1/200 high standard in 2% HNO_3 water
S3: 1/2 high standard in 2% HNO_3 water
S4: high standard solution
Load Blank, S1, S2 and S3, S4 into cups 1,2,3,4 and 5 respectively.

11.4.2 Pour the ICV/CCV solution into a tube and place it in cup 5.

11.4.3 Put the ICSA in cup 6 and the ICSAB in cup 7.

11.4.4 The ICB/CCB will read from S0 in cup 1.

11.4.5 Select the sample window from the ELAN tool bar. Click on batch tab.

11.4.5.1 Enter sample numbers (including digest workgroup numbers) in the Sample ID column.

11.4. 5.2Enter the autosampler location number for each sample in the A/S Loc. Column.

11.4.5.3 Make sure the appropriate command appears in the measurement action column for each sample. It must read "Run Sample" for samples that will not be auto-diluted by the instrument and "Run Diluted Sample" for samples that will be auto-diluted by the instrument. If the instrument is to be calibrated before the first sample is analyzed, then the measurement action cell for that sample only must read "Run Blank, Stds, and Sample" if the sample is not to be auto-diluted, and "Run Blank, Stds, and Dil. sample" if the sample is to be auto-diluted. This is edited by clicking the right mouse button on the cell that is to be edited and selecting the desired command from the list that appears using the left mouse button.

11.4.5.4Enter the factor which indicates the "fold" of dilution in the description column for each sample. For instance, if the sample is to be analyzed at a 1 to 5 dilution, then enter 5 in the description column for that sample. If the sample is not to analyzed at a dilution, then enter 1 in the description column. This must be entered regardless of whether the operator or the instrument will be performing the dilutions.

11.4.5.5 It is not necessary to enter the sample preparation factors. These will be applied during the data upload to LIMS procedure.

11.4.5.6Make sure "35" appears in the Sample Flush column, "-48" in the Sample Flush Speed, "35" in Read Delay, "15" in Delay & Analysis Speed, "200 in Wash and "-48" in the Wash Speed column for each sample to be analyzed. These cells can be edited directly.

11.4.5.7 Save the sample file.

11.4.5.8Print the run list by selecting the print command from the file menu and clicking on the print button in the ELAN file print window that appears.

11.4.6 Select the method button from the Elan tool bar. Select File/Open. When the open window appears, choose Service 6020 Methods/6020A.mth.

11.4.6.1 Select the sampling tab. Edit the Dil. Factor cell only if the auto-dilutor will be used. Again this factor should reflect the "fold" of dilution. The Dil to vol. Cell must read "5" and the 1st Dil. Pos. must read "71". If no dilutions will be performed or if the operator will be performing the dilutions, this section does not need to be edited.

11.4.6.2 Select the QC tab. Enter an "X" in the initial column next to the appropriate QC Std # for all the QC standards that must be analyzed after the calibration is finished (if the instrument is to be calibrated). A list of the names and the standards to which they correspond follows:

QC Std 1 - ICV
QC Std 2 - ICB
QC Std 3 - CRQL Check Solid
QC Std 4 - CRQL Check Water
QC Std 5 - ICSA
QC Std 6 - ICSAB
QC Std 7 - CCV
QC Std 8 - CCB

In the final column, enter an "X" beside the QC Std # that corresponds to the QC standards that must be analyzed after all the sample have been analyzed. In the before A/S Loc. Column, enter the number that corresponds to the autosampler position of the sample that will run after the standards. For instance, 12 samples are loaded in autosampler positions 11-22, and a CCV and CCB must be analyzed after the sample in A/S position 20 and at the end. An "X" must be entered next to QC Std 7 and QC Std 8 in the final column and "21" must be entered in the first before A/S Loc. Next to QC Std 7 and QC Std 8.

11.4.6.3 Click on the report tab located on the right edge of the window. Edit the cell report filename in the following manner. The file path must read "c:\elandata\ReportOutput\mmddyy.rep" where mmddyy is the current date. Edit only the current date portion of the file path. Do not edit anything else in this section.

11.4.6.4 Save the Method.

11.4.7 Prepare samples and load into autosampler positions indicated in sample file.

11.4.8 Click on the sample window from the ELAN tool bar. Choose the batch tab.

11.4.8.1 Select the samples to be analyzed by highlighting the row of the first number and, while holding down the left mouse button, moving down the row numbers until all the samples to be analyzed have been highlighted.

11.4.8.2 Aspirate the rinse for at least 10 minutes after daily optimization before beginning analysis to avoid carry-over and contamination.

11.4.8.3 Click on analyze batch button in the upper left corner of the window. The instrument should now begin analysis.

11.4.8.4 The sample results are an arithmetic mean (average) of three replicate readings per analyte. For any analyte with at result greater than the reporting detection limit, the % RSD between the replicate readings must be less than ten.

12.0 DETAILS OF CALCULATIONS

12.1 Each metal is analyzed within the calibration range. Refer to section 4 for upper limits. Dilutions must be performed if the upper limit is exceeded.

12.2 After the calibration is complete, the software performs a linear regression with a calculated intercept. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance. The results are calculated from the calibration curve.

$$COC = \frac{\Sigma xy - \frac{\Sigma x \Sigma y}{n}}{\sqrt{\left(\Sigma x^2 - \frac{(\Sigma x)^2}{n}\right)\left(\Sigma y^2 - \frac{(\Sigma y)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

12.3 Dilution factors and preparation factors are calculated into the final result, which is computed from the mean of three exposures, during the data upload procedure.

12.3.1 For Liquid Samples:

$$\text{mg/L metal in sample} = \text{mg/L in digestate} \times \frac{\text{Final Prepared Volume (ml)}}{\text{Initial Volume (ml)}} \times \frac{\text{Total Diluted Volume}}{\text{Sample aliquot}}$$

12.3.2 For Solid Samples:

$$\text{mg/kg metal in sample} = \text{mg/L in digestate} \times \frac{\text{Final Prepared volume (ml)}}{\text{Initial weight (g)}} \times \frac{\text{Total Diluted Volume}}{\text{Sample aliquot}}$$

12.4 LCS

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where: C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
%R = percent recovery

12.5 Spike Percent Recovery is calculated as follows:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

where: C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
%R = percent recovery

12.6 Relative Percent Difference (RPD) is calculated as follows:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

Where: C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.7 Percent Difference is calculated as follows:

$$\%D = \left[\frac{|C_1 - C_2|}{C_1}\right]100$$

Where: C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.8 See Figure 12.1 for a sample calculation summary.

13.0 QUALITY CONTROL (QC) REQUIREMENTS

Overview

13.1 Refer to section 8 for instrument calibration and instrument quality control samples. Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC Samples. The QC samples are prepared and digested indentically to the analytical samples. The following QC are digested and or analyzed with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Table 13.1 and 13.4.

Batch Quality Control

13.2 Method blank (Prep Blank (PB)) - An aliquot of deionized water that is digested with the sample batch and contains all reagents identical with the sample.

13.3 Laboratory Control Sample (LCS), Laboratory Control Sample Duplicate (LCS Dup) - A LCS or LCS/LSC Dup must be analyzed using the same sample preparations, analytical methods, and QA/QC procedures used for test samples. One LCS must be prepared and analyzed for each sample batch of 20 samples. Acceptance ranges are 80 – 120% for method 6020 and 85 – 115% for 200.8. The final concentration is outlined in Table 13.2. QC Acceptance ranges are outlined in Tables 13-3 and 13-5.

13.4 Duplicate sample analysis (200.8 only) - Analyze one duplicate sample in a batch of twenty samples or less. The formulas for calculation of the Relative Percent Difference (RPD) between the duplicate determinations is located in Section 12.6. A control limit of 20% RPD must not be exceeded for analyte values greater than 100 times the IDL. If the control limit is exceeded, the reason for the out of control situation must be corrected and any samples analyzed during the out of control condition reanalyzed.

13.5 Matrix spike and matrix spike duplicate – A sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spike with 0.5 mL of CAL-MS-1 for each 50 mL of sample. The final concentration spiked into the two spiked samples is outlined in Table 13.2 Batches that include samples for method 200.8 must include a spiked sample for every ten 200.8 samples.

Interference Tests – Post Digestion

13.6 Dilution Test - If the analyte concentration is within the linear range of the instrument and is a factor of at least 100 times the MDL, the analysis of a five-fold dilution of the sample must agree within 10% of the original determination. If not, an interference must be suspected. The dilution is prepared by adding one mL of sample to four mL of 2% HNO3 in dionized water.

One dilution test must be performed for each twenty samples or less of each matrix in a sample batch.

13.7 Post digestion spike - An analyte spike added to a portion of a prepared sample or its dilution must be recovered to within 75-125% of the known value of the spike or within laboratory derived acceptance criteria. The final concentration spiked into the two spiked samples is outlined in Table 13.2 The spike value should be based upon the indigenous level of the analyte in the sample. If the spike is not recovered within the acceptance limits, the sample must be diluted and reanalyzed to compensate for the matrix effect. The results of the dilution must agree within 10 % of the original determination. See 13.6. The use of the method of standard additions may be used to compensate for matrix effects.

Contingencies for Handling Out-of-Control or Unacceptable Data

13.8 All data is scrutinized by the analysts for method specific compliance as outlined in Section 11 and 13 of the individual SOPs. Check lists are utilized and accompany each data batch.

13.9 Out-of-control data may be addressed by one or more of the following approaches:

- Internal corrective actions (reanalysis)
- Internal documentation and communication (CAR)
- Discussion and qualification of data (report and narrative)
- Client notification
- Data rejection (R-flagging)

- Re-sampling and re-analysis (client decision)

13.10 When out-of-control situations are encountered, method specific corrective actions are followed. These corrective actions are outline in Tables 13-1 and 13-4. These corrective actions may include a variety of actions such as recalibration and rerunning all affected samples, and if these measures fail, may require contacting the client to inform them of the problem and to obtain their directions on re-running, re-extracting, or re-sampling the samples.

13.11 The out-of-control situations are documented on Analytical Corrective Action forms which are reviewed and signed by the Department Manager and the QA/QC Supervisor. The Client Service Manager is copied on all Corrective Action Forms, so that the Technical Service Representative may inform the client affected by the non-compliant data. These forms are kept on file and are available for review.

13.12 Depending on the decision of the client, the data may be qualified and used and a case narrative will be written to summarize the situation and advise on usability of data.

Table 13-1
Quality Control Criteria
Total Metals – ICP/MS Method 6020

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	Correlation coefficient must be ≥ 0.998	Investigate, reanalyze the aberrant standard or recalibrate
Initial Calibration Verification (ICV)	After calibration	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Low Level calibration check standard (at or below RL)	Minimum of once per calibration prior to sample analysis unless multipoint calibration with low std at or below RL is performed	All analyte(s) within ± 50% of expected value for SW-846 and AFCEE QAP 3.1. All analytes within ± 20 % of expected value for DOD Version 3 and AFCEE QAP 4.0	Correct problem then reanalyze
MS Tune	Daily 4 times prior to calibration	Measured mass within 0.1 amu of exact mass; RSD < 5%	Retune Instrument
Initial Calibration Blank	After ICV	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank	minimum every 10 samples	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reana If still outside limits, recalibrate, reanalyze all samples run after th last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RL or <MDL x 2	Stop analysis, investigate, reanalyze. If still > limit, redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
ICP interference check	Run at beginning of each run (12 hour maximum)	All non-spiked analytes <2 x MDL (unless they are a verified trace impurity from one of the spiked analytes) Spiked analytes ± 20% of expected value.	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Laboratory Control Sample (LCS) Laboratory Control Sample Duplicate (LCS Dup)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
Matrix spike/Matrix Spike Duplicate	One per batch (20 samples maximum per batch)	75 - 125% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (optional)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike
Internal Standards	Every sample	Intensity >30% <120% of that of initial calibration blank No upper limit for Method 6020 A. See Appendix I for OVAP requirements.	Stop analysis, investigate, dilute sample if interference is apparent or recalibrate and reanalyze affected samples.
Daily Performance Report (Manufacturer's recommendations)	Daily after MS Tune	Background <30 cps @ Mass 220 Rh sensitivity > 150000 cps Mg sensitivity > 20000 cps Pb sensitivity >100000 cps CeO/Ce = ≤ 0.03 Ba^{+2}/Ba = ≤ 0.03	Stop analysis, investigate, reanalyze. If still outside limit, examine and replace cones.

(1) Acceptance criteria are project specific. Consult QAPP.

Table 13-2
Spike Concentrations
Total Metals – ICP/MS
Method 6020

	LCS		MS/MSD		Post Spike	
Analyte	Water mg/L	Soil mg/Kg	Water mg/L	Soil mg/Kg	Water mg/L	Soil mg/Kg
Ag	0.125	10.0	0.125	10.0	0.125	10.0
Sb	0.125	10.0	0.125	10.0	0.125	10.0
As	0.125	10.0	0.125	10.0	0.125	10.0
Pb	0.125	10.0	0.125	10.0	0.125	10.0
Se	0.125	10.0	0.125	10.0	0.125	10.0
Tl	0.125	10.0	0.125	10.0	0.125	10.0
Ba	0.125	10.0	0.125	10.0	0.125	10.0
Cd	0.125	10.0	0.125	10.0	0.125	10.0
Cr	0.125	10.0	0.125	10.0	0.125	10.0
Co	0.125	10.0	0.125	10.0	0.125	10.0
Cu	0.125	10.0	0.125	10.0	0.125	10.0
Mn	0.125	10.0	0.125	10.0	0.125	10.0
Ni	0.125	10.0	0.125	10.0	0.125	10.0
U	0.125	10.0	0.125	10.0	0.125	10.0
V	0.125	10.0	0.125	10.0	0.125	10.0
Zn	0.125	10.0	0.125	10.0	0.125	10.0

TABLE 13-3
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR INORGANIC METALS ANALYSES OF GROUNDWATER

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% Recovery)	PRECISION (% RPD)	MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Antimony (Sb) - ICP	7440-36-0	6020	80-120	20	0.5	1.0
Arsenic (As) - ICP	7440-38-2	6020	80-120	20	0.5	1.0
Lead (Pb) - ICP	7439-92-1	6020	80-120	20	0.5	1.0
Selenium (Se) - ICP	7782-49-2	6020	80-120	20	0.5	1.0
Thallium (Tl) - ICP	7440-28-0	6020	80-120	20	0.1	0.2
Silver (Ag) – ICP	7440-22-4	6020	80-120	20	0.05	1.0
Barium	7440-39-3	6020	80-120	20	0.5	3.0
Cadmium	7440-43-9	6020	80-120	20	0.125	0.5
Chromium	7440-47-3	6020	80-120	20	0.5	2.0
Cobalt	7440-48-4	6020	80-120	20	0.25	1.0
Copper	7440.50-8	6020	80-120	20	0.5	2.0
Manganese	7439-96-5	6020	80-120	20	0.5	2.0
Nickel	7440-02-0	6020	80-120	20	1.0	4.0
Uranium	7440-61-1	6020	80-120	20	0.25	1.0
Vanadium	7440-62-2	6020	80-120	20	0.25	1.0
Zinc	7440-66-6	6020	80-120	20	5.0	25.0

TABLE 13-3 (continued)
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR INORGANIC METALS ANALYSES OF SOLID WASTE

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% Recovery)	PRECISION (% RPD)	MDL SOIL (ug/Kg)	REPORTING LIMITS SOIL (ug/Kg)
Antimony (Sb) - ICP	7440-36-0	6020	80-120	20	50	100
Arsenic (As) - ICP Trace	7440-38-2	6020	80-120	20	75	300
Lead (Pb) - ICP Trace	7439-92-1	6020	80-120	20	100	200
Selenium (Se) - ICP Trace	7782-49-2	6020	80-120	20	100	200
Thallium (Tl) - ICP	7440-28-0	6020	80-120	20	10	20
Silver (Ag) – ICP	7440-22-4	6020	80-120	20	50	200
Barium	7440-39-3	6020	80-120	20	75	300
Cadmium	7440-43-9	6020	80-120	20	25	100
Chromium	7440-47-3	6020	80-120	20	100	400
Cobalt	7440-48-4	6020	80-120	20	125	500
Copper	7440-50-8	6020	80-120	20	150	600
Manganese	7439-96-5	6020	80-120	20	50	200
Nickel	7440-02-0	6020	80-120	20	200	800
Uranium	7440-61-1	6020	80-120	20	100	400
Vandium	7440-62-2	6020	80-120	20	125	500
Zinc	7440-66-6	6020	80-120	20	625	2500

Table 13-4
Quality Control Criteria
Total Metals – ICP/MS Method 200.8

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	Correlation coefficient must be ≥ 0.998	Investigate, reanalyze the aberrant standard or recalibrate
Initial Calibration Verification (ICV)	After calibration	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	85 - 115%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Low Level calibration check standard (at or below RL)	Minimum of once per calibration prior to sample analysis unless multipoint calibration with low std at or below RL is performed	All analyte(s) with ± 50% of expected value for SW-846 / EPA Method 200.8 and AFCEE QAP 3.1. All analytes within ± 20 % of expected value for DOD Version 3 and AFCEE QAP 4.0	Correct problem then reanalyze
MS Tune	Daily, 5 times prior to calibration	Measured mass within 0.1 amu of exact mass; RSD < 5% Peak width < 0.75 amu at 5% peak height	Retune Instrument
Initial Calibration Blank	After ICV	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank	minimum every 10 samples	< RL < 1/2 RL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after t' last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RL or <MDL x 2	Stop analysis, investigate, reana. If still > limit, redigest batch (requireu for Ohio VAP projects) or qualify data and address in narrative
ICP interference check	Run at beginning of each run (12 hour maximum)	All non-spiked analytes < RL (unless they are a verified trace impurity from one of the spiked analytes) Spiked analytes ± 20% of expected value.	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Laboratory Control Sample (LCS),Laboratory Control Sample Duplicate (LCS Dup)	One per batch (20 samples maximum per batch)	Control Limits 85 - 115%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch (required by Ohio VAP projects) or qualify data and address in narrative
Matrix spike/Matrix Spike Duplicate	One per ten 200.8 samples	70 - 130% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike
Internal Standards	Every sample	60 - 125%	Stop analysis, investigate, dilute sample if interference is apparent or recalibrate and reanalyze affected samples.
Daily Performance Report (Manufacturer's recommendations)	Daily after MS Tune	Background <30 cps @ Mass 220 Rh sensitivity > 150000 cps Mg sensitivity > 20000 cps Pb sensitivity >100000 cps CeO/Ce = ≤ 0.03 Ba^{+2}/Ba = ≤ 0.03	Stop analysis, investigate, reanalyze. If still outside limit, examine and replace cones.
Quality Control Sample (QCS) (120 ppb)	Run Quarterly	90-110%	Stop analysis, investigate, reanalyze. If still outside limit, recalibrate.

(1) Acceptance criteria are project specific. Consult QAPP.

TABLE 13-5
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR INORGANIC METALS ANALYSES

PARAMETER	CAS #	EPA SW-846 METHOD	ACCURACY (% Recovery)	PRECISION (% RPD)	MDL WATER (ug/L)	REPORTING LIMITS WATER (ug/L)
Antimony (Sb) - ICP	7440-36-0	200.8	85-115	20	0.5	1.0
Arsenic (As) - ICP	7440-38-2	200.8	85-115	20	0.5	1.0
Lead (Pb) - ICP	7439-92-1	200.8	85-115	20	0.5	1.0
Selenium (Se) - ICP	7782-49-2	200.8	85-115	20	0.5	1.0
Thallium (Tl) - ICP	7440-28-0	200.8	85-115	20	0.1	0.2
Silver (Ag) – ICP	7440-22-4	200.8	85-115	20	0.05	1.0
Barium	7440-39-3	200.8	85-115	20	0.5	3.0
Cadmium	7440-43-9	200.8	85-115	20	0.125	0.5
Chromium	7440-47-3	200.8	85-115	20	0.5	2.0
Cobalt	7440-48-4	200.8	85-115	20	0.25	1.0
Copper	7440.50-8	200.8	85-115	20	0.5	2.0
Manganese	7439-96-5	200.8	85-115	20	0.5	2.0
Nickel	7440-02-0	200.8	85-115	20	1.0	4.0
Uranium	7440-61-1	200.8	85-115	20	0.25	1.0
Vanadium	7440-62-2	200.8	85-115	20	0.25	1.0
Zinc	7440-66-6	200.8	85-115	20	5.0	25.0

14.0 DATA REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client samples into Kobra. This is done via Microbac's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and reporting limits. Any elements that are not to be reported must be checked in the "Excl" box next to the element. This will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1 and 13.4. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating. The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the hardcopy raw data with QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and Kobra workgroup reports. The Data Review Checklist acts as a cover page and must be archived with the hardcopy data. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. The secondary reviewer also signs and dates the Data Review Checklist.

This review also consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness.

When all levels of review have been completed. The elements being reported on each sample are taken to a done status in Kobra.

14.2 See figure 14.1 for an example date review checklist.

15.0 PREVENTATIVE MAINTENANCE

The following sections describe some commonly occurring problems and proposed solutions.

15.1 Poor recovery on selected Quality Control Analytes: If poor recoveries are obtained on only particular analytes in a Quality Control Standard and it had been verified that the sample has been properly prepared, it may be possible that the problem could be related to a problem with the internal standard used. Look at the entire list of elements grouped with an internal standard. If the results for all elements is not satisfactory but the results for other elements not grouped with that internal standard are acceptable, there could be a problem with the internal standard used for that grouping.

15.1.1 Try using a different internal standard and reprocessing the data.

15.1.2 Look at the monitored intensities of the internal standards. If the internal standard used for the elements with unacceptable results is not within the allowable range or the percent recovery for this internal standard significantly different that the others, use a different internal standard and reprocess the data.

15.2 Poor relative standard deviation (precision) on standards and samples.

15.2.1 Poor RSDs can be caused by many things. First check that the interface cones are in good condition and the orifices of both cones are round and of the proper size.

15.2.2 Check that the nebulizer is operating properly by checking the aerosol with the plasma off and the spray chamber removed. Turn on the nebulizer gas and the peristaltic pump - must be a visible aerosol leaving the spray chamber. If there is not, clean or replace the nebulizer gem tips.

15.2.3 Check that the peristaltic pump tubing is in good condition and not worn. When the autosampler probe is removed and reinserted in the wash solution an air bubble will be visible in the tubing. Watch the progress of this bubble and adjust the tension on the pump tubing so the flow is smooth without any pulsations.

15.3 Sequence occurs improperly on startup of a batch analysis. The sample file has either not been saved or re-opened properly. Save the sample file and then reopen this same sample file. The full path name of the method must appear in the Method field of the sample list.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into 55 gallon waste drums in our explosion-proof waste building located outside of the MICROBAC laboratory facility. These drums are labeled and a manifest is created for each. They are picked up by Van Waters & Rogers Inc. on a regular basis for disposal at Von Roll America Inc. – a licensed disposal facility. Acid or base waste is neutralized and disposed in the municipal sewer system as per agreement with the City.

16.2.1 The waste streams are as follows:

Metals Laboratory - Hg waste, acid waste.

16.3 Laboratory polices and procedures for management of hazardous waste are found in SOP 33-Laboratory Waste management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job descriptions. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Inductively Coupled Plasma-Mass Spectrometry@, US EPA SW-846 Method 6020, Revision 0, September 1994, EPA Publication SW-846.

17.2 Inductively Couples Plasma-Mass Spectrometry @, US EPA SW-846 Method 6020A, Revision 1, January 1998, EPA Publication SW-846.

17.3 ELAN 6100 Hardware Manual, 1995, Perkin-Elmer Corporation.

17.4 ELAN 6100 ICP-MS Software Manual, 1995, Perkin-Elmer Corporation.

17.5 Methods for the Determination of Metals in Environmental Samples - Supplement 1@, EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942, Method 200.8.

Figure 12.1

Example 6020 Calculations
Perkin Elmer ELAN 6100

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and three standards.

2.0 Calculating the concentration (C) of an element in water using data from prep log, run log, and quantitation report (note:the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	Example:
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Final volume	100
Vi = Initial volume	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in (ug/L)	0.25

3.0 Calculating the concentration (C) of an element in soil using data from prep log, run log, and quantitation report (note: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	Example:
Cs = Concentration computed by the data system (ug/L)	0.1
Vf = Final volume	200
Vi = Initial volume	0.5
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in (ug/kg)	40

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where:	Example:
Cx = Concentration calculated as received (wet basis)	40
Px = Percent solids of sample (%-wt)	80
$Cdry$ = Concentration calculated as dry weight (ug/kg)	50

50 ug/kg = 0.050 mg/kg

Figure 14.1

Checklist ID: 36971

Microbac Laboratories Inc.

Data Checklist

Date: 20-MAR-2009
Analyst: JYH
Analyst: NA
Method: 6020
Instrument: ELAN
Curve Workgroup: 297905
Runlog ID: 27152
Analytical Workgroups: 297854,297897,297856,297750

Calibration/Linearity	X
ICV/CCV	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	X
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	X
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	
Case Narrative	420,435,437,419,438,427
Client Forms	X
Level X	
Level 3	427
Level 4	420,437,419,438
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	JYH
Secondary Reviewer	SLP

Comments

Primary Reviewer: J. Yue Hu

Secondary Reviewer: 24-MAR-2009 Esther L. Blakeney

Appendix I
Ohio Voluntary Action Program (OVAP) Requirements

Ohio Voluntary Action Program samples are run by Method 6020 instead of 6020A. Inductively Coupled Plasma-Mass Spectrometry@, US EPA SW-846 Method 6020, Revision 0, September 1994, EPA Publication SW-846, Second Update. Method 6020 may not be used to determine Selenium on Ohio-VAP samples.

Appendix II

Definitions

The following definitions may be used in the production of this SOP.

Batch:

A group of samples, which are processed together as a unit that undergo the same extraction, concentration, and cleanup.

Batch QC:

The quality control samples within a batch such as a blank, LCS, MS, MSD, and/or dup.

Blank:

An analyte free matrix that is processed with a batch to monitor contamination.

Cleanup:

A prescribed treatment of an extract to prepare the extract for analysis. This treatment may be physical or chemical in nature.

CAR:

Corrective Action Request is a program report that documents the actions taken for an out-of-control event, routine or non-routine.

CCV:

Continuing Calibration Verification is an analytical sample included in the analyses of a batch to verify that the process or measurement is in calibration.

COC:

Chain of Custody

DI Water:

Water that has had all the ions removed by an ion exchange process.

Digestate:

A sample medium treated by a digestion process to release or remove specific target analytes for subsequent analyses.

Digestion:

The process of releasing or removing target analytes from a sample so that they may be quantified in subsequent tests.

Dilution:

The process of reducing the concentration of target analytes by the addition of solvent.

Duplicate:

A split sample that is used to assess precision.

ECD:

Electron Capture Detector

EPA:

Environmental Protection Agency

EQ Blank:

Equipment blank is a sample of DI water poured into, over, or pumped into the sampling device that assesses the effectiveness of equipment decontamination.

Extraction:

The process, which releases or removes specific target analytes for subsequent analyses.

FID:

Flame Ionization Detector

Holding Time:

The maximum elapse of time that one can expect to store a sample without unacceptable changes in analyte concentrations. These times apply under prescribed storage conditions and deviations in storage conditions may affect the holding time. Extraction or digestion holding time refers to the time elapsed from sample collection to sample preparation.

Homogenized:

Samples that are in a uniform mixture and are particle sized so that the particles are uniformly small and evenly distributed.

HPLC:

High Performance Liquid Chromatography

ICV:

Internal Calibration Verification

IDL:

Instrument Detection Limit, the measure of instrument sensitivity.

LCS:

Laboratory Control Sample, a known matrix (DI water, sand, etc.) that is spiked with compound(s) representative of target analytes. This is used to document and assess the extraction or digestion.

Leachate:

A liquid that has percolated through waste, soil, rock, or other material and has mobilized chemical species in the process.

Leaching:

The separation or dissolving out of soluble constituents from a solid material or matrix by the natural action of percolating water or chemicals.

Limit of detection (LOD):

Laboratory verified MDL per SOP 45

Limit of quantitation (LOQ):

Laboratory supported quantitation limit as per SOP 45

LIMS:

Laboratory Information Management System

Matrix:

The component or substrate (water, soil, sludge, etc.) that contains the analyte of interest.

MS:

Matrix Spike is an aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the bias of a method in a given sample matrix.

MSD:

Matrix Spike Duplicate is the same as the matrix spike and it documents the precision of a method in a given sample matrix.

MDL:

Method Detection Limit is a measure of instrument sensitivity using solutions that have been subjected to sample preparation steps.

Precision:

A concept used to describe dispersion of measurements with respect to a measure of location or central tendency.

Preparation:

The action or process of making sample ready for analysis.

Reagent:

Chemical of known purity that is used in analytical methods.

Solvent:

The dissolving agent that usually makes up the greater proportion of a solution.

Soluble:

The dissolving of one substance into another substance.

SOP:

Standard Operation Procedure

Surrogate:

An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

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Document Control #: 264

STANDARD OPERATING PROCEDURE FOR THERMO JARRELL ASH IRIS ADVANTAGE INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY SW-846 Method 6010B/200.7 SOP ME600F

Revision 9

Issue/Implementation Date: 15 May 2009

Last Review Date: 15 September 2009

Microbac Laboratories, Inc.
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Maren M Beery — 09/03/2009
Maren M. Beery, Metals Supervisor — Date

David L Bumgarner — 09/03/2009
David L. Bumgarner, Technical Director/QAO — Date

David E. Vandenberg — 9/8/09
David E. Vandenberg, Managing Director — Date

Microbac

The following persons have read and understand this SOP and are using the latest version of the test method referenced on the Title Page:

Signature	Date
[illegible]	9-03-09
Sheri L. Rogers	9/3/09
[illegible]	9-3-09

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure covers the operation of a Thermo Jarrell-Ash IRIS Advantage Inductively Coupled Argon Plasma Spectrometer (IRIS) according to SW846 6010B/200.7 for the analysis of metals in digested soils, sludges, wastes, extracts and waters. Filtered waters preserved in acid may be analyzed directly by this method. Samples originating from the states of North and South Carolina will be analyzed by Method 6010C in lieu of Method 6010B.

1.2 For elements for which this method is applicable See Table 1.1. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Table 4.1 provides concentration ranges for aqueous and soil samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods (See Microbac SOPs, ME401, ME403, ME406, ME407).

1.4 The IRIS uses Echelle optics and a unique Charge Injection Device (CID) solid state detector to provide complete and continuous wavelength coverage over the typical analytical wavelength range. The Echelle optics first disperses and then focuses the emitted light onto the CID detector. The detector converts the light energy to an electrical charge, whose magnitude is proportional to the incident light intensity. The integrated charge is measured by the detector circuit and passed on to the host computer.

1.5 The host computer uses the Thermo-SPEC/CID software. This software is based in a Windows 98 environment. A basic understanding of windows operations enables the analyst to move throughout the software.

1.6 Background correction points are selected according to compared background scans of standards, blanks, and samples.

Table 1.1: Elements Analyzed, Element Symbols, Wavelengths, and CAS Numbers.

Element	Symbol	Wavelength (nm)	CAS Number
Aluminum	Al	308.2	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	455.4	7440-39-3
Beryllium	Be	313.0	7440-41-7
Boron	B	249.6	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	373.6	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	324.7	7440-50-8
Iron	Fe	271.4	7439-89-6
Iron	Fe	259.8	7439-89-6
Lead	Pb	220.3	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	277.9	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Potassium	K	766.4	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	251.6	7440-21-3
Silver	Ag	328.0	7440-22-4
Sodium	Na	589.5	7440-23-5
Strontium	Sr	215.2	7440-24-6
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	334.9	7440-32-6
Vanadium	V	310.2	7440-62-2
Zinc	Zn	213.8	7440-66-6

Note: Iron 271.4 nm is used for soil analysis and iron 259.8 nm is used for water analysis.

2.0 SAFETY PRECAUTIONS

2.1 The safety practices summarized in this section are provided to help the user to operate the instrument safely. Read these safety practices thoroughly before attempting to operate the instrument; observe them at all times.

2.2 **CAUTION:** The instrument contains electrical circuits, devices, and components operating at dangerous voltages. Contact with these circuits, devices and components can cause death, serious injury, or painful electric shock. Never open the main covers. These must be opened only by trained, qualified, or approved service engineers.

Application of the wrong supply voltage, or connection of the instrument to an incorrectly wired supply outlet, or lack of proper electrical grounding can create a fire hazard, a potentially serious shock hazard, and could seriously damage the instrument and any attached ancillary equipment.

- **ALWAYS** switch the exhaust fan on BEFORE operating the ICP.
- Regularly check the system by smoke test to ensure that the exhaust system is functioning correctly.
- **NEVER** place your head over the Torch Compartment while it is operating. This could cause inhalation of hazardous or toxic fumes, or your skin and eyes could be injured by corrosive vapors or fumes.

2.3 The IRIS is equipped with safety interlocks to protect the analyst and instrument from harm if something out of the ordinary happens. The system checks water flow, argon pressure, plasma compartment door interlocks, and plasma stability. These interlocks are constantly monitored and displayed on the screen. If any interlock is interrupted, the plasma is automatically shut down.

NEVER attempt to defeat any interlocks.

2.3.1 The following interlocks must be satisfied in order to ignite the plasma.

- The face plate on the radial plasma compartment must have the holding screws in place.
- The argon pressures for the torch must be correct.
- The cooling water must be flowing to the RF coil.

2.4 **WARNING**: Hazardous ultraviolet radiation is emitted by the plasma. This radiation can cause permanent damage to the human eye. However, the face plate contains a UV filter which allows for safe observation of the plasma.

2.5 **WARNING:** Due to the high voltages and temperatures, caution must be used in maintenance and troubleshooting. After extinguishing the plasma, let the torch cool for 10 minutes before handling, not doing so may cause injuries due to burns.

2.6 **CAUTION:** NEVER shut off the camera purge gas for extended periods of time while the CID detector refrigeration system is on. Permanent degradation of the UV performance of the detector may occur. If the instrument is to be completely shut off, refer to Section 3 of the IRIS User Guide for shut off and start up procedures.

2.7 **WARNING:** The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this view point, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. Use gloves, safety glasses, lab coats and/or other appropriate safety precautions when handling samples and reagents.

3.0 SAMPLE PRESERVATION AND STORAGE

Table 3.1: Digestion Amounts, Collection Amounts, Preservatives, & Holding Times for Analytical Parameters.

Measurement	Digestion Amount Required*	Collection Amount Required	Preservative	Holding Time **
Total Recoverable	50 mL	250 - 1000 mL	HNO_3 to pH < 2	6 mos.
Dissolved	50 mL	250 - 1000 mL	Filter on-site HNO_3 to pH < 2	6 mos.
Suspended	50 mL	250 - 1000 mL	Filter on-site	6 mos.
Total	50 mL	250 - 1000 mL	HNO_3 to pH < 2	6 mos.
Soil	1 g	200 g	4 °C	6 mos.

* If insufficient sample amount is received, a smaller amount of sample will be used and the reagents ration will be adjusted accordingly (except for soil matrices)
** Storage time allowed between sample collection and analysis when properly preserved and stored.

4.0 METHOD PERFORMANCE

4.1 See Tables 4.1 and 4.2 for linear ranges, observed method detection limits, reporting limits and precision and accuracy data. Linear range studies are performed quarterly to confirm the upper linear range. The values reported in Table 4-1 are 90% of the verified upper linear range. MDL check samples are analyzed quarterly to verify the observed MDL limits. Statistical IDL Studies are performed quarterly. The calculated IDL must be less than or equal to the relevant MDL. Precision and accuracy data are verified quarterly by statistically generated data from historical LCS's. An initial demonstration of capability was performed in accordance with Microbac SOP 45.

Table 4.1: TJA IRIS ICP-OES Wavelengths, Upper Linear Ranges, MDL's, and RL's.

Element	Wavelength (nm)	Upper Linear Range (ppm)	Water Matrix MDL (mg/L)	Water Matrix RL (mg/L)	Soil Matrix MDL (mg/Kg)	Soil Matrix RL (mg/Kg)
Aluminum	308.2	630	0.05	0.1	10	20
Antimony	206.8	45	0.05	0.2	0.5	1
Arsenic	189.0	90	0.01	0.1	0.5	1
Barium	455.4	18	0.0025	0.01	0.1	0.5
Beryllium	313.0	3.6	0.0005	0.01	0.012	0.025
Boron	249.6	72	0.05	0.1	10	25
Cadmium	228.8	9	0.0025	0.01	0.05	0.1
Calcium	373.6	450	0.1	0.2	5	10
Chromium	267.7	18	0.0025	0.02	0.12	0.25
Cobalt	228.6	90	0.0025	0.02	0.12	0.25
Copper	324.7	45	0.005	0.02	0.5	1
Iron	271.4	720	NA	NA	1	3
Iron	259.8	90	0.025	0.1	NA	NA
Lead	220.3	180	0.01	0.1	0.5	1
Lithium	670.7	9	0.05	0.1	NA	NA
Magnesium	277.9	900	0.25	0.5	12	25
Manganese	257.6	16.2	0.001	0.01	0.1	0.5
Molybdenum	202.0	27	0.005	0.1	1.5	5
Nickel	231.6	72	0.005	0.04	0.5	2
Potassium	766.4	180	0.25	1	25	50
Selenium	196.0	18	0.05	0.1	0.5	1
Silicon	251.6	45	0.25	1	NA	NA
Silver	328.0	3.6	0.005	0.01	0.25	0.5
Sodium	589.5	135	0.25	0.5	5	25
Strontium	215.2	27	0.005	0.01	0.25	0.5
Thallium	190.8	18	0.1	1	1	2
Tin	189.9	27	0.05	0.5	5	25
Titanium	334.9	22.5	0.005	0.03	0.5	2
Vanadium	310.2	90	0.005	0.01	0.25	0.5
Zinc	213.8	18	0.005	0.02	0.5	1

Note: Iron 271.4 nm is used for soil analysis and iron 259.8 nm is used for water analysis.

Table 4.2: Precision & Accuracy for Inorganic Metals Analysis of Water and Soil Matrices.

Element	EPA SW-846 Method	Accuracy Water Matrix (% Rec.)	Precision Water Matrix (% RPD)	Accuracy Soil Matrix (% Rec.)	Precision Soil Matrix (% RPD)
Aluminum	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Antimony	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Arsenic	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Barium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Beryllium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Boron	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Cadmium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Calcium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Chromium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Cobalt	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Copper	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Iron	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Lead	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Lithium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Magnesium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Manganese	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Molybdenum	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Nickel	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Potassium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Selenium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Silicon	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Silver	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Sodium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Strontium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Thallium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Tin	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Titanium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Vanadium	6010B	85 - 115	0 - 20	80 - 120	0 - 20
Zinc	6010B	85 - 115	0 - 20	80 - 120	0 - 20

5.0 INTERFERENCES & CORRECTIVE MEASURES

5.1 IEC's (Interelement Corrections) are minimized by the use of the Echelle grating. However this grating does not eliminate all interferences due to spectral overlap. Table 5-1 lists the approximate IEC's necessary in mg/L of analyte per unit (mg/L) of interferant. These IEC's are subject to change with modifications made to operating conditions, such as changes in coolant flow, power, nebulizer or even new torches.

5.2 The use of a peristaltic pump reduces physical interferences. However, samples with high dissolved solids, high acid concentration or high viscosity may need to be diluted.

Table 5.1: Interelement Correction Factors for the TJA IRIS Advantage ICP-OES.

	Interferent						
Element	**Al**	**As**	**Ba**	**Be**	**Ca**	**Co**	**Cr**
Aluminum	--	--	--	--	--	-0.007	--
Antimony	-0.00008	-0.000185	--	--	--	--	0.019
Arsenic	0.000013	--	--	--	--	--	0.00028
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	--	--	--	--
Boron	--	--	--	--	--	--	--
Cadmium	--	0.006	-0.0003	--	--	--	--
Calcium	--	--	--	--	--	--	--
Chromium	--	--	--	--	--	--	--
Cobalt	--	--	--	--	--	--	0.000045
Copper	--	--	--	--	--	--	-0.00015
Iron	--	--	--	--	--	0.101	--
Lead	0.000387	--	--	--	--	-0.0001	--
Lithium	--	--	--	--	-0.00005	--	-0.00022
Magnesium	--	-0.0936	--	--	--	--	--
Manganese	--	--	--	--	--	--	-0.053
Molybdenum	--	--	--	--	--	--	--
Nickel	--	--	--	0.0083	--	0.0004	--
Potassium	--	--	-0.03	--	--	--	--
Selenium	0.000066	--	--	--	--	0.006	--
Silicon	--	--	--	--	--	--	--
Silver	--	--	--	--	--	--	0.00052
Sodium	--	--	--	--	--	--	--
Strontium	--	--	--	--	--	0.00006	--
Thallium	--	--	--	--	--	0.004	0.00019
Tin	--	--	--	--	--	--	--
Titanium	--	--	--	--	-0.00003	--	0.0003
Vanadium	-0.000018	--	--	--	--	--	-0.00026
Zinc	--	--	--	--	--	0.0002	--

Table 5.1: Interelement Correction Factors for the TJA IRIS Advantage ICP-OES. (continued)

	Interferent						
Element	**Cu**	**Fe**	**Mg**	**Li**	**Mn**	**Mo**	**Ni**
Aluminum	--	--	--	--	0.0018	0.033	--
Antimony	--	0.00003	--	--	--	-0.004	-0.001
Arsenic	--	-0.00007	--	--	--	0.00108	--
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	--	--	-0.00005	--
Boron	--	--	--	--	--	--	--
Cadmium	--	-0.000024	--	--	--	--	-0.00017
Calcium	--	-0.01	--	--	0.0016	--	0.00065
Chromium	--	-0.00001	--	--	0.00051	--	--
Cobalt	--	0.000013	--	--	--	--	--
Copper	--	0.00005	--	--	--	--	--
Iron	--	--	0.00004	--	-0.0005	-0.0075	--
Lead	--	0.00009	--	--	--	-0.003	0.000489
Lithium	--	--	--	--	--	--	--
Magnesium	--	--	--	--	0.00132	0.048	--
Manganese	--	-0.00003	--	--	--	--	--
Molybdenum	--	--	--	--	--	--	--
Nickel	--	-0.000018	--	--	--	--	--
Potassium	--	--	--	--	--	--	--
Selenium	--	-0.00033	--	--	0.0004	0.001	--
Silicon	--	--	--	--	--	0.008	--
Silver	--	0.000012	--	--	0.00008	-0.00029	--
Sodium	--	--	--	--	--	--	--
Strontium	--	-0.000586	--	--	--	0.000252	--
Thallium	--	--	--	--	0.0018	-0.0045	--
Tin	--	--	--	--	--	--	--
Titanium	--	--	--	-0.0025	--	--	--
Vanadium	--	-0.00001	-0.000004	--	--	--	--
Zinc	0.00025	0.00005	0.00007	--	0.000279	--	0.0062

Table 5.1: Interelement Correction Factors for the TJA IRIS Advantage ICP-OES. (continued)

	Interferent				
Element	**Sb**	**Sn**	**Ti**	**V**	**Zn**
Aluminum	--	--	-0.002	-0.055	--
Antimony	-0.00042	--	0.00062	0.00007	--
Arsenic	--	--	--	--	--
Barium	--	--	--	--	--
Beryllium	--	--	--	0.00074	--
Boron	--	--	--	--	--
Cadmium	--	--	--	0.000036	--
Calcium	--	--	--	--	--
Chromium	--	--	--	0.0001	--
Cobalt	--	-0.003	0.0013	--	-0.0028
Copper	--	--	-0.00095	-0.00314	--
Iron	--	--	--	-0.0058	--
Lead	--	--	--	--	--
Lithium	--	--	--	--	--
Magnesium	--	--	--	-0.0106	--
Manganese	--	--	--	-0.000075	--
Molybdenum	--	--	--	-0.000247	0.002
Nickel	--	--	--	--	--
Potassium	--	--	--	--	--
Selenium	--	--	--	--	0.02
Silicon	--	--	0.006647	--	--
Silver	--	--	0.0032	-0.023	--
Sodium	--	--	--	--	--
Strontium	--	--	--	--	0.00039
Thallium	-0.0005	--	-0.003	-0.029	--
Tin	--	--	--	--	--
Titanium	--	--	--	--	--
Vanadium	--	--	--	--	--
Zinc	--	--	--	--	--

6.0 EQUIPMENT AND SUPPLIES

6.1 Thermo Jarrell Ash IRIS Advantage ICP (floor model).

6.2 Argon gas supply (liquid).

6.3 IBM compatible computer with ThermoSpec Software.

6.4 IBM compatible printer.

6.5 Class A glassware – Volumetric flasks and volumetric pipettes.

6.6 Variable volume pipettes with ± 1.0% accuracy confirmed daily.

6.7 Disposable plastic test tubes.

6.8 Nalgene FEP bottles for intermediate solution storage.

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be regent grade or better. Redistilled acids may be used.

- Concentrated hydrochloric acid (HCl). Baker Instra Analyzed Grade or better.
- Concentrated nitric acid (HNO_3). Baker Instra Analyzed Grade or better.

7.2 Deionized water – ASTM Type II

7.3 Standard stock solutions may be purchased or prepared from ultra high purity grade chemicals or metals (99.99 to 99.999 % pure). All salts must be dried for one (1) hour at 105° C, unless otherwise specified.

7.3.1 All purchased stock standards and reagents are logged into the LIMS System and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.4 Mixed calibration stock standards are purchased from SPEX as an XKES-5,6 set and SCP Science as custom mixes 901-6K5-001 and 600-082-501. A single element silicon standard purchased from Inorganic Ventures is also used. From this the high standard and continuing calibration verification (CCV) are made for two different matrices.

7.4.1 For the high standard water matrix, dilute 5 mL from each bottle of stock into 500 mL of 5% HCl and 2% HNO3 in deionized water. For the soil matrix, dilute into 500 mL 5% HCl and 5% HNO3 in deionized water. From the high standard the following dilutions are made with the appropriate matrix matched acid water.

S4: 1:2 dilution of High Std
S3: 1:4 of High Std
S2: 1:100 of High Std
S1: 1:200 of High Std

Standard S0 is a matrix matched blank. The concentration of each element is in Table 7.4.1.

7.4.2 The CCV is prepared for 2 different matrices. For the water matrix dilute 5 ml from each bottle of calibration stock into 1000 ml of 5% HCl and 2% HNO3 in deionized water. For the soil matrix, dilute into 1000 ml 5% HCl and 5% HNO3 in deionized water. Concentrations for each element are listed in Table 7.5.1.

Table 7.4.1: Concentrations (mg/L) of Elements in Calibration and Stock Standards.

Element	S0	S1	S2	S3	S4	S5	Stock
Aluminum	0	0.1	0.2	5	10	20	2000
Antimony	0	0.012	0.024	0.6	1.2	2.4	240
Arsenic	0	NA	0.008	0.2	0.4	0.8	80
Barium	0	0.01	0.02	0.5	1	2	200
Beryllium	0	0.0005	0.001	0.025	0.05	0.1	10
Boron	0	NA	0.01	0.25	0.5	1	100
Cadmium	0	NA	0.001	0.025	0.05	0.1	10
Calcium	0	0.1	0.2	5	10	20	2000
Chromium	0	0.005	0.01	0.25	0.5	1	100
Cobalt	0	NA	0.004	0.1	0.2	0.4	40
Copper	0	0.005	0.01	0.25	0.5	1	100
Iron	0	0.04	0.08	2	4	8	800
Lead	0	NA	0.01	0.25	0.5	1	100
Lithium	0	0.01	0.02	0.5	1	2	200
Magnesium	0	0.1	0.2	5	10	20	2000
Manganese	0	0.005	0.01	0.25	0.5	1	100
Molybdenum	0	0.01	0.02	0.5	1	2	200
Nickel	0	0.005	0.01	0.25	0.5	1	100
Potassium	0	0.5	1	25	50	100	10000
Selenium	0	NA	0.008	0.2	0.4	0.8	80
Silicon	0	0.05	0.1	2.5	5	10	1000
Silver	0	0.004	0.008	0.2	0.4	0.8	80
Sodium	0	0.5	1	25	50	100	10000
Strontium	0	0.01	0.02	0.5	1	2	200
Thallium	0	NA	0.01	0.25	0.5	1	100
Tin	0	0.01	0.02	0.5	1	2	200
Titanium	0	0.01	0.02	0.5	1	2	200
Vanadium	0	0.01	0.02	0.5	1	2	200
Zinc	0	0.01	0.02	0.5	1	2	200

7.5 The Initial Calibration Verification (ICV) stock solution is purchased from Inorganic Ventures as KEM-CONC-1A,2,3A standards set. The ICV concentration is at the same level as the midpoint standard used in the calibration and is a separately prepared, quality control analyzed and certified source from that of the calibration standards.

7.5.1 The ICV is prepared for 2 different matrices. For the water matrix dilute 1 ml from each bottle of ICV stock into 200 ml of 5% HCl and 2% HNO3 in deionized water. For the soil matrix, dilute into 200 ml 5% HCl and 5% HNO3 in deionized water. Concentrations for each element are listed in Table 7.5.1.

Table 7.5.1: Concentrations (mg/L) of Elements in Initial and Continuing Calibration Verification Standards.

Element	ICV/CCV	Stock
Aluminum	10	2000
Antimony	1.2	240
Arsenic	0.4	80
Barium	1	200
Beryllium	0.05	10
Boron	0.5	100
Cadmium	0.05	10
Calcium	10	2000
Chromium	0.5	100
Cobalt	0.2	40
Copper	0.5	100
Iron	4	800
Lead	0.5	100
Lithium	1	200
Magnesium	10	2000
Manganese	0.5	100
Molybdenum	1	200
Nickel	0.5	100
Potassium	50	10000
Selenium	0.4	80
Silicon	5	1000
Silver	0.4	80
Sodium	50	10000
Strontium	1	200
Thallium	0.5	100
Tin	1	200
Titanium	1	200
Vanadium	1	200
Zinc	1	200

7.6 Three blanks are used in analysis.

7.6.1 A calibration blank of 5% HNO_3, 5% HCl for soil and 2% HNO_3, 5% HCl for water is used to establish the analytical curve.

7.6.2 A method blank is used to check for purity of acids and cleanliness of glassware used in digestion. The method blank must contain all the reagents in the same volume as used in the samples and must be carried through the complete digestion procedure.

7.6.3 An Initial Calibration Blank (ICB) is analyzed after the Initial Calibration Verification (ICV) and a Continuing Blank (CCB) is analyzed after all Continuing Calibration Verifications (CCV). These blanks are also matrix matched.

7.7 Interference Check Standards

7.7.1 An ICSA stock solution is purchased from Inorganic Ventures as CLPP-ICS-A. The ICSA is prepared for 2 different matrices. For the water matrix, dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 2% HNO3 in deionized water. For the soil matrix, dilute into 200 mL 5% HCl and 5% HNO3 in deionized water. The ICSA solution will contain only aluminum, calcium, iron and magnesium in high concentrations. See Table 7.7.1.

7.7.2 ICSAB stock solutions are purchased from Inorganic Ventures as CLPP-ICS-A, CLPP-ICS-B and KEM-ICS-B-1A. The ICSAB is made for 2 different matrices. For the water matrix, dilute 10 mL of CLPP-ICS-A, 1 mL of CLPP-ICS-B and 1 mL of KEM-ICS-B-1 into 200 mL 5% HCl and 2% HNO3 in deionized water. For the soil matrix, dilute into 200 mL 5% HCl and 5% HNO3 in deionized water. The ICSAB will have the same concentrations for aluminum, calcium, iron and magnesium as the ICSA with additional metals spiked at detectable levels. See Table 7.7.1.

Table 7.7.1: Concentrations (mg/L) of Elements in Interference Check Standards A & AB and Stock Standards.

Element	ICSA	ICSAB	Stock
Aluminum	250	250	5000
Antimony	--	0.5	100
Arsenic	--	0.25	50
Barium	--	0.25	50
Beryllium	--	0.25	50
Boron	--	--	0
Cadmium	--	0.5	100
Calcium	250	250	5000
Chromium	--	0.25	50
Cobalt	--	0.25	50
Copper	--	0.25	50
Iron	100	100	2000
Lead	--	0.5	100
Lithium	--	--	0
Magnesium	250	250	5000
Manganese	--	0.25	50
Molybdenum	--	--	0
Nickel	--	0.5	100
Potassium	--	5	1000
Selenium	--	0.25	50
Silicon	--	--	0
Silver	--	0.5	100
Sodium	--	5	1000
Strontium	--	--	0
Thallium	--	0.5	100
Tin	--	--	0
Titanium	--	--	0
Vanadium	--	0.25	50
Zinc	--	0.5	100

7.8 Laboratory Control Sample

7.8.1 Aqueous - Purchased Standard KEM-SPK-1G from Inorganic Ventures with the following concentrations:

7.8.2 Soil/Sediment – 1.0 gram of PTFE Boiling Stones (Teflon Chemware) spiked as in 13.3.2 prior to digestion.

Table 7.8.1: Concentrations of Elements in Purchased Standard KEM-SPK-1G.

Element	Concentration (mg/L)
Potassium Sodium	250
Aluminum Calcium Magnesium	50
Silicon	25
Iron	20
Antimony	6
Barium Lithium Molybdenum Strontium Titanium Vanadium Zinc	5
Boron Chromium Copper Manganese Nickel Lead Thallium	2.5
Arsenic Selenium Silver	2
Cobalt	1
Beryllium Cadmium	0.25

8.0 CALIBRATION PROCEDURES

8.1 Instrument start-up

8.1.1 Pump tubing on instrument should be pliable and have only slight discoloration. If tubing is worn, replace. If tubing appears to be in good condition or once new tubing is installed, then connect the pump winding.

8.1.2 Place sample sipper in its holder on the auto sampler.

8.1.3 Turn on computer. Instrument and argon remain on at all times except when the instrument is to be shut down for an extended period of time.

8.1.4 Open the Thermo SPEC/CID Software by double clicking on the icon.

8.1.5 On the tool bar, click on "Plasma Control Panel" icon. Then click on the "Ignite" icon. This will open the **Post Ignition Parameter** box. The settings will read approximately:

R.F Power	1350 watts
Auxiliary Flow	0.5 1/min
Nebulizer Flow	28.06 PSI
Pump Rate	120 RPM
Purge Time	90 Sec.

Click on the [OK] to start the Ignition sequence.

8.1.6 The ICP must be allowed to become thermally stable before beginning analysis. (This usually requires at least 1/2 an hour of operation prior to calibration.)

8.2 Set up of analysis:

8.2.1 After igniting plasma, click on the "Auto Sampler Setup" icon on the tool bar. This will open the **Automated Analysis** screen. Highlight the appropriate autosampler table. In this table the name of the sample file, analytical method and the analytical protocol are set up.

8.2.2 To set up the sample table right click on [Samples] in the **Automated Analysis** screen. Open an old table and clear out the list. Click on the "Add Samples" icon. This will open the screen where sample information is entered. Make sure that the "operator ID" box is filled in. Click on [Save] or hit the entry key after entering each sample. Click on [Done] after the last sample name has been entered. Save the sample table and close the screen.

8.2.3 After the sample table is done, edit the autosampler table. Click on [Edit]. Under "Sample File Count" enter the sample table name that is to be used. The number of samples entered will appear below the name. Enter the appropriate protocols. The method will be correct if the appropriate autosampler table was chosen. Save table and close screen.

8.2.4 If a protocol needs to be edited, click on [Advanced] with the right mouse button. Choose the protocol that needs editing. After changes have been made, save and close screen. The protocol is where the analytical run sequence is set up. See figure 8.2.4 for the correct run sequence. (See Section 13 for Quality Controls to be followed prior to analysis of the first samples.)

FIGURE 8.2.4

Calibration (See Section 8.3)

ICV
ICB
ICSA
ICSAB
CCV
CCB
10 or less samples
CCV
CCB
Continue with last 3 steps until end of run
Always end run with CCV/CCB

8.3 Calibration

8.3.1 Standards are prepared from Inorganic Ventures, SCP Science and SPEX stock. The dilutions of the calibration standards are listed in Section 7.4. The concentrations are listed in Table 7.4.1. The ICV, CCV, ICSA and ICSAB concentrations are listed in Tables 7.5.1 and 7.7.1.

8.3.2 The prepared calibration standards, ICV, CCV, ICB/CCB, ISCA and ICSAB are placed into 30 ml cups and are assigned positions by the computer program. To see what the positions are click on [Run] in the **Automated Analysis** screen. This will open the "Run Autosampler (name of table)" screen. Click on [Setup] and this will open the "Setup Autosampler screen. Click on [Samples] and this will show where all the cups should be positioned on the racks. This can be printed out by clicking [Print]. Click on [OK] when done.

8.3.3 The prepared calibration standards, ICV, CCV, ICB/CCB, ICSA & ICSAB are analyzed in three replicates with the reported results being the average of the three replicate readings.

8.3.4 To start calibration click on [Run] in the "Run Autosampler (name of table)" screen.

8.3.5 The instrument performs a weighted linear regression with a calculated intercept for all analyzed metals. When calibration is complete the correlation coefficients are printed out. They should be 0.995 or better for all elements needed for analysis. Calibration is automatically stored until the program is shut down or a new calibration is performed.

8.3.6 Refer to Tables 13-1 and 13-2 for passing criteria for ICV, CCV, ICSA and ICSAB.

8.4 All analytical data is stored automatically.

9.0 SAMPLE PREPARATIONS

Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

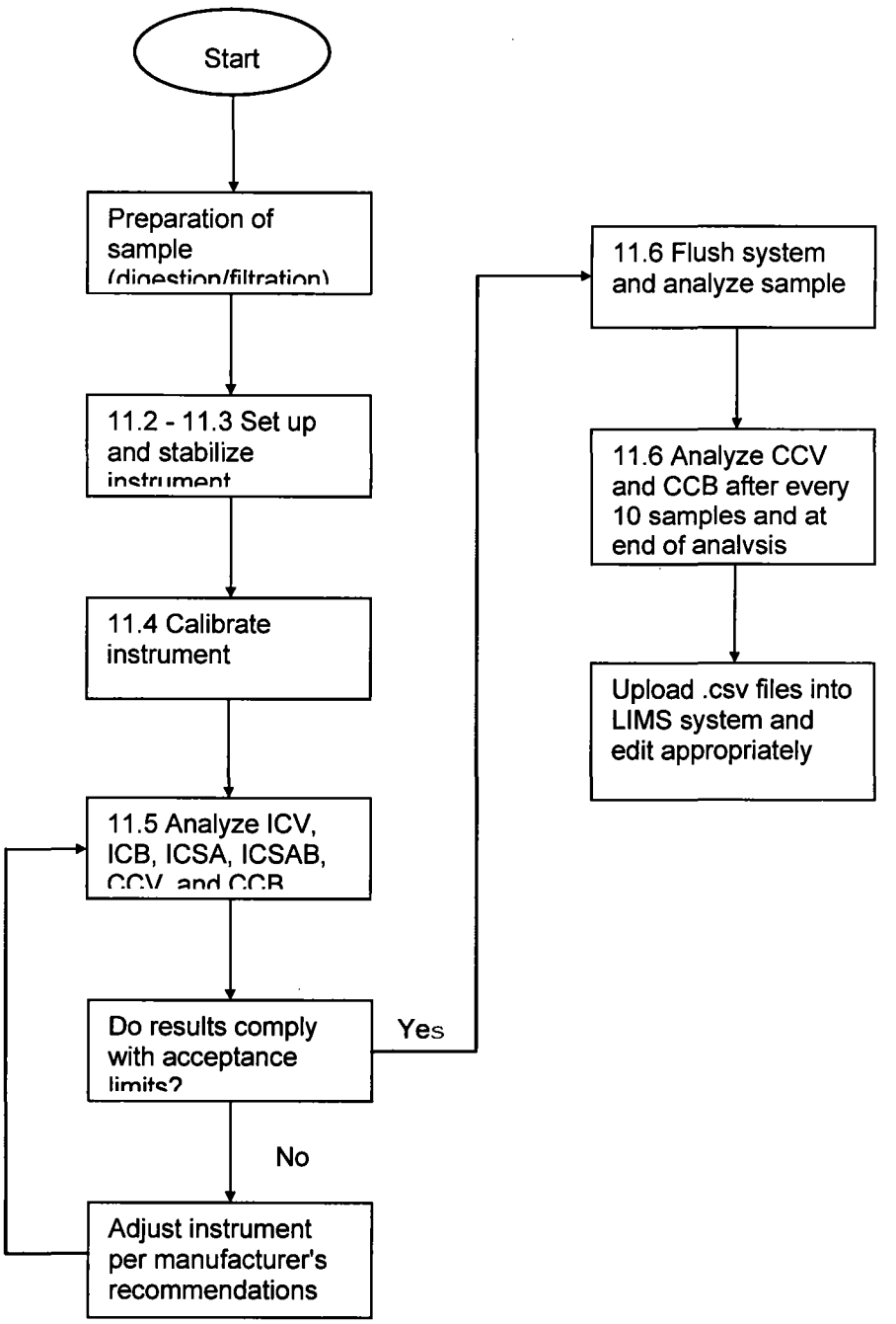
ME401 - Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP, SW846 Method 3005A.

ME403 - Acid Digestion of Soil Sediment, SW846 Method 3050B.

ME406 - Microwave Digestion of Sediments/Sludges/Soils/Oils, SW846 Method 3051.

ME407 - Microwave Digestion – Aqueous, SW846 Method 3015.

10.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



11.0 ANALYTICAL PROCEDURES

11.1 For startup and calibration procedures see Section 8.

11.2 Sample Analysis

11.2.1 Enter sample Ids as indicated in Section 8.2.2. This is best done before calibrating, but is not required.

11.2.2 Designate one post spike and/or one serial dilution per work group. In general it is preferable to analyze the batch blank and LCS prior to analysis of samples.

11.2.3 Fill tubes in Rack 1 in the same sequence as indicated in the runlog beginning with the first sample in position 1-1. See "**Setup Autosampler**" screen as indicated in Section 8.3.2. This will show where all the standard and sample cups should be positioned.

11.2.4 After calibration is complete the system will continue to run the sample set indicated in the autosampler table. **CAUTION:** No more than 10 samples may be analyzed between check standards. This is set up in the "**Protocol Table**".

11.3 Monitor results throughout the analytical run.

11.3.1 The sample results are an average of three replicate readings per analyte. For any analyte with at result greater than the reporting detection limit, the % RSD between the replicate readings must be less than ten.

11.3.2 Look for "High" flags per element and dilute the sample to within the linear range. Look for "Low" flags to indicate matrix interference.

11.3.3 Look for "Pass" or "Fail" flags per element on Check and QC samples. See Table 13 for acceptance criteria and corrective action.

11.3.4 Check MS, post spike and serial dilution. See Table 13 for acceptance criteria and corrective action.

11.3.5 Check TCLP sample results to determine if standard additions are necessary.

11.4 Instrument Shutdown

11.4.1 Pull the sipper out of the holder and put it in a cup of deionized water. Let it rinse for about 15 minutes.

11.4.2 Close all the autosampler screens.

11.4.3 Click on the "Plasma Control Panel" icon and click on [extinguish].

11.4.4 Wait for system to shut itself down and then release the pressure on the tubing.

11.4.5 Remove sipper from cup and lay it on a clean paper towel.

12.0 DETAILS OF CALCULATIONS

12.1 Example calculations are included in each Level 4 data package. See figure 12.1.

12.2 After the calibration is complete, the software performs a weighted linear regression with a calculated intercept for each metals and the analyst can view each curve for acceptance.

$$COC = \frac{\Sigma xy - \frac{\Sigma x \Sigma y}{n}}{\sqrt{\left(\Sigma x^2 - \frac{(\Sigma x)^2}{n}\right)\left(\Sigma y^2 - \frac{(\Sigma y)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

The results are calculated from the calibration curve.

12.3 Dilution factors and preparation factors are calculated into the final result which is computed from the mean of three exposures.

12.3.1 For Liquid Samples:

$$\text{(mg/L) metal in sample} = \text{(mg/L) metal in digestate} \times \frac{\text{final prepared volume (mL)}}{\text{initial volumne (mL)}} \times \frac{\text{total diluted volume (mL)}}{\text{sample aliquot (mL)}}$$

12.3.2 For Solid Samples:

$$\text{(mg/Kg) metal in sample} = \text{(mg/L) metal in digestate} \times \frac{\text{final prepared volume (mL)}}{\text{initial weight (g)}} \times \frac{\text{total diluted volume (mL)}}{\text{sample aliquot (mL)}}$$

12.3.3 LCS % Recovery

$$\% \text{ recovery} = \frac{C_s}{C_t} \times 100$$

where:

C_s = the LCS sample result
C_t = the LCS true value

12.3.4 MS/MSD % Recovery

$$\% \, recovery = \frac{(Cs - Ca)}{Ct} * 100\%$$

where:

Cs = the MS/MSD sample result
Ca = the reference sample result
Ct = the MS/MSD true value

12.3.5 Post Digestion Spike Recovery

$$\% \, recovery = \frac{(Cs - BCa)}{Ct} * 100\%$$

where:

Cs = the spike sample result
Ca = the reference sample result
B = a factor to account for the dilution of the spiked sample relative to the reference sample (usually B = 0.9)
Ct = the spike true value

12.3.6 Duplicate RPD

$$RPD = \frac{|Ca - Cb|}{(Ca + Cb)/2} * 100\%$$

where:

Ca = the reference sample result
Cb = the duplicate sample result

12.3.7 Serial Dilution % Difference

$$\%D = \frac{|Ca - 5Cb|}{Ca} * 100\%$$

where:

Ca = the reference sample result
Cb = the diluted sample result

12.4 Calcium and magnesium results can be used to calculate water hardness results. The LIMS will use the uploaded calcium and magnesium results to calculate the hardness results. The automated hardness calculation is manually verified annually.

Hardness (mg/L) = 2.497[Ca (mg/L)] + 4.118[Mg (mg/L)]

13.0 QUALITY CONTROL (QC) REQUIREMENTS

Overview

13.1 Refer to Section 8 for instrument calibration and instrument quality control samples. Each analytical batch (or workgroup) consists of a maximum of twenty (20) samples plus QC samples. The QC samples are prepared and digested identically to the analytical samples. The frequency, acceptance criteria and corrective action for this QC is listed in Table 13.1 and 13.2.

Batch Quality Control

13.2 Method blank (Prep Blank (PB)) an aliquot of deionized water that is digested with the sample batch and contains all reagents identical with the samples.

13.3 Laboratory Control Sample (LCS) - for water matrix, a spiked deionized water that is digested with the sample batch. The LCS is prepared by diluting 5.0 ml of KEM-SPK-1G (see Section 7.8.1 for initial concentrations) into a 50.0 ml volume of deionized water.

For soil matrix; 1.0 gram of PTFE Boiling Stones (Teflon) Chemware is spiked with 5.0 ml of KEM-SPK-1G prior to digestion.

A laboratory control sample duplicate (LCSD) may also be prepared with an analytical batch and is prepared the same way as the LCS.

The final concentration of the aqueous and soil LCS are as follows:

Table 13.3.2: Concentrations of Elements in LCS & LCSD

Element	Water Matrix (mg/L)	Soil Matrix (mg/Kg)
Potassium Sodium	25	1250
Aluminum Calcium Magnesium	5	250
Silicon	2.5	NA
Iron	2	100
Antimony	0.6	30
Barium Lithium Molybdenum Strontium Titanium Vanadium Zinc	0.5	25
Boron Chromium Copper Manganese Nickel Lead Thallium	0.25	12.5
Arsenic Selenium Silver	0.2	10
Cobalt	0.1	5
Beryllium Cadmium	0.025	1.25

13.4 Sample duplicate (optional) - a sample prepared in duplicate, both carried through the digestion procedure.

13.5 Matrix spike and matrix spike duplicate - a sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spiked with 5 ml of KEM-SPK-1G for each 50 ml of sample. The final concentration spiked into the two spiked samples will be the same as the aqueous and soil LCS level in Table 13.3.2. An LCSD may be prepared instead of an MS/MSD for an analytical batch. The preparation is the same as for an LCS and is described in Section 13.3.2 with the final concentrations listed in Table 13.3.2.

Interference Tests – Post Digestion

13.6 Serial dilution: If the analyte concentration is sufficiently high (at least a factor of 50 above the method detection limit), analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.7 Post Digestion Spike: an analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% of the known value. The spike addition should produce a minimum level of 10 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.8 Method of standard additions: This method must be employed for certain TCLP extracts and can also be used to compensate for sample constituents which enhance or depress the analyte signal producing a slope different from that of the calibration standards. It will not correct for additive interferences which cause a base line shift.

To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration
is approximately 50 percent of the expected concentration from the endogenous analyte in the sample.

Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample concentration. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis.

When the resulting line is extrapolated to zero concentration, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

Linear Range Analysis/High-Level Standard

13.9 A linear range analysis is performed quarterly to determine the highest concentrations for each analyte at which the instrument yields a result within 10% of the true concentration. When use of the Department of Defense version 3 QAP is indicated, a high-level check standard with analyte concentrations at the linear range of the instrument may be analyzed subsequent to sample analysis for any analyte which exceeds the calibration range. The recovery must be within 10% of the true concentration.

Contingencies for Handling Out-of-control or Unacceptable Data

13.10 All data is scrutinized by the analysts for method specific compliance as outlined in sections 11 and 13 of the individual SOPs. Check lists are utilized and accompany each data batch.

13.11 Out-of-control data may be addressed by one or more of the following approaches:

- Internal corrective actions (reanalysis)
- Internal documentation and communication (CAR)
- Discussion and qualification of data (report and narrative)
- Client notification
- Data rejection (R-flagging)
- Re-sampling and re-analysis (client decision)

13.12 When out-of-control situations are encountered, method specific corrective actions are followed. These corrective actions are outlined in Table 13-1. These corrective actions may include a variety of actions such as recalibration and rerunning all affected samples, and if these measures fail, may require contacting the client to inform them of the problem and to obtain their directions on re-running, re-extracting, or re-sampling the samples.

13.13 The out-of-control situations are documented on Analytical Corrective Action forms which are reviewed and signed by the Department Manager and the QA/QC Supervisor. The Client Service Manager is copied on all Corrective Action Forms, so that the Technical Service Representative may inform the client affected by the non-compliant data. These forms are kept on file and are available for review.

13.14 Depending on the decision of the client, the data may be qualified and used and a case narrative will be written to summarize the situation and advise on usability of data.

Table 13.1: Quality Control Criteria for Method 6010B.

Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	Daily, at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	90 - 110 % Recovery	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After every 10 samples and at end of run	90 - 110 % Recovery	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV.
Initial Calibration Blank (ICB)*	After ICV	< RDL , < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)*	After CCV	< RDL, < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB.
Interference Check Standards (ICSA, ICSAB)*	After calibration, prior to sample analysis	Nonspiked elements < RDL or < 2X MDL, Spiked elements ± 20% of spiked concentration	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Method/Prep Blank*	One per analytical batch (20 samples max. per batch)	< RDL, < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative.
Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)	One per analytical batch (20 samples max. per batch)	80 - 120 % Recovery (Solids) 85 – 115 % Recovery (Waters)	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per ten samples (20 samples max. per batch)	75 – 125 % Recovery RPD ≤ 20 %	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate	One per analytical batch (20 samples max. per batch) per client request	RDP ≤ 20 %	Qualify data and address in narrative if client specified.
Post Digestion Spike	One per analytical batch (20 samples max. per batch)	75 - 125 % Recovery	Perform serial dilution if detectable element > 50X MDL.
Serial Dilution	When post digestion spike fails	± 10 % of original determination	Dilute and repeat post digestion spike.

*Acceptance criteria are analyte and instrument specific.

Table 13.1: Quality Control Criteria for Method 200.7.

Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	Daily, at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	95 - 105 % Recovery	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After every 10 samples and at end of run	90 - 110 % Recovery	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV.
Initial Calibration Blank (ICB)*	After ICV	< RDL , < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)*	After CCV	< RDL, < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB.
Interference Check Standards (ICSA, ICSAB)*	After calibration, prior to sample analysis	Nonspiked elements < RDL or < 2X MDL, Spiked elements ± 20% of spiked concentration	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Method/Prep Blank*	One per analytical batch (20 samples max. per batch)	< RDL, < 1/2 RDL < 2X MDL	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative.
Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)	One per analytical batch (20 samples max. per batch)	85 – 115 % Recovery	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per analytical batch (20 samples max. per batch)	75 – 125 % Recovery RPD ≤ 20 %	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate	One per analytical batch (20 samples max. per batch) per client request	RDP ≤ 20 %	Qualify data and address in narrative if client specified.
Post Digestion Spike	One per analytical batch (20 samples max. per batch)	85 - 115 % Recovery	Perform serial dilution if detectable element > 50X MDL.
Serial Dilution	When post digestion spike fails	± 10 % of original determination	Dilute and repeat post digestion spike.

*Acceptance criteria are analyte and instrument specific.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client samples into Seedpak. This is done via Microbac's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and reporting limits. Any elements that are not to be reported must be excluded. The exclusion of data will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1 and 13.2. The analyst must certify the primary review has been done by completing the Data Review Checklist (Figure 14.1), signing, and dating. The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the hardcopy raw data with QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and Kobra Lims workgroup reports. The Data Review Checklist acts as a cover page and will be archived with the hardcopy data. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance.

This review also consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness. The secondary reviewer also signs and dates the Data Review Checklist.

When all levels of review have been completed. The elements being reported on each sample are taken to a done status in Kobra LIMS.

15.0 PREVENTATIVE MAINTENANCE

15.1 Check Nebulizer and spray chamber every day for steady flow of mist. The nebulizer spray chamber and torch should be cleaned when needed.

15.2 Tubing needs to be changed when it loses pliability and is worn.

15.3 The pump rollers need to be cleaned and lubricated about once a month.

15.4 Record daily instrument maintenance on the Maintenance log.

15.5 The instruments are under service contracts so that every year a service representative will perform a systems check.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

Laboratory policies and procedures for management of hazardous waste are found in the SOP33-Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee received training in the proper handling and disposal of hazardous waste that this is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into 55 gallon waste drums in our explosion-proof waste building located outside of the MICROBAC laboratory facility. These drums are labeled and a manifest is created for each. They are picked up by Van Waters & Rogers Inc. on a regular basis for disposal at Von Roll America Inc. – a licensed disposal facility. Acid or base waste is neutralized and disposed in the municipal sewer system as per agreement with the City.

The waste streams for the Metals Laboratory are as follows:

The metals laboratory waste is neutralized with sodium bicarbonate and flushed down the drain with tap water as per agreement with the Marietta Department of Water and Waste Water.

17.0 REFERENCES

17.1 U. S. Environmental Protection Agency, *Test Method for Evaluating Solid Waste*, SW-846, Revision 2, December, 1996. Method 6010B Inductively Coupled Plasma-Atomic Emission Spectroscopy.

17.2 U. S. Environmental Protection Agency, *Method for Chemical Analysis of Water and Wastes*, EPA-600/4-70-020 (Rev. 4.4 May , 1994). Method 200.7 Determination of metals and Trace Elements in water and wastes by Inductively Coupled Plasma-Atomic Emission Spectroscopy.

17.3 Microbac SOP 45, *Method Validation Procedures*

17.4 Microbac SOP ME401, *Method 3005A Acid Digestion of Aqueous Matrices for ICP Spectroscopy*

17.6 Microbac SOP ME406, *Method 3051 Microwave Digestion for Solid Matrices*

17.7 Microbac SOP ME407, *Method 3015 Microwave Digestion for Aqueous Matrices*

17.8 U. S. Environmental Protection Agency, *Inductively Coupled Plasma – Atomic Emission Spectroscopy,* Method 6010C, Revision 3, February 2007.

Figure 12.1

Example 6010 Calculations
Thermo Scientific iCAP 6500

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and four standards.

2.0 Calculating the concentration (C) of an element in water using data from prep log, run log, and quantitation report (note:the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	Example:
Cs = Concentration computed by the data system in ug/mL (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial volume (mL)	50
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/mL (mg/L)	0.1

3.0 Calculating the concentration (C) of an element in soil using data from prep log, run log, and quantitation report (note: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	Example:
Cs = Concentration computed by the data system (mg/L) (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial weight (g)	1
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/g (mg/kg)	5

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where:	Example:
Cx = Concentration calculated as received (wet basis)	5
Px = Percent solids of sample (%wt)	80
$Cdry$ = Concentration calculated as dry weight (mg/kg)	6.25

Figure 14.1

Checklist ID: 37009

Microbac Laboratories Inc.

Data Checklist

Date: 23-MAR-2009
Analyst: KHR
Analyst: NA
Method: 6010B
Instrument: IRIS-ICP
Curve Workgroup: 298039
Runlog ID: 27184
Analytical Workgroups: 297966, 297823, 297967, 297970, 297972, 297975

Calibration/Linearity	X
ICV/CCV	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	
Case Narrative	X
Client Forms	X
Level X	
Level 3	
Level 4	352, 355, 358, 368, 369
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	KHR
Secondary Reviewer	MMB
Comments	

Primary Reviewer: 24-MAR-2009 Kim H. Rhoden

Secondary Reviewer: 24-MAR-2009 Maren Berry

Definitions

The following definitions may be used in the production of this SOP.

Batch:

A group of samples, which are processed together as a unit that undergo the same extraction, concentration, and cleanup.

Batch QC:

The quality control samples within a batch such as a blank, LCS, MS, MSD, and/or dup.

Blank:

An analyte free matrix that is processed with a batch to monitor contamination.

Cleanup:

A prescribed treatment of an extract to prepare the extract for analysis. This treatment may be physical or chemical in nature.

CAR:

Corrective Action Request is a program report that documents the actions taken for an out-of-control event, routine or non-routine.

CCV:

Continuing Calibration Verification is an analytical sample included in the analyses of a batch to verify that the process or measurement is in calibration.

COC:

Chain of Custody

DI Water:

Water that has had all the ions removed by an ion exchange process.

Digestate:

A sample medium treated by a digestion process to release or remove specific target analytes for subsequent analyses.

Digestion:

The process of releasing or removing target analytes from a sample so that they may be quantified in subsequent tests.

Dilution:

The process of reducing the concentration of target analytes by the addition of solvent.

Duplicate:

A split sample that is used to assess precision.

ECD:

Electron Capture Detector

EPA:

Environmental Protection Agency

EQ Blank:

Equipment blank is a sample of DI water poured into, over, or pumped into the sampling device that assesses the effectiveness of equipment decontamination.

Extraction:

The process, which releases or removes specific target analytes for subsequent analyses.

FID:

Flame Ionization Detector

Holding Time:

The maximum elapse of time that one can expect to store a sample without unacceptable changes in analyte concentrations. These times apply under prescribed storage conditions and deviations in storage conditions may affect the holding time. Extraction or digestion holding time refers to the time elapsed from sample collection to sample preparation.

Homogenized:

Samples that are in a uniform mixture and are particle sized so that the particles are uniformly small and evenly distributed.

HPLC:

High Performance Liquid Chromatography

ICV:

Internal Calibration Verification

IDL:

Instrument Detection Limit, the measure of instrument sensitivity.

LCS:

Laboratory Control Sample, a known matrix (DI water, sand, etc.) that is spiked with compound(s) representative of target analytes. This is used to document and assess the extraction or digestion.

Leachate:

A liquid that has percolated through waste, soil, rock, or other material and has mobilized chemical species in the process.

Leaching:

The separation or dissolving out of soluble constituents from a solid material or matrix by the natural action of percolating water or chemicals.

Limit of detection (LOD):

Laboratory verified MDL per SOP 45

Limit of quantitation (LOQ):

Laboratory supported quantitation limit as per SOP 45

LIMS:

Laboratory Information Management System

Matrix:

The component or substrate (water, soil, sludge, etc.) that contains the analyte of interest.

MS:

Matrix Spike is an aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. It is used to document the bias of a method in a given sample matrix.

MSD:

Matrix Spike Duplicate is the same as the matrix spike and it documents the precision of a method in a given sample matrix.

MDL:

Method Detection Limit is a measure of instrument sensitivity using solutions that have been subjected to sample preparation steps.

Precision:

A concept used to describe dispersion of measurements with respect to a measure of location or central tendency.

Preparation:

The action or process of making sample ready for analysis.

Reagent:

Chemical of known purity that is used in analytical methods.

Solvent:

The dissolving agent that usually makes up the greater proportion of a solution.

Soluble:

The dissolving of one substance into another substance.

SOP:

Standard Operation Procedure

Surrogate:

An organic compound which is similar to the target analytes in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.